

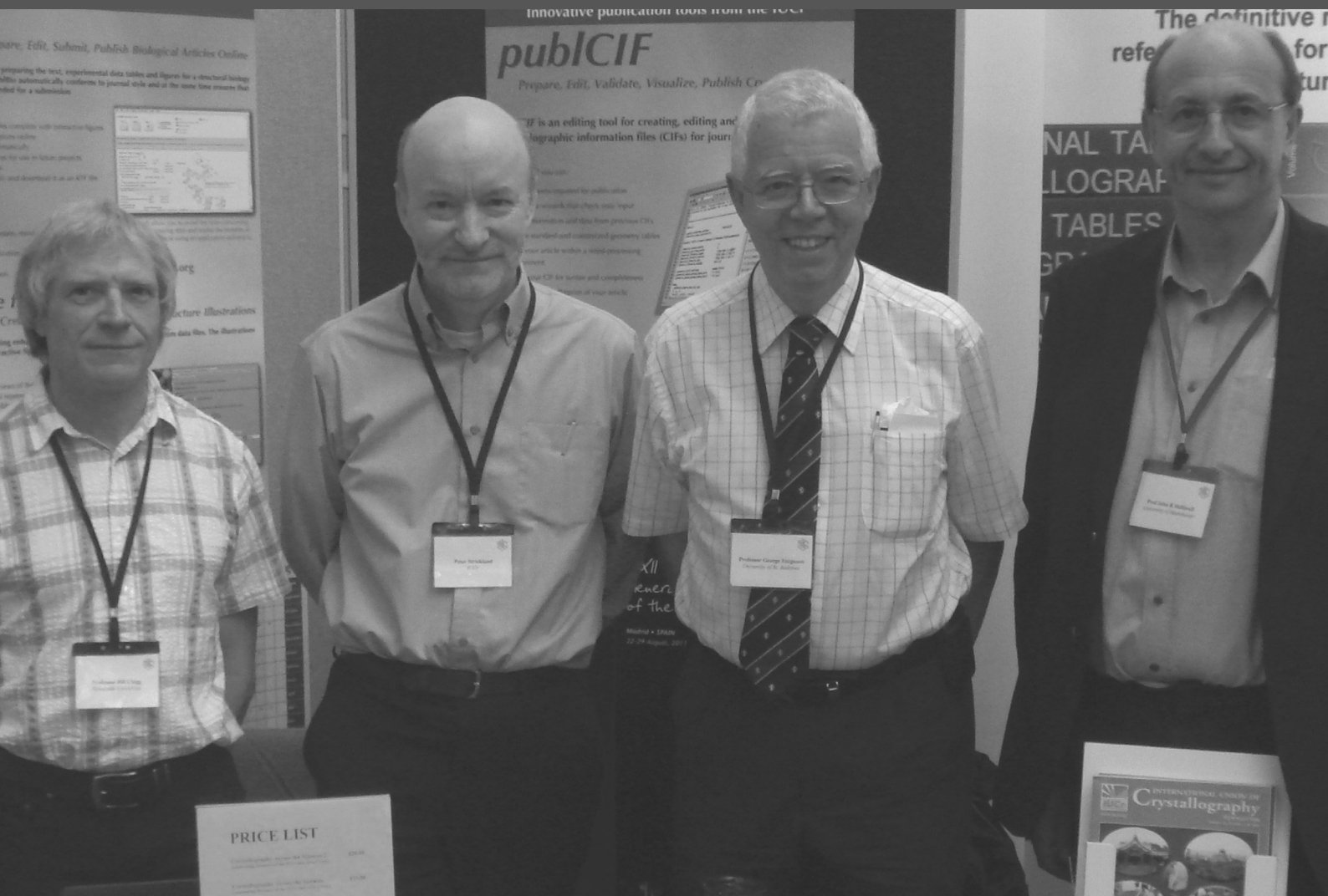
Crystallography News

British Crystallographic Association



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Warwick April 2010 p6

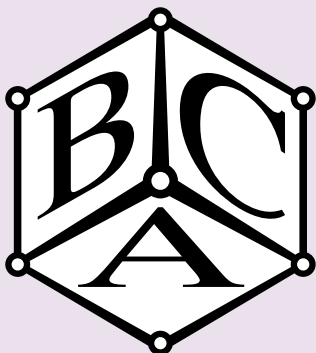


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Crystallographer's Perspective p22



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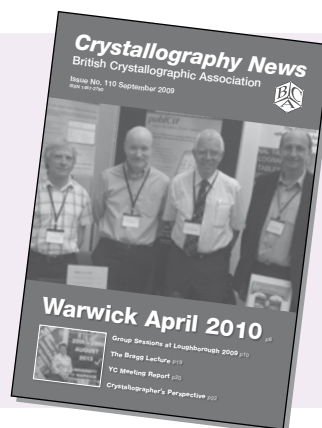
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This month's cover:

Acta Cryst. editors
at Loughborough;
good news for
2013.



From the President



THE summer weather has treated us well so far although the rain is beating down as I write this. I hope those of you with teaching responsibilities are enjoying well earned respite from them to gain energy and enthusiasm for the next academic year and some thinking time for new research projects.

This time of year brings the conference season, and the ACA in Toronto is underway and the ECM in Istanbul is imminent. Both look to have some exciting science to divulge. As I mentioned in the last issue of Crystallography News, in Istanbul the BCA will bid to host the 2013 ECM (the 28th) at Warwick University. On a recent site visit there, I was most impressed by the facilities at the new Arts Centre and the spacious green site. Coincidentally, summer 2013 is also the centenary of the first crystal structure being solved by the Braggs while at Leeds University, and since the last ECM to be held in the UK was in Oxford in 1977, it seems a fitting year in which to hold it on this side of the Channel.

Apropos of the ECM, there have been some questions from BCA members as to the benefits and entitlements gained by Individual ECA Membership (50 euros for 5 years). For this you get (see <http://www.ecanews.org/benefit.php>):

- the discounted rate for ECM registration;
- a vote for Council members;
- the right to propose new Special Interest Groups (SIGs);
- membership of SIGs;
- a discounted subscription to Crystallography Reviews which is the same for BCA Members (£25 for 4 issues).

Note that members of BCA are not individual members of ECA automatically. They are covered by the BCA's National Membership, but this does not entitle them to any of the benefits awarded to individual members listed above.

Another important summer activity is the Protein Crystallography Summer School in St. Andrews organised by Jim Naismith and Gary Taylor, very nobly for the sixth time. This summer school takes place annually and caters for around 40 students, but is alternately in the 'north' (this year) and the 'south' of the UK: currently the 'south' means Oxford organised by Martin Noble and yours truly. They are intense 5 day courses which aim to cover the whole 'pipeline' of macromolecular crystallography from protein purification to validation of solved structures. It is my observation that students can teach each other by solving problems together even more effectively that they can be taught by expert lecturers. These summer schools provide an ideal opportunity for the students to form inter-laboratory and inter-country connections by making new friends involved in similar efforts. They thus extend the base over which they can exchange ideas and seek information, support and advice, as well as - of course - laying the foundation for crystallographic

cooperation on EU Grant applications for the next 30 years! I know that the BCA/CCG biennial Intensive School in Durham fulfils a similar very important role. It is always wonderful to meet ex-students at conferences round the world who attended a course on which one taught years ago, and hear about their life paths. I am a long term addict for such teaching, and because of this unfortunately missed the first day of the BCA meeting in Loughborough this year since at 7 pm the night before the AGM I was still on a macromolecular crystallography beamline at the NSLS in Brookhaven cryo-cooling crystals with a group of very enthusiastic Brazilian graduate students. The Mexican link with my group all stemmed from a student I met 7 years ago while teaching on a course in the US.

Planning for the BCA 2010 Spring Meeting, coincidentally also in Warwick, is now well underway led by Simon Coles, and some details of the programme are already available later in this issue. The Bragg, Hodgkin and Parkin lectures will be given, and we are fortunate enough to have Sir **John Meurig Thomas** FRS and Professor Dame **Louise Johnson** FRS respectively to deliver the first two of these. On a less cheerful note, the last two Spring Meetings have both made substantial losses (in the region of £20,000 each), despite the fact that there were over 375 delegates registered in 2009. Thus, with Northern Networking, the BCA Officers have had to take a long hard look at costings and projections for the next meeting, since as an organisation we cannot sustain this level of subsidy. The result is that speakers at the 2010 meeting will receive a discount of £50, but we will not be able to offer them free registration for the Spring Meeting.

The last IUCr Newsletter featured Part I of Crystallography in Great Britain and Ireland and Part II will appear in the third issue of 2009. **Bob Gould** is to be again congratulated on his great efforts, up to and including some 'interesting' last-minute hitches with the cover and pictures, with which he dealt with his usual diplomacy and quiet efficiency.

During the inadvertent experimental sending out of the pdf of Crystallography News to Members (thanks for all the feedback by the way...), it transpired that nearly one in six of our Members have incorrect e-mail addresses in the Northern Networking database: that means the messages bounce right back. Due to the efforts of one of my summer students (thanks Olga!), a third of these wrong addresses have now been traced and corrected, but if you have colleagues who are members of the BCA and are not receiving any e-mails, please could you ask them to e-mail me (elspeth.garman@bioch.ox.ac.uk) and let me know of their new location and address. Many thanks.

Elsbeth Garman

P.S. At the European Crystallographic Meeting in Istanbul, the bid submitted by the UK to host the ECM in 2013 at the University of Warwick was successful. More details will follow.

BCA Council 2009

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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 Editor



AS I write this column, I am between conferences. I have just returned from a very stimulating and well-organised meeting of the American Crystallographic Association in Toronto. Once again I was impressed with the way Canadians combine

efficiency and kindness. Now I am looking forward to further exciting crystallography at the European Crystallographic Meeting in the exotic surroundings of Istanbul. Reports on both meetings will appear in the December issue. Here we have a full set of reports on the BCA meeting last April, which took place in the more familiar surroundings of Loughborough, and early notice of the 2010 BCA meeting at Warwick.

Along with double acclaim for our future hosts at Warwick, our cover features a concatenation of *Acta Crystallographica* editors (**William Clegg, Peter Strickland, George Ferguson** and **John Helliwell**) at Loughborough. I feel that we did not adequately celebrate the 60th anniversary of *Acta Cryst.* last year, so let this piece commemorate the 61st. The influence of *Acta Cryst.* extends well beyond its own pages. The CIF developed under the auspices of the IUCr and required by *Acta Cryst.* has now been adopted by many other journals. In 2006 the importance of CIF and the value of its *checkCIF* web-based service for the validation of structural data were recognised by the Award for Publishing Innovation of the Association of Learned and Professional Society Publishers (ALPSP). The reliability and accessibility guaranteed by these standards have greatly helped to maintain the respect other scientists have for crystallographers, and saved our subject from becoming “just another analytical technique.” More recently *Acta Cryst.* has been a leader in the adoption of electronic publishing and open access.

On the subject of anniversaries, **Derry Jones** commemorates the 50th anniversary of **C. P. Snow**'s famous Rede Lecture in which he deplored the split into “Two Cultures” of the community of educated people. An interesting attempt to bridge the Two Cultures of science and humanities was the Art in Crystallography exhibit at the recent ACA meeting. I had intended to publish a photograph I took of this exhibit, severely angled to include many pictures but exclude background clutter. Due to the viewing angle it would have provided the physicists among our readers with a fine example of distortion. However, the latest IUCr newsletter (volume 17, number 2) has just

appeared, featuring exactly these images on the cover in glorious colour and undistorted. Although I cannot find any attribution, I wonder if photographer extraordinaire **Bill Duax** took these beautiful pictures.

Wherever we work, during the past few years most of us have had to get used to the experience of “being managed”, losing autonomy. Therefore it was really refreshing to be consulted about the role and form National Services for crystallography should take. I hope as many BCA members as possible took part in the consultation. I hope that members also considered the information circulated by the BCA Council about the new policy announced by the EPSRC in March to introduce “blacklisting” of researchers whose success in grant applications fell below a certain level, and many were motivated to express their opinion.

John Helliwell gives us an informative account of his stewardship of the organization of the Bragg Lectures. In the BCA we are privileged to have many brilliant speakers delivering Plenary Lectures in areas of interest to our Groups. The lectures at our Loughborough meeting were a shining example. However, the Bragg lecturers, like the Braggs themselves, tend to combine expertise in crystallography and in an adjacent field, thereby supplying a thought-provoking new perspective. Adding to the inducements to come to Warwick next spring, 2010 is the next Bragg Lecture year. The speaker will be Professor Sir **John Meurig Thomas** FRS of the Department of Materials Science and Metallurgy, University of Cambridge.

Recently I made use of the Government's scrappage scheme to trade the family's old banger for a shiny new car. Now, if only there were a similar scheme for X-ray equipment...

Before you read this final paragraph, please be sure you are firmly seated in your chair so you won't fall out with surprise. This is an appeal on behalf of a charity NOT to send money. If you have a standing order regularly paying your dues to the BCA, please cancel it. The receiving account has been closed, so any standing order payments will bypass the BCA and end up somewhere in the banking system. If despite recent disclosures of huge bank profits and the reappearance of huge bankers' bonuses you still wish to make a donation to the Unknown Banker, feel free to do so. However, for your BCA dues please pay by cheque or credit card if you want control, or by direct debit if you want a painless method (we promise you won't feel a thing).

Carl Schwalbe

Puzzle Corner

NAME THE CCDC CHILDREN

AS readers of Sam Motherwell's meeting report in this issue will find out, the CCDC staff are very prolific at turning out software packages.

The younger ones seem good at producing children as well. Because it sometimes can be difficult for new parents, I call upon our readers to help them with suitable names for such children. Cambridge Structural Database names follow the six-letter sequence consonant-vowel-consonant-consonant-vowel-consonant. In this scheme only A-E-I-O-U count as vowels, and W or Y can be freely used in diphthongs.

The children will need a first name that obeys this rule, followed as a middle name by the surname of a crystallographer-the more famous, the better-that also obeys the rule. Please supply a wide choice of appropriate names, or else successive generations within the same family will need to keep the same name followed by 01, 02, etc., and colleagues' children's names will have to end with 10, 20, etc.!



BCA Corporate Membership

The BCA values its close ties with commercial companies -involved with crystallography. To enhance these contacts, the BCA offers Corporate Membership. Corporate Membership is available on an annual basis running from 1 January to 31 March and includes the following benefits:

- Up to 10 free BCA memberships for your employees.
- A 10% discount on exhibition stands on the annual BCA Spring Meeting, OR - A promotional poster at the annual BCA Spring Meeting.
- Free insert in the annual Spring Meeting delegate bag.
- Two free full registrations to the annual Spring Meeting.
- Ten complimentary copies of the quarterly BCA Newsletter.
- Corporate Members will be listed in every BCA Newsletter and on the BCA Web Site with links to your corporate site.

The cost of this membership is **£750.00** per annum. To apply for Corporate Membership, or if you have any enquiries, please contact:

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BCA AGM 2010

BCA Annual Spring Meeting University of Warwick 12th-15th April 2010 “Data Matters”

WHY break a winning formula?! The main Spring Meeting will run, as in previous years, from 11:30 on Tuesday 13th April to 13:30 on Thursday 15th April. The title of the meeting is “Data Matters”, a theme designed to cut across all the groups and be applicable to all areas of crystallographic study: from collection and techniques, through processing and analysis, to publication and reuse. There will be 19 symposia (titles and timetable below) based around this theme all of which will have 2 invited speakers and a third selected from submitted abstracts. In addition to the usual Exhibition, there will be a ‘drop in Software Fayre’ (Organisers **Horst Puschmann & Simon Coles**) running throughout the meeting where specific software demonstrations and problem sessions will be conducted.

During the meeting there will be a workshop on ICDD organized by **Judith Shackleton & Martin Gill**. However, in addition there will be two satellite events:

- The Young Crystallographers will again organise their Satellite Meeting from 1pm on Monday 12th April and finishing at 11.15 on Tuesday 13th April. As announced at last years YC satellite meeting, 2010 will see the establishment of the Parkin Lecture as the YCG prize lecture in special recognition of the late Dr **Andy Parkin** and his contributions to the Group. The YCG committee is proud to present the Foundation Lecturer for the inaugural Parkin Lecture: Prof. **Simon Parsons** (University of Edinburgh). The lecture is scheduled for 10:30 on Tuesday, 13th April, and will be this year’s link between the YC Satellite and the main Spring Meeting - so all main meeting delegates are welcome to attend!
- A second hands-on workshop: Local structure, data correction matters (How to collect and correct data for Pair Distribution Function and total scattering analysis) will be held after the main meeting closes at 13:30 on Thursday 15th April. This workshop will follow on from the PDF and Reverse Monte Carlo analysis workshop held before the 2007 BCA spring meeting. The aim will be to enable people to produce data suitable for the analysis tools demonstrated in the last workshop. If you

would like to attend this workshop or would like further information please email the organizer, **Matt Tucker** (matt.tucker@sftc.ac.uk), so the level of interest can be gauged.

Prize Lectures and Plenary Speakers

This year we are due to present two prize lectures and are honoured by the presence of two eminent scientists who are both FRS and also knighted!

Professor Dame **Louise Johnson** DBE FRS (Dept of Biochemistry, University of Oxford & Diamond Light Source Ltd) is the awardee of the Hodgkin prize and will open the meeting with a lecture entitled, “Forty years of structural biology: where we have come from and where might we be going?”

Professor Sir **John Meurig Thomas** FRS (Dept of Materials Science and Metallurgy, University of Cambridge) is the recipient of the prestigious Bragg Lecture prize and will give a lecture entitled “The Essence and Promise of 4D Electron Microscopy”. JMT will speak about real space crystallography, electron energy-loss spectroscopy and many things which are currently of interest to synchrotron-oriented studies. He will also pay homage to the Braggs and recall some important history about the Royal Institution. To quote, “It will be an interesting journey through X-ray crystallography, Imaging and Electron spectroscopy”!

Our plenary speakers this year will be Professor **Simon Billinge** (Columbia University), who will give the IG Teaching Plenary and Dr **Lynne McCusker** (ETH Zürich) who will deliver the PCG Plenary. There will also be the usual CCDC Younger Scientist, PCG Thesis and Young Crystallographers Industrial Group Prize Lectures in addition to the **Alun Bowen** lecture to be given by the retiring BCA devotee, **David Taylor**.

Dates, Links & Abstract Submission

Warwick University: <http://www2.warwick.ac.uk/>

All information relating to the conference and submission of talk and poster abstracts will be provided through the Conference Website:

<http://crystallography.org.uk/spring-meeting>

The deadline for oral presentations to be considered for

symposia is 2nd October. Abstracts should be submitted using the template and website at:

http://www.crystallography-meetings.org.uk/uploader/cp/upload_form.php. As ever, the poster session is a very important part of the meeting and the deadline for abstracts will be 5th February 2010.

Abstract Submission for YC2010: Following the format of previous YCG Satellites, there will be three sessions of oral presentations as well as one poster session. These sessions provide PhD students and early post docs (within 5 years of graduation) with a unique opportunity to present and discuss their research in a friendly and relaxed atmosphere. Please submit abstracts to be considered for oral contributions by 15th January 2010, and for posters by 5th February 2010.

Programme Committee

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 Professor **Elsbeth Garman** (BCA President)
 Professor **Sandy Blake** (BCA Deputy President)
 Mr **David Massey** (Northern Networking)
 Professor **Vilmos Fülöp** (BSG)
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 Dr **Matthew Johnson** (IG)
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 Dr **Matthew Tucker** (PCG)
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| Title | Group(s) | Chair(s) |
|--|----------|-------------------------------|
| Data; What Goes In | IG | Matthew Johnson |
| Data; What Comes Out | IG | Brett Cooper & Cheryl Doherty |
| Complementary Non Ambient Techniques | IG | David Beveridge |
| Unpublished Data And Almighty Blunders | IG | Luca Russo & Richard Morris |
| Electron Diffraction | PCG | Kirsten Christensen |
| Resonant X-Ray Diffraction | PCG | Peter Hatton |
| New Approaches To Structure Solution | PCG | David Keen |
| PDF: Local Structure | PCG | Matt Tucker |
| Structure And Property Prediction | CCG | Stephen Moggach |
| Databases & Data Mining | CCG | Hazel Sparkes |
| Data & Structure Validation | CCG | Ross Harrington |
| High Throughput: Membrane Proteins | BSG | Steve Prince |
| High Throughput & Screening 2 | BSG | Bill Hunter |
| Dealing With Difficult Data | CCG | David Watkin |
| Data Management | BSG | Keith Wilson |
| Dynamics | BSG | Mark Sansom |
| Hot Structures | BSG | Steve Smerdon |
| Membrane Structures | BSG | Liz Carpenter |
| New Techniques | BSG/YCG | Robin Owen |

| | Mon April 12th | Tuesday April 13th | | | |
|-------|-----------------------|--|--|---|------------------------|
| 9:00 | | LT3 | | | |
| 9:15 | | YC 4 | | | |
| 9:30 | | | | | |
| 9:45 | | | | | |
| 10:00 | | | | | |
| 10:15 | | LT3 Parkin Lecture: Simon Parsons | Registration/Exhibition 10:00-11:30 | | |
| 10:30 | | | | | |
| 10:45 | | | | | |
| 11:00 | | | | | |
| 11:15 | | | | | |
| 11:30 | | LT3 Hodgkin Lecture: Prof. Dame Louise Johnson FRS Chair: Elspeth Garman | | | |
| 11:45 | | | | | |
| 12:00 | | | | | |
| 12:15 | | | | | |
| 12:30 | | | | | |
| 12:45 | | Lunch/Registratio/Exhibition 12.30-13.30 | | | |
| 13:00 | PLT | | | | |
| 13:15 | YC 1 | Sessions 13.30-15.00 | | | |
| 13:30 | | | | | |
| 13:45 | | LT4 | LT5 | PLT | |
| 14:00 | | Resonant X-ray Diffraction PCG | Structure & Property Prediction CCG | New Techniques BSG/YCG | |
| 14:15 | | | | ICDD Workshop IG | |
| 14:30 | | | | | |
| 14:45 | | | | | |
| 15:00 | Coffee 15.00-15.30 | Coffee 15.00-15.30 | | | |
| 15:15 | | | | | |
| 15:30 | PLT | Sessions 15.30-17.00 | | | |
| 15:45 | YC 2 | LT 1 | LT2 | LT3 | |
| 16:00 | | New Approaches to Structure Solution PCG | High Throughput 1 BSG | Alun Bowen Lecture David Taylor IG | |
| 16:15 | | | | | ICDD Workshop IG |
| 16:30 | | | | | |
| 16:45 | | | | | |
| 17:00 | Break | Break | | | |
| 17:15 | PLT | LT3 | | | |
| 17:30 | YC 3 | | | | |
| 17:45 | | Exhibitors Forum 17.15-18.45 Chair: Elspeth Garman | | | |
| 18:00 | | | | | |
| 18:15 | | | | | |
| 18:30 | | | | | |
| 18:45 | YC Dinner | | | | |
| 19:00 | | | | | |
| 19:15 | | | | | |
| 19:30 | | | | | |
| 19:45 | | Dinner, Exhibition and Posters | | | |
| 20:00 | | | | | |
| 20:15 | | | | | |
| 20:30 | | | | | |
| 20:45 | | | | | |
| 21:00 | | | | | |

| Wednesday April 14th | | | Thursday April 15th | | |
|---|-----------------------------|------------------------------|---|--------------------------|---|
| LT3 PCG Plenary: Dr Lynne McCusker Chair: David Keen | | | LT3 IG Teaching Plenary: Prof. Simon Billinge Chair: Matt Johnson | | |
| Coffee 9.45-10.15 | | | Coffee 9.45-10.15 | | |
| Sessions 10.15-11.45 | | | Sessions 10.15-11.45 | | |
| LT4 | LT5 | PLT | LT4 | LT5 | PLT |
| Dealing with Difficult Data CCG | Dynamics BSG | Electron Diffraction PCG | Complementary & non-ambient techniques IG | High Throughput 2 BSG | Data & Structure validation CCG |
| IG AGM 11.45-12:30 | BSG AGM 11.45-12.30 | PCG AGM 11.45-12.30 | Break | | |
| Lunch, Exhibition 11.45-13.30 12.30-1.30 | | | Sessions 12.00-13.30 | | |
| | | CCG AGM 12.30-1.15 LT1 | LT4 | LT5 | PLT |
| | | | PDF: Local Structure PCG | Hot Structures BSG | Unpublished Data & Almighty Blunders CCG/IG |
| Sessions 13.30-15.00 | | | Close 13.30 | | |
| LT4 | LT5 | PLT | Location TBA | | |
| PCG Prize Chair D. Keen | Data; what goes in IG | Membrane Structures BSG | PDF Workshop | | |
| CCDC Prize Chair A. Bond | | | | | |
| Coffee 15.00-15.30 | | | | | |
| Sessions 15.30-17.00 | | | | | |
| LT4 | LT5 | PLT | | | |
| Databases & Data Mining CCG | Data Management BSG | Data; what comes out | | | |
| | | YC Prize IG Sponsored | | | |
| Break | | | | | |
| LT3 Bragg Lecture: Prof. Sir John Meurig Thomas FRS Chair: Elspeth Garman | | | | | |
| LT3 | | | | | |
| BCA AGM 18.00-19.00 | | | | | |
| Comfort Time | | | | | |
| Conference Dinner 19.30 for 20.00 | | | | | |

Group Sessions at Loughborough

XRD Session Crystallography in the Pharmaceutical pipeline



Speakers - Left to Right: Jeremy Cockcroft, Matthew Johnson (Chair), David England, Cheryl Doherty.

AS crystallographers we have a great understanding and appreciation for molecular structures and the interactions that occur at this scale. Utilising this understanding, shifting the knowledge so that it can be applied constructively to problems beyond crystal structures, within the pharmaceutical industry, is the great challenge. This session focused on the application of crystallographic data to problems outside the usual comfort zone of collecting and determining a crystal structure.

Crystallography in Drug Development

Cheryl Doherty, Pfizer

Cheryl gave an excellent talk presenting two case studies illustrating two uses of crystallography in the pipeline. The first case study described how computational methods such as polymorph prediction and molecular dynamics can be used to influence a polymorph screen by focusing the experiments to engineer a desired drug form with the correct hydrogen bonding. The second case study utilised non-ambient humidity conditions to collect crystal structures of a variable hydrate to understand the mechanism by which the compound could dehydrate and rehydrate. The information gathered from these crystal structures and solid state NMR, identified disordering of the fluoro-phenyl group as a potential channel for solvent movement, flipping of the ring acting as a revolving door.

The Crystal Structure is the Gold Standard for Proving of Structure of a Drug Substance. What can be achieved for the Drug Product?

David England, Sanofi-Aventis

David's talk focused on the drug product, an area of research which in the past has had little interaction with the crystallographer, but with the advances of computational modelling software, computer hardware and imaging techniques that can probe to sub-micron levels, the molecular and the macroscales are merging. The first case study used mesoscale simulations to rationalize the behaviour of complex polymer-based drug formulations and help to understand changes which occur during stability studies. The modelling showed how the drug interacted within the formulation, self-assembling to create a shell of drug forms around the propylene oxide rich regions, which is driven by the hydrophobic nature of the drug. The second study looked at tablet blends formulated by wet granulation and how accurate mapping of the surfaces by methods such as Raman and TOF SIMS mass spectrometry can be used to define the location of components and the degree of mixing. The sub-micron resolution of these techniques gives immense information regarding the interactions of the components and by utilising this knowledge it may be possible to speculate which components will mix favourably.

From the Industrial via the Academic Laboratory to the Court Room: Ciclovir - A Pharmaceutical PXRD Case Study

Jeremy K Cockcroft, UCL

Jeremy's talk covered the court room and the synchrotron, two different areas of pharmaceutical endeavour. He presented the case of Roche versus Ranbaxy over valganciclovir and his personal account of his involvement in the case: from testing powder samples under non-ambient conditions, through producing documents to the court, to the final court appearance. Ranbaxy argued that the amorphous form they manufactured did not infringe on the crystalline patent of Roche. Jeremy demonstrated by quantitative analysis of amorphous content of the samples by XRPD, that the samples were X-ray diffraction amorphous and therefore demonstrated the information disclosed in the Ranbaxy patent to be true. The case is still in the court.

Jeremy also presented the data he collected on I11, Diamond Synchrotron, for famciclovir, demonstrating that high resolution data can be collected on multiple forms to develop an intricate phase diagram quickly using this facility.

Matthew Johnson, GSK

XRF / XRD Plenary Environmental Applications and associated session



Speaker - Left to Right: David Taylor (chair), Nick Marsh

X-Ray Analytical Techniques and Global Warming

Nick Marsh, University of Leicester

There is a lot of information in climate records, which can lead to predictions about what we might be doing to the climate. It's worth remembering that earth's climate has varied considerably over geological time, and there is no particular reason why it should stay as it is over such timescales. In the Jurassic period, for instance, what is now Great Britain was an archipelago of tropical islands.

Oxygen has two reasonably abundant stable isotopes, ^{16}O and ^{18}O . Chemical reactions often discriminate between them, but differentiation is greater in biological systems. Early work on geological and related samples involved carbonates, because it is easy to extract the CO_2 . Unfortunately, not all marine and lake environments are conducive to retaining carbonate phases, so there are gaps in the record. There are further complications, in that chemical change carries on after the deposition of the original sediment. Dolomitisation, for instance, causes a change in the oxygen isotope ratio.

Some plankton such as diatoms and radiolarians use silica to produce their skeletal frameworks. These can be used to plug the gaps in the oxygen isotope record, and also give us access to information from freshwater lakes. Analytically, the oxygen is harder to get at, often requiring the use of

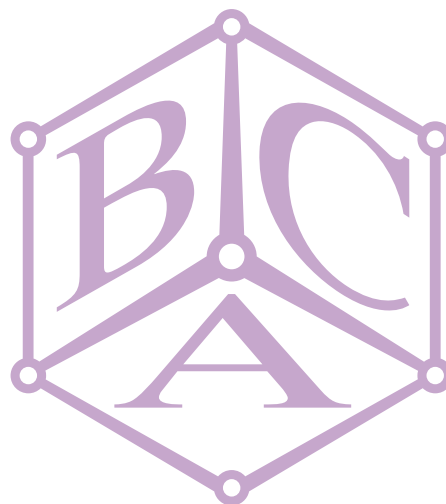
HF. The problem gets worse, through the contamination of biogenic silicate samples with a variety of mineral and/ or rock fragments with markedly different oxygen isotope signatures to diatoms. Diatoms are hollow, and grains of 'sand' (volcanic dust, calcite, whatever) can get inside them.

Decontamination of these samples can be complicated. Acid digestion is risky; it is moderately safe for the removal of carbonates, but does tend to use water, which of course contains oxygen. Oxidation of organics is often useful. A range of physical techniques can be applied, but they tend to fail when there are particles inside a diatom. XRD can be used to identify the contaminants, but this has its limitations: volcanic ash is often glassy, and the diatom silica tends to be amorphous too. With SEM and EDAX, it is possible to use XRF identification of the chemical signatures of the contaminants, and then remove their effects by a chemical mass balance: diatoms are roughly 92% SiO_2 , 2% Al_2O_3 , 6% H_2O , and little else.

Samples, as received in the lab, are often only 50 - 60 mg. The customers want as much of the periodic table as possible, plus loss on ignition. The order of priority is usually the loss on ignition, followed by high-quality determinations of the main elements. The first choice for the technique is fusion beads with a high flux/sample ratio: the usual flux is $\text{Li}_2\text{B}_4\text{O}_7$. It is normal to find traces of Pt and Au in the beads - the crucibles (95Pt - 5Au) are not totally insoluble in the flux! A catch-weight system is used, without matrix corrections as the dilutions are so high.

The results are generally good for the major elements, but a number of special techniques have to be used in some cases: Zr/Sr and Y/Rb overlaps have to be corrected for. Pb tends to give odd results which are not yet fully understood. Work is still very much in progress improving the techniques. Cu, Ni and Cr are affected by contributions from various bits of the spectrometer: this could be hard to circumvent. Some results were presented from Lake Baikal in Russia and Lake Tilo in Ethiopia: in the latter case, the effect of volcanic ash is significant. To sum up, with equipment which is modern in all respects, and reliable reference materials, it is possible to get good results.

David Beveridge, HARMAN Technology



XRD Session

Understanding API Phase Transitions



Speakers - Left to Right: Brett Cooper (Chair), Russell Johnstone, Nick Blagden, Paolo Avalle.

Thermal Properties of Tolbutamide and Paracetamol by Real-Time Variable Temperature Raman Microscopy

Paolo Avalle, MSD

In the first IG presentation of the morning Paolo Avalle really hit the theme of the conference for dynamic crystallography, by demonstrating how, by applying Raman spectroscopy in real time, you can gain some insightful information into what is really happening during an API Phase Transition. Paolo described how the individual peaks in the Raman pattern could be assigned to specific areas of the molecule. He then demonstrated how these peaks could be tracked in real time during the heating of samples of paracetamol or tolbutamide. Then during phase transitions, he demonstrated how these peaks shifted. By looking at the crystal structures of the molecules he could then gain information about how the molecules were reorganising during the polymorphic transitions. By the use of principal component analysis he could plot the phase transitions with temperature and determine how many distinct phases were present during the transitions.

Co-Crystals and Crystal Growth

Nicholas Blagden, University of Bradford

The second presentation of the morning saw a change to the scheduled program. **J.Y. Khoo** was unable to attend the conference so **Nicholas Blagden** heroically stepped in at the last minute to take the slot. Nick managed to put together an excellent presentation in only a couple of hours and even managed to aim towards the conference and session themes too. Nick described some of the

theories of co-crystals and crystal growth. Explaining how co-crystal pairs could be selected by the likely strength of their inter-molecular interactions, in a crystal engineering approach, he then demonstrated how you could screen for co-crystal formation using the Kofler contact method. He showed an example video of co-crystal growth at the interface between isonicotinamide and benzoic acid by hot stage polarised microscopy. He also demonstrated how ternary phase diagrams could be applied to solvent based co-crystallisation studies.

Identification of Driving Forces in High Pressure Phase Transitions Using the PIXEL Method

Russell Johnstone, University of Edinburgh

In the last talk of the session Russell Johnstone applied real pressure! Up to 10 GPa in fact by the use of a Merrill-Bassett Diamond Anvil Cell. Russell described how crystals could be subjected to very high pressures while collecting single crystal X-ray or neutron powder data. He then described the phase transition that occurs at high temperature for L-serine monohydrate, showing the changes that occur to the crystal structure and intermolecular bond lengths. He also demonstrated how the PIXEL method can be used to understand why pressure-induced phase transitions occur.

Brett Cooper, MSD

XRD Monitoring Crystals

'Monitoring Crystals, Crystallization and Transformations' 1

Chairs: **Nick Blagden & Anne Kavanagh**

Chick Wilson opened this session with an overview of 'crystallisation phase space' in which he posed the question: should we expect the unexpected? And answered: yes, particularly polymorphism. Chick explored the question of whether favourable (even tunable) solid state properties of molecular materials can be achieved by understanding how those properties are determined by crystal structure, and how these desirable structures can be achieved. To do this, the structural chemist attempts to understand and control intermolecular bonds, particularly hydrogen bonds, using crystallisation, crystallography, solid state analysis and quantum chemical calculations.

Paul Barnes looked back at how in-situ synchrotron X-Ray diffraction has been applied, to study both the formation and the performance of functional materials. Time-resolved powder diffraction has been used to study rapid changes

taking place in response to changes in temperature, pressure and chemical conditions in systems as diverse as gypsum (dehydration), cement sintering, and catalyst studies. TEDDI (Tomographic Energy Dispersive Diffraction Imaging) has allowed spatially-resolved information to be gained in systems such as crystallisation of zeolite A on a ceramic base, (in which the crystallisation occurred in an unexpected place: the underside of the platform!). The last part of Paul's talk explored how the current limitation of TEDDI: data collection speed and limited analysis volume might be overcome in the future.

Robert Hammond described how crystallisation control techniques can be used to produce 'the right stuff' for the pharmaceutical industry: crystals of the desired polymorph, crystal habit and size distribution. Control of polymorph is an essential in production of pharmaceuticals, but crystal habit and particle size distribution can also be important, since they affect secondary processing, such as filtration and powder flow. Robert explained how inline monitoring of particle size, polymorph and supersaturation can be achieved, and also described the use of 'closed loop' crystallisation of glutamic acid, in which low supersaturation, and seeding, were used to ensure the production of well-facetted crystals.

'Monitoring Crystals, Crystallization and Transformations' 2

Chairs: Nick Blagden & Anne Kavanagh

Roger Davey gave the first talk in this session, on work aimed at understanding how structure evolves during the nucleation process. He investigated the relationship between the structure in the liquid crystal phase of *p*-azoxyanisole (*pAA*), and the structure of the crystalline phase which nucleated from the liquid crystal on cooling. He found that one polymorph, Form 1, was always formed, and Form 3 was never formed directly from the liquid crystalline phase. Roger concluded that the structure of the crystalline phase is dictated by the ordering within the supersaturated liquid crystalline phase, but that an energy barrier to nucleation still exists, since crystallisation was not observed, even after several weeks, at temperatures above 80°C.

Andrew Fogg described the use of *in-situ* synchrotron XRPD to follow the extent of reactions with time, and hence obtain invaluable information, such as activation energies, reaction order and nucleation models. Andrew gave an overview of some of the systems which have been studied. These included layered double hydroxides used as ion exchange materials, in which the stacking sequence and the formation of intermediates were investigated, and lanthanide intercalation compounds in which the original two dimensional structure was seen to convert to a three-dimensional structure, this observation being confirmed by high resolution electron microscopy.

The finale of the session was the announcement of the

winner of the Industrial Group Prize for the best, industrially-relevant talk to be given in the Young Crystallographers Meeting. The prize was awarded to **Anne Stevenson** from Bath University, who described her work on novel metal-organic framework compounds. These compounds contain pores whose size can be controlled, and also high surface area, and these features make them of interest for a variety of applications including gas storage and purification, catalysis, and even medical applications. Anne received a cash prize and a bottle of bubbly from the IG Committee.

Anne Kavanagh, AstraZeneca

XRD Environmental Applications



Speakers - Left to Right: Richard Morris (chair), Peter Stacey, Didier Bonvin, Clive Roberts

New Applications in the Use of X-Ray Diffraction at The Health and Safety Laboratory:- A Case Study Using XRD to Assess Emissions Across Construction Sites

P Stacey, Health and Safety Laboratory

Peter treated us to a salutary reminder of the safety aspects of dealing with free silica. He outlined first what the HSE remit is, including a proficiency testing scheme, forensic investigations, data preparation for court cases and environmental measurements. He then detailed a case study measuring respirable silica dust at a construction site; there is no safe lower level; measurements were taken both up wind and down wind of the active construction site. In this case no hazard to the general public was identified.

Heavy Metal Content in Canal Sediments in the Black Country

Clive Roberts, University of Wolverhampton

Clive described his study of industrial contamination in

canals as a work in progress with As, Cd, Cr, Pb, Hg & Se all being harmful to health. Lead was found to increase near to motorways. Many of the industries that previously lined the canals have contributed to specific contamination problems. Coal transshipment or collieries themselves raised arsenic levels. Zinc was high in the vicinity of a zinc plating works & high phosphorus near a match factory. Bi-valves (Swan Mussels) were found to be living on and not in the sediment of the canal bottom. Some heavy metals were found in the sediment that exceeded EU threshold levels by an order of magnitude, but these were not being taken up by the water column which rarely exceeded EU levels.

Matching XRF and XRD Solutions with Analytical Needs for Cleaner, Safer and Healthier Environment

Didier Bonvin, Thermo Fisher Scientific

Didier spoke to us about environmental limits and outlined some specific applications. He discussed the measurement of sulphur in fuels and checking alternative fuels for contamination before allowing them to be burnt in, for instance, the lime industry. Another example was the screening of soils where old industrial sites are required to be turned safely into playgrounds. This limited the amount of soil for disposal and thereby trimmed the overall cost of such projects. He also mentioned the measurement of lead dust in old paints and wallpaper in the home. He then moved on to the analysis of waste solvents prior to disposal. Here method development was used to speed up the analysis and assist in the choice of a safe disposal route. XRD; XRF; EDXRF & WDXRF were the valuable tools used in all of the above examples and also to aid quality control of drug products and baby milk powder.

Martin Gill, Natural History Museum

XRF Session Environmental Applications



Speakers - Left to Right: Richard Morris, Christine Vanhoof, Ian Croudace, Dave Taylor (chair).

FORTIFIED by lunch we continued the environmental theme with a talk by **Chris Vanhoof** from the **Flemish Institute for Technological Research (VITO)** on **How XRF fits into ROHS analysis. Directive 2002/95/EC restricts the use of certain hazardous materials in electrical and electronic equipment and this talk described the support given to the RoHS inspection campaign in Belgium. The analytical framework covered quality control to validate incoming raw materials and/or the final product as well as inspection control to enforce the legislation. Analytes of interest were Pb, Hg, Cr(VI), Cd, polybrominated biphenyls and polybrominated diphenyl ethers. Energy dispersive XRF was the test method of choice for total element determinations providing simultaneous determination for Pb, Cr, Cd, Hg and Br:Sb ratio, the latter being a useful measure as an indicator for the presence of PBDE in flame retardants.**

Chris stressed that the quality of the analytical results was dependent upon how a sample was analysed, with inter-element effects and spectral line interferences being prime concerns. Examples of the analysis of small components, a printed circuit board and a hair drier, showed the ability of a hand-held system to do the job. Screening and mapping by XRF techniques of glass light bulbs were also shown to be effective. The draft document IEC 62321 provided information on reference methods whilst CRM 680 was used as a control reference sample. XRF was shown to be a useful technique for the screening of "suspect" components.

David Taylor, who chaired this session, then introduced **Richard Morris, Morris Analytical X-ray Ltd**, who described his work to evaluate soil from excavated brown-field sites for use as top-soil. Richard was true to form in giving his talk the intriguing title *Where there's muck there's brass and iron and lead and chromium...* He first described how concrete and metals were manually removed from mixed loads of up to 40 tons deposited for processing with the remainder screened using a 30 ton mechanical vibrator and electromagnet to generate coarse aggregate and the "top-soil". This work supports the DEFRA 2005 initiative on the built environment to work towards sustainability, in this case, in the West Midlands. Richard devised a sampling strategy for the "top-soil", taking 5 kg lots to the laboratory and using BS 3882:2007, and designed an analytical method to satisfy the legislation for the determination of As, Cd, Cr, inorganic Hg, Ni, Pb & Se. A typical analytical report also included a host of physical properties such as visual inspection, pH, electrical conductivity, particle size analysis, total organic matter and extractible P, K & Mg.

Techniques to support this work include XRF, ICP, GC-MS and NMR. Pressed powder pellets were prepared using 8.5g of specimen + 1.5 g of Carbowax for XRF analysis using a Spectro XEPOS EDS bench-top spectrometer. The results were assessed using EA CLR guidelines to show that the process "top-soil" was almost within specification and therefore needed to be diluted with less contaminated

soil before being considered safe for use as domestic soil. As is often the case, Richard proved that a more systematic sampling plan was needed to ensure that the analytical data could be used to support a pricing strategy for the weekly processing of Black Country Muck.

David then introduced **Ian Croudace** from the **National Oceanography Centre** in Southampton who described his Itrax micro-XRF core scanner, just what the environmental sciences needed. This wondrous device is a multi-functional high-resolution XRF instrument that can measure element availability from Al to U with concentrations from trace to major levels from step-sizes as small as 200 microns. X-rays are generated from a 3 kW Mo target tube and focused through a flat capillary waveguide with detection by the increasingly popular high resolution SDD device. X-ray dwell time (for X-radiography), counting time (for XRF) and measurement step size were all user definable. The equipment combined simultaneous high-resolution optical and X-radiographic images with geochemical data to investigate core sediments for marine research institutions.

The Itrax was first proposed by Ian in 2001 and has now been installed in 15 labs worldwide. It can cope with samples up to 1.75 m in length and benefits from bespoke "Navigator" software. Examples of the impressive output were shown for not only marine, estuarine and lake sediments but also wood-cores and cave deposits (speleothems). Results were validated by conventional WD-XRF proving the benefit that this innovative device can offer environmental scientists.

Margaret West, West X-ray Solutions Ltd

General Applications



Speakers - Left to Right: Yoshiyuki Kataoka, Simon FitzGerald, David Beveridge (chair), Stephen Davies.

Calibration Maintenance: Food for Thought

Stephen Davies, PANalytical

Sources and causes of instrument drift were discussed-

such as tubes, detectors and crystals. By far the largest cause of drift is the x-ray tube which deteriorates over time as tungsten coats the inside of the beryllium window. This effect is accentuated at high powers and a loss of intensity results in poor analytical precision. Some GLP schemes suggest that if drift of more than 10% is observed then a brand new calibration is required so clearly controlling drift is essential to the effective operation of XRF systems.

Tests were made at Manchester University for 7 elements using an XRF at 4kW. The current was reduced over several measurements for 2 separate calibrations - drift corrected and non-corrected. It was noted that drift is by far the largest source of error contributing to analytical problems and if it is not corrected for then other instrument problems can be exacerbated. Compared to drift, the problems associated with inter-element corrections are insignificant.

The 10 Micron Innovation - Applications in Micro-XRF

Simon Fitzgerald, HORIBA Jobin Yvon Ltd

Horiba manufacture a lot of different instruments and traditionally their XRF units have been for bulk sample analysis. New systems now make mapping with Micro-XRF possible. A parallel x-ray beam is vital for this and 3 types are available. An aperture is a low cost option with low intensities, a mono-cap gives higher intensities at medium cost and a poly cap is best of all for intensities but is the most expensive.

SLICE software (Spectral Library Identification and Classification Explorer) allows a match of produced x-ray spectra to a library of many material types. This is utilised by the FBI. Applications of this include analysis of gemstones, glass, gunshot residue, paint, artefacts, electronics and geological samples.

Micro Spot analysis of electronic components using a polycapillary lens in standard WD-XRF

Yoshiyuki Kataoka, Rigaku, Japan

Micro spot technology in a WD-XRF yields high intensities, including light element performance, from a spot size of 100 microns. The spectrometer has a 4KW rhodium tube, sample masks, 10 crystals, 2 cameras, 2 detectors and the polycap system. The unit can be optimised for heavy elements with 50-80 micron spatial resolution. The design means that the spectrometer can be used as a regular WDX spectrometer - but have the additional functionality of the polycapillary technology.

Applications presented were the point analysis of electric terminals and polymers, thickness determination of multilayer electrodes and the measurement of the composition of a coated piece of Indium Tin Oxide.

Andy Scothern, Saint-Gobain Gyproc

Method Validation



Speakers - Left to Right: Ros Schwarz (chair), Debra Schofield, Eddie Birch, David Lowe, Paul Thomas.

KEYNOTE LECTURE: Accreditation and Method Validation

David Lowe, United Kingdom Accreditation Service (UKAS)

UKAS is the UK's national accreditation body and has over 150 employees. Accreditation gives confidence to customers of laboratory services and recognition to the laboratory. Accreditation standards are all based on ISO9001, and IS17025 is used for laboratory accreditation. This covers management and systems such as record keeping and document control. Technical requirements like training, equipment, sampling and traceability are also covered.

Validation means that a method is 'fit for use' in terms of precision, bias, uncertainty, range and detection limits. Proficiency testing and use of Certified Reference Materials is also covered. In this sense XRF is not different to any other technique.

Method Validation - Working Our Way Through the ASTM Process

Debra Schofield, Oxford Instruments Analytical Ltd

Producing a new ASTM method is a 2 year process and is driven by people with sufficient time and money. A draft method is submitted to ASTM that shows a need for a technological breakthrough. Round Robin analyses are carried out to test the suitability of the new method. Participants and test samples are required and the whole process takes 6 months and is very loosely controlled.

Statistics are carried out on the Round Robin data and ballots are set up for members to accept or decline the new method. There are four types of votes and the procedure can be quite controversial.

A Practical Approach to ISO17025 Accreditation

Eddie Birch, CIQ Audit

Further advantages of accreditation include insurance claims, legal defence, efficiency and training. Carrying out a 'gap analysis' is a good plan for accreditation - what is done well, badly and what is missing. A Quality Manual should be produced that defines roles and policies and ensures that standard methods are followed for which proof is available.

ISO17025 calls for 18 sets of records and demonstrable staff training is important. UKAS should be booked 2 months before the accreditation is due.

Statistical Analysis and Associated Work Flows in Quantitative Measurement Operations

Prof. **Paul Thomas**, University of Loughborough

The Centre for Analytical Science at Loughborough University stipulates that for analytical data to be meaningful it has to be understood by an 'educated 14 year old'. This means that all data should be presented with rigour and pride and this is the basis of a 100 hour post graduate training program.

Precision is best reported through the use of Confidence Limits and indeed the use of language in statistics is vital if we are to express the meaning of our data in a suitable manner. An advanced level in MS Excel should be attained and the purchase of the Pro-XL software package was recommended.

Andy Scothern, Saint-Gobain Gyproc

XRF Session Hand Held Applications Workshop



Speakers - Left to Right: Chris Calam, Mike Dobby, John Hurley, Debra Schofield, Richard Kilworth, Margaret West (chair), Steve Allott.

THE workshop began with an overview by the chair **Margaret West** of the use of hand held instruments. She covered the regulations involved in the safe working practice of these devices, the 1999 Ionising radiation regulations (mainly section 16), risk assessments and registration with the HSE. Also covered was the use of either a radioactive source or the now more common low wattage miniature x-ray tube typical of modern instruments. Miniaturisation of the components for these devices over recent years has transformed the older “luggable” instruments into the modern truly hand held and portable devices. Silicon drift detectors (SDD) are now the detector of choice with their higher resolution and lower Z detection over the earlier PIN devices. They often work with a Personal Digital Assistant (PDA) running Windows mobile and bespoke software to cope with matrix effects, line overlaps, quantitative or qualitative analysis with pre-calibration available.

These small hand held devices allow *In situ* analysis away from the confines of the laboratory. The instrument is placed in contact with the sample and analytical results are immediately available to the operator allowing a value judgement on where to measure next. With no sample preparation only the surface is measured with the bulk inaccessible and penetration limited by critical depth - a few microns for low Z elements and 1-2 mm for the K lines of high Z elements. The obvious problems from surface measurements are: patinas, contamination, weathering and moisture in soils. Applications include: contaminated land, workplace monitoring, particulates on filters, surfaces and coatings, metal and alloy sorting, architectural buildings and monuments, geochemical prospecting, archaeological investigations, works of art and cultural heritage.

Margaret's presentation was followed by live demonstrations of the “helping hands” from four hand held manufacturers in an area at the front of the lecture theatre that ensured that delegates were at least 2 metres from the working area. These were in order, Spectro, Oxford Instruments, Bruker AXS and Thermo Niton. The team and their instruments are shown in the above photograph. They covered a range of topics in their demonstrations including safe use, plastics, soils, metals and many more.

Dave Taylor, ICDD

XRF Session Portable Instruments

Portable X-Ray Fluorescence Analysis - New Opportunities, New Challenges -

Phil Potts, Open University

Phil used a portable “luggable” X-Ray spectrometer



Speakers - Left to Right: Deborah Cane, Duncan Starke, Margaret West (chair), Phil Potts

(Spectrace TN9000), to analyse geochemical samples including a rock outcrop in the Preseli Mountains in North Wales. It was advantageous to use a portable X-Ray system to analyse this outcrop because conventional sampling is not permitted, as removal of rock material is illegal. Factors that affected the analysis were weathering and contamination of the rock samples with lichen and moss. These rocks were used to form the inner circle at Stonehenge.

A project he also worked on was determining the provenance of Neolithic stone axes found in various locations around the UK to determine which had originated from the Preseli Mountains in North Wales; as an outcome of his research half the axes found and previously attributed to the Preseli Mountains were determined to have originated elsewhere when non destructive X-Ray analysis was conducted.

Penguins & Precious Metals - the use of Portable XRF at Birmingham Museum & Art Gallery

Deborah Cane & Duncan Starke,
Birmingham Museum & Art Gallery

Deborah and Duncan gave a two-part presentation on the use of a Bruker portable XRF at Birmingham Museum & Art Gallery.

First up was Deborah who uses the portable XRF to analyse samples from the natural history collection for the presence of toxic metals (incl. lead, arsenic and mercury) that may have been used in the taxidermy process. This is of particular importance because the exhibits may be housed in an area where they can come into contact with visitors who may become contaminated with these toxic metals if exhibits are touched. Exhibits that contain these metals are only placed where they cannot come into contact with visitors.

Further work that Deborah carries out includes determining the provenance of artefacts including Egyptian bronze statues where simple non-destructive analysis helps determine fakes compared to the genuine artefacts. Secondly Duncan gave a presentation on how he uses the portable XRF in administering the “Treasure Act 1996” to facilitate the rapid identification of an item's composition, which has been found by people using metal detectors.

This is of importance because historically any item that had been found required sending to the British Museum for formal identification. This process is not quick, and the delay could annoy the finder and thus work against future cooperation with the system. Examples of items he has checked include medieval annular brooches, Iron Age torcs and Roman coins.

Darren Musgrove, HSL

XRF Session New Developments in Instrumentation and TXRF



Speakers - Left to Right: Margaret West (chair), Christina Strel, Malcolm Haigh, Armin Gross

Developments in TXRF Analysis

Christina Strel, TU Wien, Atominstitut der Österreichischen Universitäten

A rapid overview of the applications of TXRF was presented, in particular the benefits of the technique, such as small required sample size. Techniques of increasing signal and optimising output were described, explaining how the technique allows the detector to be closer to the sample. A brief description was given of how to determine whether the point of interest was within or on the surface of a sample. This is done by using variations in the incident angle of x-rays.

Further slides were used to present Christina's current research, particularly using synchrotron x-rays as a source, which gives distinct benefits over a standard x-ray tube. Rays are significantly more collimated, with higher power, reducing noise to increase detection limits of the light elements; but also synchrotron rays open up the possibility of exciting heavy element K-lines. Synchrotron radiation was used in parallel with absorbance spectroscopy for this work, giving additional information, such as the ability to determine the valence state of arsenic within the sample.

The talk finished with a summary of an advanced use of TXRF to map the location of contamination on silica

wafers, whilst at the same time analysing whether sample compositions are within specification limits. This is potentially a very useful technique in the electronics industry.

Trace Element Analysis of Pharmacological, Medical and Biological Samples by TXRF

Armin Gross, Bruker AXS Microanalysis GmbH

Armin gave a background to TXRF, and its application as a technique for analysis of liquids, powders, suspensions and thin films. Particular reference was made to the ability to analyse very small sample quantities through use of deposited thin films. These thin films give negligible matrix effects due to their lack of thickness, making quantification possible by using standard dopant elements without the need for further calibration. TXRF applied in this way can detect elements from Na-U.

Discussion then continued with an introduction to the PicoFox bench-top TXRF. Armin highlighted the very short time to results achievable using this technique. Applications of bench-top TXRF were discussed, including analysis of trace elements in pharmaceutical and food applications, as well as the ability to look for Cu, Fe, Zn and Se in blood samples. The key application benefit was the lack of need for specialist sample treatment, and the ability to calibrate measurements by adding only 1 standard calibration material to the samples. A case study was presented in which 50µg of sample were analysed from the organs of a mouse, without the need for an acid digestion step.

Energy Dispersive XRF - its Diversity and Capabilities

Malcolm Haigh, Spectro Analytical UK Ltd

Malcolm presented a series of slides to highlight the diversity of EDXRF as an analysis technique. Techniques, applications and typical uses of the main types of EDXRF, e.g. Low Resolution, Direct Excitation EDXRF were presented. These were linked to real situations and markets where the techniques were suitable.

An in-depth discussion of the benefits of polarised EDXRF was then presented, in which significant improvements in background noise and trace element detection were presented. The talk finished with a brief overview of the applications of Micro-XRF, including capillary optics, for use in situations such as RosH analysis. All three talks gave an excellent portrayal of recent advances in EDXRF technology and its consequent application to novel and interesting scientific challenges. The ability to analyse new materials, both through sampling and in-situ presents an exciting future for XRF. A particular point that became clear is the ability of XRF to provide a suitable alternative to trace-element analysis by ICP, a very exciting prospect indeed!

Daniel Capon/Henry Foxhall, Glass Technology Services

The Bragg Lecture

Report of the Bragg Lecture Fund (BLF) Committee Chairman Prof J R Helliwell for the period 2002 to 2007 inclusive (6 years)

I offer here my report here in the BCA Newsletter as outgoing Chairman of the Bragg Lecture Fund Committee.

The Membership of the Committee in my period (basically a member drawn each from where Sir William Henry and Sir William Lawrence Bragg had worked) was:-

J R Helliwell (Chair) (Manchester);
John Davies (Cambridge)
Simon Phillips (Leeds)
Richard Catlow (London & The RI)
Pam Thomas (Secy) 2002 to 2003

The Chair of the Committee immediately before me was Prof **A C T North** (Leeds) and my successor is Prof **S E V Phillips** (Leeds and now Diamond Light Source).

We inherited the selection, but organised the speaker and venue practical details for:-
2002 **Dave Stuart** (biology) (The RI and Reading BCA).

We consulted closely by email and successively selected the following Bragg Lecturers:-
2005 **John Finney** (physics) (The RI and UMIST BCA);
and 2007 **Sir Roger Penrose** (maths) (The RI and Canterbury BCA).

An attempt to achieve a full coverage of topic areas is emphasised here, i.e. topic area given in brackets.

The expenses of the Bragg Lecturer were all handled by The Royal Institution of Great Britain (RIGB) who administer the Bragg Lecture Fund as per Rule 5 of the Bragg Lecture Fund Constitution and Rules. The current assets as of 30/9/2008 held by the RIGB are £14419; these are included in the RIGB's 'Main Charity Fund', for ease of auditing, and managed by the RIGB for us as a 'Restricted Fund'.
Other practical details:-

The Lecturer generally does two presentations, one always at The RIGB. In my term of office I preferred one of these presentations to always be at the BCA Spring meeting.

Thus **Dave Stuart** lectured at the RIGB and the BCA held in Reading.

John Finney lectured at the RIGB and at the BCA held at UMIST.

Sir **Roger Penrose** Lectured at the BCA held at the University of Kent in Canterbury and at the RIGB.

The RIGB handles the speakers' expenses claims (both presentations ie not only the one at the RIGB).

Each speaker of the above three speakers has an inscribed glass trophy. [In the immediate past a goblet had been presented.]

Overall the list of Bragg Lecturers has thereby been successfully continued and is therefore (in full):-

- 1962 **Paul P Ewald** (Leeds & RI)
- 1965 **Dame Kathleen Lonsdale** (Melbourne, Adelaide & Perth)
- 1968 **Dorothy Hodgkin** (Manchester)
- 1970 **B E Warren** (RI)
- 1973 **R W G Wyckoff** (Cambridge)
- 1981 **Henry Lipson** (RI & Leeds)
- 1982 **Michael M Woolfson** (Manchester & Cambridge)
- 1985 **Sir David Phillips** (Leeds & RI)
- 1987 **Brian W Matthews** (Perth & Adelaide)
- 1993 **Sir Gordon Cox** and **Max Perutz** (Manchester BCA and The RI)
- 1994 **A M Glazer** (Newcastle BCA and The RI)
- 1996 **K C Holmes** (Cambridge BCA and The RI)
- 1997 **Durward W J Cruickshank** (The RI and Leeds BCA)
- 1999 **Jack Dunitz** (chemistry) (The RI and IUCr Glasgow Congress)
- 2002 **Dave Stuart** (biology) (The RI and Reading BCA)
- 2005 **John Finney** (physics) (The RI and UMIST BCA)
- 2007 **Roger Penrose** (maths) (The RI and Canterbury BCA)

I wish to acknowledge all my Bragg Lecture Fund Committee colleagues, the RIGB and of course the Bragg Lecturers themselves during my term of office; Prof Dave Stuart, Prof John Finney and Sir Roger Penrose.

John R Helliwell DSc Professor of Structural Chemistry, University of Manchester, M13 9PL

YC Meeting Report

THE Spring Meeting of the British Crystallographic Association 2009 was held in Loughborough and was based around the theme of Dynamic Crystallography. As in previous years the Young Crystallographers (YC) meeting started proceedings with their Satellite on 20th - 21st April. The range of research presented, highlighted the diverse range of applications and subject areas where crystallography can be applied, and the following is a collection of impressions from this meeting.

The first plenary talk seemed to come from outer space as **Dominic Fortes** introduced the world of ammonia hydrates on Titan, the largest moon of Saturn. Crystallography finally appeared as a fully integrated method for his study, with a few years spent analyzing diffraction patterns.

The first 'real' Young Crystallographer up was **Edward Bilbe** who gave a very interesting talk on 1-D mixed metal systems using cyanide ligands. He explained how information about the order (and disorder) of metals and cyanides was obtained using total neutron diffraction and how to utilize pair correlation functions.

This was followed by **Karkthik Paithankar** who showed the advantages of using multiple single crystals to improve data collection of proteins, a technique which will prove invaluable when dealing with temperamental proteins such as membranous proteins where growing a large single crystal is incredibly difficult.

Craig Robertson presented a lot of results from his research of sulphur and selenium containing radicals. These molecular compounds are particularly interesting for the development of data storage devices due to their magnetic properties.

The next presentation by **Jeppe Christensen** focused on phase diagrams of binary intermetallics, in particular the Sn-Sb system. Jeppe showed how incommensurate modulation could be used to clarify the presence of various structure types in the Sn-Sb phase space.

Arefeh Seyedarabi's talk on her "naughty" protein from a pathogenic bacterium caught the audience's attention as she gave a complete overview of the protein role and structure, emphasizing the importance of domain arrangement for protein biological functions.

The first session was concluded by **James Haestier**, who discussed the effects of unit cell standard uncertainties on derived parameters such as atomic positions. James

outlined how the calculation of errors could be improved by a combined variance-covariance matrix, but also pointed out that there is a lack of uniformity when it comes to determining the standard uncertainties on the unit cell parameters.

The second session started with a plenary lecture from **John Helliwell**, which gave a very scientific answer to a general public question on "why does a lobster change colour on cooking". More importantly it illustrated the advantages of studying protein complexes with carotenoid molecules by advanced macromolecular crystallography techniques using longer wavelength X-ray and neutron diffraction.

David Millar then talked about the phase transformations under varying temperature and pressure conditions of the explosive material RDX. Studying the structural changes carefully enabled the identification of a new high-temperature, high-pressure polymorphic form, which had previously been mistaken to be the same as the β -form.

Ivan Campeotto presented some work, which would become topical with the crystal structure of a mutant enzyme involved in the discovery of sialic acid analogues, which are the main anti-viral drugs against Influenza virus. The substrate bound protein structures highlighted the residues involved in the catalysis allowing a better understanding of the mechanism and specificity of the enzyme.

Next came **Kirsten Christensen** and her talk on open-framework germanates. Kirsten showed how she had used different sized germanate polyhedra to generate clusters that could be used as building blocks to form porous materials. Two novel germanate frameworks were found that contained a mixture of clusters of different types.

Adam Cowell, the last speaker of the day, enthusiastically talked about co-crystals and salts and the issues that arise if only powders are available. The combination of powder diffraction with other techniques, including a computational evolution algorithm, elegantly resolved whether structure solution from powders can be obtained for multi-component systems: Yes, we can!

A break was welcome as the day was flying by with very high quality presentations. The YCG meeting went smoothly and illustrated the good organization throughout the day. The poster flash presentation was fairly relaxed even though it took a bit of thinking to sell one's poster to a wide audience. The evening poster session was a good time

to catch up with fellow crystallographers, and exchange thoughts about each other's work.

The last YC session on Tuesday morning was a themed session to honour Dr **Andy Parkin**'s contributions to the Young Crystallographers Group and was mainly focused on chemical crystallography to reflect Andy's research interests.

The plenary speaker was **Frank Allen** and his talk "Energy Matters!" demonstrated how energy calculations are useful in understanding intermolecular interactions. In one example it was rationalised how the strength of a hydrogen bond depends on the D-H...A angle.

Next was an excellent talk on Metal Organic Frameworks given by **Anna Stevenson**. This covered the exciting field of porous MOF chemistry giving details on newly synthesised MOFs with varying pore sizes and the significance of the increase of these on gas adsorption. The talk was a deserving winner of the IG Young Crystallographers prize, and it was presented for a second time on the Wednesday during the IG session.

Andras Kallay's talk on co-crystals, which are formed between benzoic acid derivatives and the DMAN proton sponge, contained a lot of nice Fourier difference maps that showed the electron density of the hydrogen atom involved in hydrogen bonding between the two chemical systems. Most remarkably the complex of ortho-chlorobenzoic acid and DMAN crystallizes in a 1:1 ratio, but with one protonated and one neutral DMAN molecule in the asymmetric unit (ASU), whereas all other halogen analogues crystallize with 2 benzoic acid molecules per DMAN molecule in the ASU.

The next presentation by **Samantha Callear** looked at the salt and co-crystal formation when reacting pyridine derivatives with dicarboxylic acids. The crystal structures were compared showing similar packing and intermolecular interactions dependent on the chain length of the dicarboxylic acids.

Graham Findlay then introduced cluster analysis and the dSNAP algorithm and explained how dSNAP can be used to cluster results from a CSD substructure search into groups of similar geometrical parameters. From the clustering conclusions can be drawn as to the effect of substituents on the molecular geometry.

This was followed by **Graham Tizzard** and the introduction of another program that assesses similarity, XPac. Graham had used this program to analyse the crystal structures of 40 disubstituted chalcogens and despite the lack of any strong intermolecular interactions the majority of structures contained the same row of molecules.

The final speaker of the YC meeting was **Craig Martin** and he presented his work on co-crystals of benzimidazole with the aim of understanding the mechanism behind co-crystal formation. A combination of diffraction techniques and

Differential Scanning Calorimetry was used to investigate any polymorphism in the co-crystals due to different solvents and varying temperatures.

The session was concluded by session chair **Suzanne Buttar** with some final remarks and some pictures of Andy Parkin, and with that another successful YC meeting came to its end. It was generally perceived as a good experience to share results and hear of the latest in crystallography.

James Holcroft (University of Newcastle), **Claire Murray** (University of Reading), **Geoffrey Masuyer** (University of Bath), **Susanne Huth** (University of Southampton)



Biological Perspective

A Biological Crystallographer's Perspective

TUESDAY'S introduction began with the Lonsdale lecture delivered by David Watkin. The talk was based around the progression of crystallography from its very beginnings in 1664 with speculations over crystal formations and ordered groups; through to the modern day including calculations by hand, the introduction of the electronic calculator and the impact of computers on structure solving. The difference in time between structure solving then and now is quite remarkable.

I attended the BSG meetings that afternoon where **Clemens Schultze-Briese** spoke about new beamlines at the SLS Swiss Light Source which opened in 2008, and their successes so far in 'protein crystallography with 6 million detectors'. **Robin Owen** took over the session on "New synchrotron instrumentation" to discuss spectroscopic techniques used on beamlines. These included UV-vis and Raman spectroscopies, which are being encouraged for use due to the extra information they reveal about the protein in the crystal. The session was wrapped up by **Armin Wagner** who gave a very interesting talk regarding the moving of microcrystals by laser through solution carefully avoiding other crystals in a drop and mounting on a grid.

Wednesday's session on "Reactions in macromolecular crystals" was kicked off by **Andrea Mozzarelli** whose talk drew attention to the lack of protein structures tested for dynamics and function whilst in the crystalline state, and supported the idea of spectroscopic methods being applied while at the beamline. The aim here was to point out differences between function of proteins in crystals and in solution. The following two talks were both regarding dioxygenases, the first by **Elena Kovaleva** about understanding a reaction cycle by trapping and studying the intermediate protein structures. **Andrea Hadfield's** talk also demonstrated the structure of intermediates in a different pathway.

After lunch came another batch of talks this time regarding "Metalloproteins: structure and dynamics". **Paul Ortiz de Montellano** started the session with cytochrome p450 enzymes, mentioning the importance of UV-vis techniques in aiding determination of drug-enzyme complexes. This was followed by an interesting talk by **Emma Raven** about heme containing proteins, in particular the peroxidases, focussing on the movement of histidine interaction with

the iron effecting the on/off function of the enzyme. **Mark Banfield** finished off with his talk about the structure and function studies of a metal binding loop in the electron transfer protein azurin.

The third BSG session of the day continued after a short break with "Dynamics at the membrane" with **David Stuart** starting off discussing membrane proteins in viruses, including fusion machines over evolution. The session was carried on by **Alex Cameron** describing transporters in the membrane of *Microbacterium liquefaciens*, and **Steve Prince** followed on the theme with his talk about structural studies on ion channels, which due to their being difficult to crystallize have been studied by electron microscopy and small angle scattering. This was quickly followed by the BSG Plenary lecture by **Venki Ramankrishnan** on the ribosome structure throughout translation, which highlighted recognition factor function.

The last day of the BCA Spring meeting consisted of two BSG sessions. The first, on the "dynamics of informative radiation damage" was started off with an interesting talk by **Colin Nave** about how damage occurs, what it means for interpreting the data, and that using cryotemperatures doesn't prevent specific damage and highlighted that metal containing proteins are particularly susceptible. **Martin Weik** followed on with controlling damage by controlling temperature where temperature dependence can alter the protein, solvent and water molecule flexibility. Neutron scattering was presented as a complementary technique to crystallography. **John McGeehan** gave a particularly notable presentation about the apparent difference in radiation damage between proteins and DNA. The original idea being to test the robustness of the DNA molecule, a hierarchy was found in the sites which are damaged; and in DNA/protein complexes, the protein has much more specific damage than the DNA. The application of Raman spectroscopy was also highlighted here.

The last session of the day about "snapshots of dynamic processes" was started by **Richard Lewis** about the stressosome of *Bacillus subtilis* and its structure determination through a combination of x-ray crystallography and cryo-EM. **Jasper van Thor** wrapped up the meeting with time-resolved structure solutions. It was then time to pack up the poster and return to the car for the trip back home in the sunshine!

Jenna Bailey
Keele University

Physical Crystallography

Physical Crystallography Lectures

THURSDAY was the main PCG day, starting with a plenary teaching lecture presented by **Martin Dove** who gave an insight into how combining the Bragg diffraction with the background of diffuse scattering using reverse Monte Carlo methods provides information regarding the dynamics of atoms. In the following Hydrogen Storage session a range of materials were discussed. **Martin Jones** demonstrated the utility of the Intelligent Gravimetric Analyzer for Neutrons which, as the name suggests, allows thermogravimetric and neutron diffraction measurements to be performed simultaneously thus enabling full characterisation of the hydrogen and dehydrogenation profile of hydrogen storage materials. Following this, **Andrea Baldi** talked about thin films comprising multiple layers of Mg and Ti which showed interesting optical and electrical properties depending upon the level of hydrogenation. **Neal Skipper** concluded the session with a presentation on hydrogen delocalisation in KC8 and KC24 graphite intercalators.

Samantha Callear

IN the Dynamics in Framework Structures session, different ways of studying flexible frameworks and their properties were presented with a focus on Metal-Organic Frameworks. Two of the speakers focussed on the “breathing” properties of MIL-53, where the unit-cell changed with incorporation of different organic guest species. **Richard Walton** was trying to understand the reactivity of Fe-MIL-53 using time-resolved diffraction. He could determine that some of the liquid-phase exchange

is taking place via intermediate phases, but overall it is a subtle balance in energetics that gives the change. **Robert G. Bell** was doing calculations on Cr-MIL-53 using DFT- and Force-field calculations in order to understand the CO₂ uptake. Whether the framework is closing or not is highly dependent on guest molecules as there are several minima in configurational energy in the breathing MOF. **Ashleigh Fletcher** presented a study of adsorption in flexible structures such as MOF's. This is a way to understand the kinetics in these types of structures. Using the same experimental techniques as for a classical adsorbent it was possible for Ashleigh Fletcher to show that a structural change can be seen in the isotherms.

Kirsten Christensen

IN the Crystallography Near the Edge session, **M. Helliwell** showed how fast and reliable wavelength tuning at synchrotron beamlines allow for the collection of multiple wavelength data sets on a given sample. It was demonstrated how tuning the wavelength to the absorption edge of each element present in the sample can resolve the ambiguity of occupancy of a crystallographic site, when the elements present are neighbours in the periodic table. In the following talk by **G. Sankar**, the anomalous dispersion experiments were combined with EXAFS and entered into a simultaneous refinement. This new method allowed for the investigations into how catalytically active Co atoms are distributed in a host aluminophosphate lattice. The final talk of the session, given by **A. J. Davenport**, was on studies of how corrosion at a metal surface occurs. X-ray diffraction from the salt film formed at the solution – metal interface gave the surprising discovery of sublayers of different hydration states within the salt film.

Jeppe Christensen

Puzzle Corner... ...JUNE ANSWER

ANSWERS to June questions: what is their radiation, and where are they?

ALBA - synchrotron - Vallés, Spain

ANKA - synchrotron - Karlsruhe, Germany

BACH - synchrotron - ELETTRA, Trieste, Italy

BEAR - synchrotron - ELETTRA, Trieste, Italy

GEM - neutrons - ISIS, RAL, UK

HANARO - neutrons - KAERI, Korea

MANDI - neutrons - SNS, Oak Ridge, USA

MUSTANG - synchrotron - BESSY, Berlin, Germany

NIMROD - neutrons - ISIS, RAL, UK

OPAL - neutrons - ANSTO, Lucas Heights, Australia

ORIENT EXPRESS - neutrons - ILL, Grenoble, France

PEARL - neutrons - ISIS, RAL, UK

SALSA - neutrons - ILL, Grenoble, France

SESAME - synchrotron - Allaan, Jordan

SNAP - neutrons - SNS, Oak Ridge, USA

TOMCAT - synchrotron - SLS, Villigen, Switzerland

VESUVIO - neutrons - ISIS, RAL, UK

VIVALDI - neutrons - ILL, Grenoble, France

Chemical Crystallography

Chemical Crystallography Lectures

Computational Crystallography (CCG/PCG)

Chair: **Richard Cooper** (oXray)

THE Computational Crystallography session began with a talk by **Frank Leusen** (IPI) on *A Breakthrough in Crystal Structure Prediction*. He discussed the various successes and failures since a regular set of blind crystal structure prediction challenges began in 1999, giving some insight into the challenges of structure prediction, specifically the number of degrees of freedom and energy calculations. The methodology leading to a successful set of predictions by Leusen *et al* in 2007 was reported, the crucial part being a hybrid Molecular Mechanics / Density Functional Theory computation to provide reliable stability ranking of the many generated structures. This breakthrough resulted in the group correctly predicting (as the top candidate in each case) all four blind test structures. Future challenges, in addition to maintaining this level of success, include predicting polymorph stability, dealing with salts and solvates and very large, flexible molecules. Next, **Mustapha Sadki** (Oxford) gave a detailed talk about a *New Framework for Reliable Refinement Data Types* in which he described the frequently encountered difficulty of extending and scaling existing crystallographic software due to the close coupling of the least squares refinement (or other data fitting methods) with domain specific models (in this case the crystallographic model). As the antidote to this problem, he presented a framework which separates the refinement mathematics from the crystallographic model. This approach has two key benefits: firstly the model can be easily extended and enhanced, particularly by generating derivatives using automatic differentiation, allowing novel restraints and constraints to be easily tested and implemented; and secondly, minimisation algorithms which have been rigorously developed and supported by a wide mathematical community, can be plugged in and tested with ease. Some animations of the convergence of refinements using the framework and a standard crystal structure were shown. The final talk was by **Philippe Aeberhard**, a DPhil student from Oxford, entitled *Exploring Hydrogen Storage Materials using Density Functional Theory Calculations*. After an overview of the factors driving research in hydrogen storage, he went on to present results from a system where quantum chemical calculations had enhanced the understanding of the structure of a hydrogen storage material, and which had subsequently been verified by additional experimental data.

Reactivity in Crystals

REACTIVITY IN CRYSTALS I

THE Wednesday morning session with the theme 'Reactivity in Crystals', chaired by **Andrew Bond**, began with a talk by **Marc Messerschmidt** entitled *'Time Resolved Diffraction Studies on Tetrathiofulvalene-P-Chloranil – New Aspects From Polychromatic Experiments'*. Over the next half hour, Marc described to us how a photo-excited state TTF-CA was obtained at the ESRF using a polycrystalline sample for Laue diffraction experiments. The data collected from this sample were free from the issue of time delay in the change of diffraction intensities – a problem which is significant in larger crystals. Use of the full X-ray flux of the beam negated the potential problems arising from a small sample size and allowed for fast data collection with sufficient I values. Using this approach, structural changes in the crystals could be identified within 50 ps and from these data, a structure with respectable R-values was attained.

Stephen Moggach continued the session with an informative talk on crystal responses at elevated pressures, with the aptly titled *'Reactivity in Crystals at High Pressure'*. He started by relating the pressure regime being considered in his research (up to 10 GPa) with other levels of pressure that the audience perhaps had a better grasp of, such as those found in deep sea trenches and, of course, the processing pressure of guacamole. A brief overview of the available instrumentation followed this before the main body of the talk which illustrated how pressure can be used to alter the geometry of various inorganic complexes. One such study on a copper-containing complex demonstrated the effect of compressive forces exerted on a Jahn-Teller distorted bond and how its geometry, symmetry and copper-coordination environment changed, following phase transitions to two additional new polymorphs.

Stefanie Schiffrers rounded off the first half of the 'Reactivity in Crystals' session with her talk, *'Crystal Engineering and Reactions in the Solid State'*. Stefanie focussed the subject of her talk toward her research on photo-excited [2+2] cycloaddition reactions. Some of the limiting factors as to whether the reaction will take place were described, including the fact that the reaction takes place between nearest neighbours or that the molecules need to be oriented such that pairs of double bonds have a parallel alignment. In order to encourage, or engineer, the latter of these criteria to occur, an NCS- ligand was employed as a 'steering agent' for use with an array of various metal-halogen complexes. For a cobalt containing complex, it was shown that there are many photoreactions that can take place – resulting in many different structural possibilities. It was also shown that other anions can be used in place of the NCS- ligand to steer double bonds into alignment.

REACTIVITY IN CRYSTALS II

ALEX GRIFFIN opened the second half of the 'Reactivity in Crystals' session by introducing the first speaker, **Lee Brammer**. Lee's talk, '*Flexibility and Dynamics in Metal-Organic Materials*' provided an extremely clear insight into the general characteristics of framework materials and his research within this field. He began by outlining the 'node and spacer approach' and how this results either in 2-dimensional planar grids or a full 3-dimensional twisted array. Lee presented some examples of his work to us including a paddlewheel MOF, consisting of zinc centres and camphorate ligands. One particular experiment with this MOF involved measurement of the uptake of DABCO and ethanol over a 24 hour period. A second example described to us was a silver(I) perfluorocarboxylate complex which possessed no pores. He showed that alcohol could be inserted into this complex between carboxylate moieties and stripped out again at increased temperatures. The final example in the presentation was another paddlewheel-type MOF where ethanol could be reversibly bound in the MOF cavities.

Ann Chippindale gave the penultimate talk of the session, '*Bending, Twisting, Breaking and Shifting: Structural Transformations in Metal Cyanide and Phosphate Systems*', in which she described her work on several inorganic complexes. The first of these systems was a TiGaPO framework which underwent a temperature-induced phase transition from an octahedrally coordinated, hydrated material to a tetrahedrally coordinated, dehydrated material. This structural alteration was found to be reversible on cooling where water molecules were taken up again. Using thermogravimetric analysis, a second system - $K_2NiCu_2(CN)_6 \cdot 2H_2O$ dehydrated at 315 K and underwent a phase transition to a new single crystal polymorph. Another thermogravimetric analysis, complemented

with Rietveld refinement of $Ni(CN)_2 \cdot nH_2O$ showed that 'buckling' of layers in the crystal was greatly dependent on the value of n . A fourth study using combined EXAFS and X-ray data on a nickel cyanide species illustrated its potential as a molecular switch as hydration/dehydration modified the electronic configuration of the complex as it moved between high and low spin states. The final example Ann presented us with were chains of CsCuAg cyanides. New polymorphs were obtained when left in a bomb for a few months and these transitions were shown to be reversible upon heating.

The closing talk of the session was the CCDC prize lecture, given by **Hazel Sparkes** who presented her research to on, '*Exploiting Charge Density – Insights Into Structure, Bonding and Reactivity*'. The first part to Hazel's talk showed the complexities of modelling bond electron density. Identification of the shape of the electron density around a bond combined with knowing the position of the bond critical point enables elucidation of the bond type, i.e. covalent or ionic. To adequately model the electron density, there are experimental requirements including, the need for a high resolution and intensity, low temperature and preferably a high symmetry crystal. She then went on to show us how this analytical technique has been applied to [2+2] cycloaddition reactions. In the second part of her talk, Hazel demonstrated the application of the technique to rhodium complexes – systems that are commonly used for carbon-carbon bond activation. The charge locations of the individual atoms in the complex were deduced; thus the bond path and bond critical points could be identified. The results of this study indicated that the bonding character associated with the rhodium atom is partially ionic and partially covalent.

Nick Funnell
University of Edinburgh

C P Snow and Crystallography

IT is half a century since the novelist **C (Charles) P Snow (1905-1980)** introduced the phrase *The Two Cultures* in his Rede Lecture at Cambridge, published in *Encounter* in June-July, 1959.

Crystallographers have a special link with Snow. He had researched in molecular science at Cambridge in the 1930s (although acquiring a reputation for atrocious practical work among his chemistry contemporaries at U C Leicester in the 1920s) and during World War II was concerned with recruiting scientists; the crystallographer Uli Arndt, when about to graduate, was interviewed by Snow at the Joint Recruiting Board. (For a review of Arndt's engaging autobiography see our December 2008 issue.) Snow was a Civil Service Commissioner from 1945 to 1960, and, as Lord Snow, served for a time in the Wilson government. His experience in both science and government-the cor-

ridors of power-figured in his later novels in the *Strangers and Brothers* sequence, published from 1940 onwards.

However, Snow's early novel, published in 1934, *The Search*, was a first-person narrative about an X-ray crystallographer, Miles, supposedly born about 1901. There are references to **W L Bragg** at Cambridge and to crystallography at the Royal Institution in the early days of photographic recording. Snow was a friend and great admirer of the crystallographer **J D Bernal**, calling him the bravest man he had known. Bernal was clearly the inspiration for the character Constantine in *The Search*; Constantine was a brilliant maverick scientist (though possessing a much wider factual knowledge) with a tawny mane, and had-like the narrator-a vision of an interdisciplinary protein research institute. *The Search* must be one of very few novels featuring a crystallographer.

Derry W. Jones
University of Bradford

The Frank Allen Symposium

The Frank Allen Symposium 23-24 April 2009

Summary Report by Sam Motherwell

THE symposium marked the occasion of Frank Allen's retirement from the Cambridge Crystallographic Data Centre (CCDC), which he joined in 1970: a total of 39 years of scientific and data base achievement. The 12 talks represented well the extent of Frank's work, particularly his interest and his personal contribution to research (over 200 papers). Many CCDC funded research students have benefited from Frank's support and personal supervision. In the early 70's the Cambridge Structural Database (CSD) contained only a few thousand structures, and the focus was still on molecular structure – many bond types and chemical structures were being synthesized and observed for the first time. However Frank's vision of the database was that more can be learnt by systematic comparison of structures than can be observed in the individual cases. It should be remembered also that the concept of on-line searching of databases only became possible in the 1980's and the original database had to be laboriously searched on sequential computer tapes. However, the groundwork of software for systematic comparison of molecular structure was laid down in the 70's to which Frank made a significant contribution. This work culminated in published tables of Interatomic Distances which became a standard reference source. As computers became faster in the 80's the original sequential processing software was integrated into the QUEST program, which developed in due course to present day ConQuest as the definitive search program for the CSD. Frank was also an innovator of the statistical analysis program for CSD data (VISTA).

Research groups, both within CCDC and internationally, have identified trends and relationships between molecular parameters, and principles have been established which would not have been possible without the CSD (2261 published papers are available at www.ccdc.cam.ac.uk/free_services/webcite). As interest extended to inter- rather than intra-molecular interactions, Frank's work led the way to such things as classification of H-bonding motifs, organometallic interactions, studies of $Z' > 1$, packing pattern recognition, analysis of space group frequency, molecular and crystal symmetry relationships. Many of these research fields were represented in the symposium talks.

Neil Feeder (Pfizer, standing in for Bob Docherty) gave an account of the importance of crystallography and the database in selection of the actual delivery of active pharmaceuticals in solid state form. Besides issues of patent law, it is of great importance that the polymorph selected is known to be stable, as solubility and other physical properties (e.g. particle shape) can have a crucial role in the physical delivery of a drug. Once a chemical molecule has been shown to be pharmaceutically active it is necessary to establish all known polymorphs, both of the pure compound and its salt forms (usually necessary to promote bio-solubility). Matters such as hydrate formation and co-crystals are all part of the selection process. Examples were given of catastrophic manufacturing problems when a new polymorph appears at the production stage. The importance of obtaining early warning that a given polymorph may not be the most stable was explained, and also the necessity of reducing the time spent looking for a suitable solid form. Examples were given particularly of use of the CSD to determine that a candidate polymorph showed a bifurcated and rather weak H-bond between certain groups, where the CSD interactions of these groups showed always a single strong H-bond.

Lee Brammer represented the application of CSD to organometallic compounds. He described the discovery and development of novel interactions between carbon-halides and halides bonded to metal (C-X ... X'-M). Systematic studies have shown that this interaction is primarily electrostatic rather than charge-transfer, and electronic description of these kinds of 'halogen bonds' can be viable, comparable to the description of strong and weak H-bonds. Some examples were given of crystal structural packing patterns which had a degree of predictability.

Kirsty Anderson (University of Durham) described work on CSD structures which have more than one molecule of the same species in the asymmetric unit (about 8% of the CSD). A special database of this subset of structures is available at www.durham.ac.uk/zprime. Investigations have shown that the likelihood of a given molecule forming a $Z' > 1$ polymorph may be related to its ability to also form co-crystals. Effects of chirality and molecular size and shape have also been studied.

Laszlo Fabian (CCDC) described work on prediction of co-crystal formation (i.e. no ionic species, but molecular components A:B uncharged). Excluding solvents, all organic co-crystals in the CSD have been examined in a special sub-database. Factors other than H-bonded synthon complementarity are considered to be important. Molecular descriptors were generated for all molecules in this subset,

and statistical correlation sought between components A and B. The strongest correlations are for molecular polarity compatibility, fraction of N,O atoms per molecule, and molecular shape (defined in terms of ratio of an enclosing box of sides L,M,S, and using the aspect ratios M/L and S/L). These statistics were used to apply prioritization to experimental screening by synthesis using lists of possible components B for a given molecule A. A key result showed that using only the most 'likely' components can increase efficiency of the screening experiment by 50%, while losing only 15% of the co-crystals.

Carol Brock (University of Kentucky) presented a talk on co-crystals of molecular isomers or near-isomers. These co-crystals represent a failure of conventional fractional crystallization of a mixture. A subset of the CSD was formed where there are two non-charged components in the crystal, same chemical sum formula but different connectivity. A further list of molecules in CSD which are near-regioisomers, diastereoisomers, or enantiomers, or just 'nearly the same' was compiled (ca. 4000). There is no obvious energetic advantage of formation of such co-crystals over the formation of pure components, as isomeric compounds can often form the same H-bond motifs in either case. A possible driver for formation of such co-crystals was proposed as an increase of density, in other words tighter molecular fit. A list of 140 co-crystals was made; most (70%) were quasi-racemates often using non-crystallographic symmetry operators, especially an inversion centre. If the molecules can form H-bonds, the formation of a co-crystal seems more likely than for non H-bonding molecules.

Pete Wood (CCDC) gave a talk on carbamazepine co-crystals. This drug molecule has been extensively studied with regard to co-crystal formation (50 structures). Some of these compounds contain varying amounts of solvent molecules in channels, suggesting possibilities for controlling solubility of pharmaceutical cocrystals. Pete showed how the Mercury Materials software was used to analyze and classify packing patterns by geometric comparison of the coordination shell of 14 molecules in the crystal. H-bonding patterns were also analyzed in terms of chain and ring motifs. The PIXEL program was used to explore the energy of interaction of molecular pairs - a notable result being that although a H-bonded dimer often occurs at -28 kJ/mol, the stacking of molecules (dispersion forces) gives recurring interaction of -15 kJ/mol. The conclusions are that H-bonding does give recurring patterns in a range of cocrystals, but dispersion forces are very important i.e. molecular shape interactions.

Angelo Gavezzotti (University of Milan) reviewed 30 years of polymorphism in the CSD. This introduced the topic of 'crystal structure prediction' in this symposium. Angelo expressed how the advent of the CSD on his personal computer revolutionized his research. He acknowledged the difficulty that the CSD often contains only one polymorph for a compound, simply because it was not appropriate to search for others (a time consuming process). He reviewed his early 1982 analysis of packing patterns in CSD

molecules, and his methodology for searching for possible polymorphs using frequency of symmetry operators. He established in 1995 that when polymorphic forms are observed there are very small differences in lattice energy and density. He explained the development of the PIXEL program which calculates whole molecular interactions in pair-wise fashion, also allowing partition of the energy into electrostatic and dispersion components. He explained how often 50% or more of organic crystal lattice energy is composed of dispersion components, so that focusing attention exclusively on atom-atom pair-wise interactions is not a viable explanation of the 'driving force' in determining the polymorphic structure. His current work is in the direction of simulation of nucleation in crystals, in order to explore why certain polymorphs of almost equal energy and density are found experimentally and others are not.

Peter Galek (CCDC) gave a talk on assessment of the stability of pharmaceutical polymorphs through H-bond prediction. Most drug molecules exhibit H-bonding and it has been shown that examination of a relevant set of molecules from the CSD can predict the probability of H-bond formation between the limited set of donors and acceptors on the target molecule. Statistical methods have been applied to analyze a chosen CSD subset to derive a probability function giving the propensity for formation of each pair of functional groups e.g. C-OH alcohol ... C=O keto. The important factors in this function are (a) competition effect depending on number of groups, (b) observed frequencies of formation of H-bond pair, (c) steric accessibility of groups. It has been shown that a function fitted to a given training subset of the CSD can give good prediction of H-bond pairing for a new unknown compound. This prediction is of vital importance in development of new drug solid state forms, because if predicted pairs of H-bonded groups do not appear this is an indicator that the polymorph is probably not the lowest energy possible, and further experimental screening should continue to establish the most stable form. Several case studies were presented.

Sally Price (University College London) gave a review of progress in the science of organic crystal structure prediction. The problem is defined as the prediction of the most stable polymorphs of a given pure compound, knowing only its chemical structural formula. Sally discussed the findings of a series of 4 blind tests of crystal structure prediction, which had been organized by CCDC at approximately 2 yearly intervals. Categories of organic molecules were chosen in order of perceived difficulty, (a) small rigid C H N O atoms only, (b) Rigid but with more challenging atoms such as Cl, Br, I, S, (c) limited flexibility to two torsional angles, and (d) two component systems of rigid molecules e.g. cocrystals. Sally explained the strategy for calculating and searching for all possible crystal structures for a given molecule. There has been a significant increase in the success rate in successive tests, and the latest (conducted 2007) shows real progress in that correct structures were predicted in all 4 categories, and one group of researchers were successful in all 4. For small rigid molecules it seems now accepted that there is a high probability of a correct prediction. Not all structures are of the same difficulty to predict; the energy landscape

landscape of the thousands of calculated possible structures shows a clear single low-energy candidate for only certain molecules, probably as a factor of the molecular shape and certain advantageous pair-wise packings. Improved force fields are being developed, derived in part from *ab initio* DFT, and partly from empirical fitting (to known structures via the CSD). As flexibility of the molecule and number of molecular components (including solvents) increases, the number of possible polymorphs increases steeply, and strategy in searching this increasing polymorph space remains a challenge, but results are encouraging.

Simon Parsons (University of Edinburgh) gave a talk on high pressure induced phase transitions in molecular crystals. The experimental techniques have been refined so it is now routinely possible to examine organic crystals in the laboratory at room temperature up to a pressure of 5 -10 Gpa, where there is sufficient change in the PV term of the free energy $G = U - TS + PV$, to cause a phase transition to a more stable form. Results were presented for several molecules showing the steady decrease in void space between molecules with increasing pressure, until a critical point is reached and a phase transition occurs. This often involves keeping the same arrangement of layers of molecules in the crystal but with a displacement in some direction. Studies have shown that the closest approach of specific pairs of atoms below their normal van der Waals contact can be tolerated up to a certain point. The sudden phase transition can in some cases be related to a certain critical close approach which is so far into the repulsive region of inter-atomic potential that it must be avoided.

Robin Taylor (former CCDC Development Director) gave a review of the development of life science software at CCDC. The major software product in this area (GOLD) was aimed at a central problem in drug design: predicting how a small molecule might bind to a protein. This program had considerable success, using data based on extensive analysis of interatomic contacts between functional groups in the CSD and the PDB. Other products were developed, aimed primarily (though not exclusively) at industrial molecular modeling, in the area of protein-ligand interaction. Robin then gave the background to why the CCDC had ventured into this area, as it is primarily the curator of the definitive small molecule database. The answer lies in the independent nature of CCDC, a not-for-profit organization, which receives little or no public funding. There was of course the scientific challenge of the life sciences where it may be regarded as a natural expansion of the research activities in CCDC, which have been a very significant at CCDC from the very beginning. But to maintain independence and financial stability it was important to create a financial model where industry would support the CCDC by lease of the CSD, and equally important the CSD related software. The other remit of CCDC continues to be to supply academia with the state of the art software, in recognition of the contribution of the data, without which the CSD would not exist in its comprehensive and scientific integrity.

Colin Groom (Director CCDC) gave a concluding talk, paying tribute to Frank's achievements and discussing future directions for the CCDC. Colin illustrated in a humorous fashion the dangers of uncritical use of data, especially via the internet (there are many Frank Allen's on Google!). The serious point is that the CCDC maintains the CSD for the international community, and that there is added-value to the data records by consistent quality checking throughout the whole database. The other added value is the software for searching and analysis - the continuing development of ConQuest and Mercury is aimed at every user, to make the data more accessible and useful without needing specialist knowledge of crystallography. The CSD also has a future role in the field of chemistry education. The CCDC will respond to changing technology, for example the expectation of internet access is being addressed. The so-called 'free' availability of data is a challenge, but the CSD model of quality and completeness is of paramount importance to most users. Colin concluded with an outline of CCDC funding, reminding us of its fundamental aims: "In seeking to further the cause of science the CCDC is not motivated by profit maximization." The actual cost of running the CCDC is a tiny fraction of the value of the data. People pay for completeness, quality, ease-of-use, convenience, confidence in results, associated software. The CCDC will adapt to changing needs (e.g. internet), competition ('Free data'), changes in the pharmaceutical industry, technology and software. Colin finished with acknowledgement of the support of all CSD users, CCDC staff over the years, and particularly Frank.



Paul Raithby awards Life Membership of the BCA to Frank Allen

News from the Groups

Biological Structures Group Winter Meeting 2009

THE Biological Structures Group winter meeting will be held on Friday December 18th 2009 at the Royal Free Hospital in Hampstead (north London) starting at 11.00 am in the Atrium lecture theatre.

The theme of the meeting is 'Pathological Proteins' ...which should be interpreted in its broadest context. The aim is for the conference to encompass structural studies of proteins involved in a wide range of disease processes including neurological disorders, atherosclerosis, amyloid disease, infection and immunity, and prospects for therapy.

INVITED SPEAKERS INCLUDE:

Prof. **Salam Al-Karadaghi**, Department of Molecular Biophysics, University of Lund, Sweden. *'Insights into the molecular mechanism of Friedreich's ataxia from X-ray crystallography and electron microscopy studies of frataxin.'*

Dr **Kate Brown**, Department of Life Sciences, Imperial College London.

Dr **Arefeh Seyedarabi**, School of Biological and Chemical Sciences, Queen Mary, University of London. *'Structural and functional studies of IpaH9.8 from Shigella flexneri.'*

Dr **Ian Taylor**, National Institute of Medical Research, Mill Hill, London. *'The retroviral capsid and restriction factors.'*

Prof **Gabriel Waksman**, ISMB, Birkbeck / UCL. *'Structural Biology of Type IV Secretion Systems.'*

Prof **Steve Wood**, UCL Department of Medicine (Royal Free Campus). *'Targeting transthyretin - X-ray crystallography and rational drug design.'*

The Royal Free Hospital is situated close to leafy Hampstead Heath and is a short walk from Belsize Park tube station which is about 10 - 20 minutes on the underground from most London main line rail terminals.

More details about the meeting will be available during the Autumn via the BCA / BSG website. The meeting is being generously sponsored by Bruker-AXS, GSK, Molecular Dimensions, Oxford Diffraction and Rigaku.

Nominations for speakers and other enquiries should be made to the organiser: **Jon Cooper** by e-mail to jbcooper@medsch.ucl.ac.uk

CCG Autumn Meeting Chemistry Research Laboratory, University of Oxford Wednesday 18th November 2009

| | |
|--------------------|---|
| 10.30–11.15 | Registration with coffee/tea |
| 11.15–12.00 | Professor Kenneth Harris, Cardiff <i>"Exploiting a multi-technique experimental strategy to probe crystallization processes and structural transformations for organic materials."</i> |
| 12.00–12.30 | Dr Stewart Parker, ISIS <i>"Vibrational Spectroscopy with Neutrons."</i> |
| 12.30–14.00 | CCG Extraordinary General Meeting followed by Lunch |
| 14.00–14.30 | Dr Moniek Tromp, Southampton <i>"What X-ray Absorption Fine Structure Spectroscopy can do in structure determination - Strengths and limitations compared to crystallography."</i> |
| 14.30–15.00 | Katharina Fucke, Durham <i>"Getting hot in solid-state: what a crystallographer can learn from thermal analysis."</i> |
| 15.00–15.30 | Coffee |
| 15.30–16.00 | Dr Louis Farrugia, Glasgow <i>"Chemistry from Charge Densities."</i> |
| 16.00–16.30 | Dr Matt Tucker, ISIS <i>Disorder, crystals and the forgotten parts of powder diffraction</i> |
| 16.30 | Close |

Details of CCG Extraordinary General Meeting

An Extraordinary General Meeting of the Chemical Crystallography Group is called to address an unusual situation that has arisen with the CCG Committee. The Committee currently comprises three Officers (each serving a 2-year term) and six Ordinary Members (each serving

a 3-year term). The expectation is that there should be a changeover of two Ordinary Members each year.

Due to a sequence of early retirements from the Committee, five of the six current Ordinary Members are due to retire at the next CCG AGM (April 2010). The other ordinary member is due to retire in April 2012.

The purpose of the Extraordinary General Meeting is to ratify measures that will assist continuity within the Committee and restore a more regular sequence for the changeover of Ordinary Members.

The Committee proposes the following action:

- (i) Three of the five current Ordinary Members will retire as expected at the CCG AGM 2010. Two of the current Ordinary Members will remain on the Committee for one further year. Two new Ordinary Members will be elected in the usual way.
- (ii) The two Ordinary Members serving extended terms will retire at the CCG AGM 2011 and be replaced by two new Ordinary Members in the usual way.
- (iii) One current Ordinary Member will retire as expected at the CCG AGM 2012. Two new Ordinary Members will be elected in the usual way.

In this way, the Committee will comprise five Ordinary Members during the period 2010–2012 and be restored to six Ordinary Members in 2012 with a regular changeover sequence from that point onwards.

Full details of the proposals will be made available on the CCG website (<http://ccg.crystallography.org.uk/>) and will be circulated to all CCG members via e-mail four weeks before the Extraordinary General Meeting.

PCG Winter Meeting

THE PCG/SCMP Group's Winter Meeting will be held at Cosener's House on Thursday 5th and Friday 6th November.

The focus will be on "New Results from New Facilities" and will once again be held jointly with the ISIS Crystallography User group meeting. More details will follow soon.

Autumn Meeting 5th November 2009 The World of Glass, St. Helens, Merseyside Impact of Crystallography in an Industrial Environment

THE morning session is for Young Crystallographer (YC) presentations. The afternoon session will flagship the role of crystallography in industry. The following talks have been submitted.

Preparation, Characterisation and Prediction of Physical Stability of Amorphous Materials during Pharmaceutical Development: Pair-Wise Distribution Function
Helen Blade, AstraZeneca

Applications of Crystallography in the Aerospace Industry
Judith Shackleton, Materials Science Centre, University of Manchester

XRD N' Chips - What makes good semiconductor devices
Chris Staddon Senior Experimental Officer, School of Physics and Astronomy, University of Nottingham.

Powder X-ray Diffraction for Process and Product Support in Shell Global Solutions
Graham C. Smith, Shell Global Solutions UK

XRD in the Imaging Industry
David Beveridge, Harman Technology

See web site for map and how to get there by train or car.

Call for Papers - Please call **John Kaniuka**
Tel: **01695 54303** (Young Crystallographers) or
Mark Farnworth with offers of talks at this meeting.

Organiser:
Mark Farnworth
Tel: **01695 54639**
Email: Mark.Farnworth@pilkington.com



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, c.h.schwalbe@aston.ac.uk. The help of the IUCr listing is gratefully acknowledged.

6-8 September 2009

BACG 40th Anniversary Meeting, 2009, Bristol, United Kingdom
<http://www.bacg.org.uk/BACG.NET/default.aspx>

7-8 September 2009

7th International NCCR Symposium on New Trends In Structural Biology, Swiss Federal Institute of Technology (ETH), Zurich, Switzerland
<http://www.structuralbiology.uzh.ch/symposium2009/>

8 September 2009

Annual Meeting of the SGK/SSCr, Fribourg, Switzerland
<http://www.sgk-sscr.ch/>

8-11 September 2009

Synchrotron Radiation and Polymer Science 4, Rolduc, The Netherlands
<http://srps4.chem.tue.nl/index1.html>

13-18 September 2009

Aperiodic09, University of Liverpool, UK
<http://www.aperiodic09.org>

13-18 September 2009

XIV International Conference on Small-Angle Scattering (SAS-2009), Oxford, UK
<http://www.sas2009.org/>

20-23 September 2009

European Conference on Solid-State Chemistry, University of Münster, Germany.
http://www.gdch.de/vas/tagungen/tg/5585__e.htm

20-23 September 2009

Grazing Incidence Small Angle Scattering (GISAS) Conference DESY, Hamburg, Germany
<https://indico.desy.de/conferenceDisplay.py?confId=797>

20-24 September 2009

XXI Conference on Applied Crystallography, Zakopane, Poland
<http://crystallography.us.edu.pl/>

21-25 September 2009

Clays, Clay Minerals and Layered Materials – 2009, Moscow, Russia
<http://www.cmlm2009.ru/>

27 September – 2 October 2009

SRI2009: 10th International Conference on Synchrotron Radiation Instrumentation, Melbourne, Australia
<http://www.sri09.org/>

5-7 October 2009

Studying Kinetics with Neutrons by SANS and Reflectometry, SKIN2009, Grenoble, France
<http://www.ill.eu/news-events/workshops-events/skin2009/>

5-8 October 2009

JCNS Workshop : Trends and Perspectives in Neutron Scattering on Soft Matter, Tutzing, Germany
<http://www.jcns.info/Workshop/>

19-23 October 2009

Basic and Advanced Rietveld Refinement & Indexing Workshops, Newtown Square, PA, USA
<http://www.icdd.com/education/rietveld-workshop.htm>

26-28 October 2009

Polymorphism & Crystallization Scientific Forum, Philadelphia, PA, USA
<http://www.polymorphismforum.com/Event.aspx?id=205286>

28-30 October 2009

International Workshop on the Analysis and Refinement of the Electron Density, Marrakech, Morocco
<http://www.ucam.ac.ma/fssm/adr/>

5 November 2009

Industrial Group Meeting: Impact of Crystallography in an Industrial Environment, The World of Glass, St. Helens, Merseyside
<http://ig.crystallography.org.uk/ig.htm>

5-6 November 2009

Physical Crystallography Group/SCMP autumn meeting: New Results from New Facilities, Cosener's House, Abingdon, Oxfordshire
http://www.pcg-scmp.org/Main_Page

18 November 2009

Chemical Crystallography Group autumn meeting: Methods Complementary to Crystallography, Chemistry Research Laboratory, University of Oxford
<http://ccg.crystallography.org.uk/>

18 December 2009

Biological Structures Group winter meeting: Pathological Proteins, Royal Free Hospital, Hampstead, north London
<http://bsg.crystallography.org.uk/>

27-29 January 2010

Flipper 2010: International Workshop on Single-Crystal Diffraction with Polarised Neutrons, Grenoble, France
<http://www.ill.eu/news-events/workshops-events/flipper-2010>

12-13 April 2010

Young Crystallographers satellite meeting, University of Warwick
<http://www.chem.gla.ac.uk/yc/>

13-15 April 2010

BCA Spring Meeting: Data Matters, University of Warwick
<http://www.crystallography.org.uk/>

18-22 April 2010

PCG Rietveld School, Durham
<http://www.crystallography.org.uk>

12 May 2010

BCA Industrial Group XRF users' meeting, British Geological Survey, Keyworth, Nottingham
<http://ig.crystallography.org.uk/ig.htm>

13 May 2010

BCA Industrial Group minerals meeting, Between the Sheets! British Geological Survey, Keyworth, Nottingham
<http://ig.crystallography.org.uk/ig.htm>

24-29 July 2010

American Crystallographic Association Meeting, Chicago, Illinois, USA.
http://www.americalassn.org/meetingspg_list/futuremeetings.html

27-30 August 2010

EPDIC12. 12th European Powder Diffraction Conference, Darmstadt, Germany.
<http://www.epdic12.org/>

29 August - 2 September 2010

26th European Crystallographic Meeting, Darmstadt, Germany.
<http://www.ecm26.org/>

25-29 August 2013

28th European Crystallographic Meeting, University of Warwick.
<http://www.crystallography.org.uk/>

**BIOMOLECULAR
CRYSTALLOGRAPHY**

Principles, Practice, and Application to Structural Biology

Bernhard Rupp



Bernhard Rupp,
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