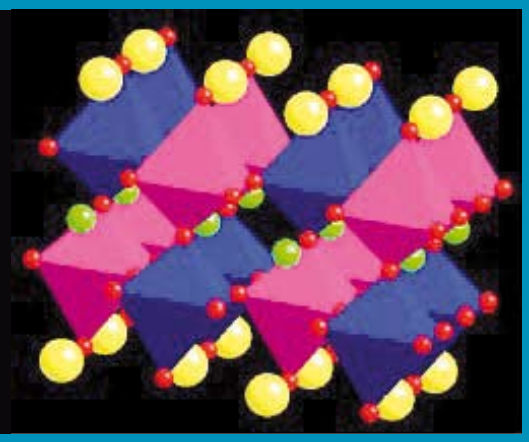
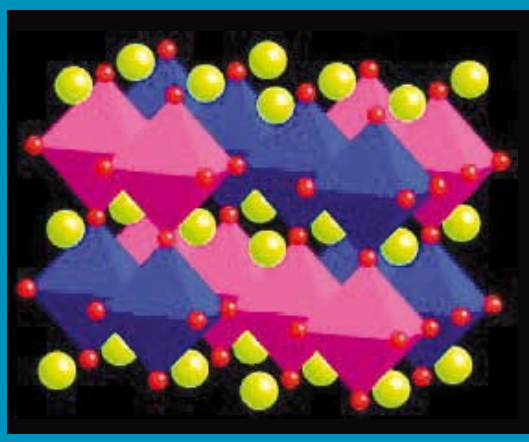
Crystallography News

British Crystallographic Association

Issue No. 106 September 2008
ISSN 1467-2790





Annual Meeting Loughborough 2009 p6-9

Contents

BCA Administrative Office,
David Massey,
Northern Networking Events Ltd.

1 Tennant Avenue,
College Milton South,
East Kilbride, Glasgow G74 5NA
Scotland, UK

Tel: + 44 1355 244966
Fax: + 44 1355 249959

e-mail: **bca@glasconf.demon.co.uk**

CRYSTALLOGRAPHY NEWS is published quarterly (March, June, September and December) by the British Crystallographic Association, and printed by William Anderson and Sons Ltd, Glasgow. Text should preferably be sent electronically as MSword documents (any version - .doc, .rtf or .txt files) or else on a PC disk. Diagrams and figures are most welcome, but please send them separately from text as .jpg, .gif, .tif, or .bmp files. Items may include technical articles, news about people (e.g. awards, honours, retirements etc.), reports on past meetings of interest to crystallographers, notices of future meetings, historical reminiscences, letters to the editor, book, hardware or software reviews. Please ensure that items for inclusion in the December 2008 issue are sent to the Editor to arrive before 25th October 2008.

Carl Schwalbe
School of Life & Health Sciences
Aston University
Aston Triangle
Birmingham B4 7ET
Tel: 0121 204 4009
e-mail: c.h.schwalbe@aston.ac.uk

The British Crystallographic Association is a Registered Charity (#284718)
As required by the DATA PROTECTION ACT, the BCA is notifying members that we store your contact information on a computer database to simplify our administration. These details are not divulged to any others without your permission. You may inspect your entry during the Annual Meeting, or otherwise by application to the BCA Administrative Office. We will be happy to amend entries at any time.

Designed & printed by Wm. Anderson & Sons Ltd.
34 Loanbank Quadrant, Glasgow.
Tel: 0141 440 2881
e-mail: enquiry@andersonprinters.com

This month's cover:

SPring-8, the first charge-ordered polymorphs, Osaka Castle; award winner Paul Raithby

Photo credits: SPring-8: Wikimedia Commons en:Image:SPring-8 2007 12img pano.jpg,
Osaka Castle: kindly supplied by the IUCr Secretariat, Charge-ordered polymorph: Paul Attfield

From the President

IN response to a comment made by the recently retired Crystallography News Editor, Bob Gould, that I never write my presidential pieces from anywhere exotic unlike previous presidents, I am writing this article from the work area on Station 9.8 at the Daresbury Laboratory Can one get more exotic than that? It is a sunny weekend in early July and it is my last visit ever to the SRS while it is in operation as Station 9.8 closes at the end of July. This station has been of great service to a significant proportion of the Chemical Crystallography community for more than a decade and I would like to add my personal thanks to all those who have been involved in the design, building and running of Station 9.8. The facility will be sadly missed by many and life just will not be the same without weekends spent at Daresbury! However, by the time that this article appears in print the commissioning of Station I19 at Diamond will be well underway and the new Station should be fully operational by early 2009, so there will be lots of new, exciting synchrotron-based experiments to do in the future.

Moving on to other matters, I am very pleased to say that the planning for the Spring Meeting at Loughborough, which takes place between 21st-23rd April, 2009, is already well advanced, and I am most grateful to **Simon Parsons** and the Programme Committee for all the hard work that they have already put in assembling a very interesting and wide ranging programme. The outline of the programme appears in this issue of *Crystallography News* and the full details will be published in the December issue and on the conference website. Among the highlights is the Lonsdale Lecture which starts off the main Meeting and is to be given on this occasion by **David Watkin**, from the University of Oxford. David is recognised as a world leading expert in the area of crystallographic computing and will provide a highly stimulating and enlightening lecture on the area. This year after the formal end of the Meeting there will be a satellite meeting in honour of **Frank Allen**, who will be retiring as Director of the Cambridge Crystallographic Data Centre in September this year. Frank and the CCDC team have made an enormous contribution to all areas of crystallography and this symposium gives the opportunity to Frank's many friends and colleagues to recognise these contributions and look to the future developments of the CCDC.

One of the key conferences this summer is the XXI Congress of the IUCr held in Osaka between the 23rd and 31st August, so by the time that you read this many of you will just have returned from it. As usual, there was a strong UK representation at the meeting and the BCA provided 10 bursaries to assist young scientists to travel to and from the conference.

It is again the time of year when I have the very pleasurable duty to invite nominations for Honorary Members of the BCA, and I would also like to thank several of you who have already sent me nominations. Honorary Membership is the highest membership accolade of the BCA, and is awarded to a small and select group of colleagues who have contributed significantly to crystallographic science and to the work of the BCA. In recent years we have elected **Paul Barnes**, **Paul Fewster**, **Chris Gilmore**, **Mike Glaser**, **Bob Gould**, **Mike Hursthouse**, **Peter Main** and **George Sheldrick**. In the coming year we anticipate electing one or two new Honorary Members. Please send your nominations, together with a short supporting case to me at president@crystallography.org.uk by the 31st October, 2008.

Finally, it is with great sadness that I have to report the death of A**ndrew Lang**, FRS, on 30th June, 2008. Andrew was an Honorary Member of the BCA, and was recognised for his pioneering work in the area of X-ray diffraction physics. He was a great supporter of the BCA and continued to contribute enthusiastically to the Association until a few months before his death. He will be sadly missed.

Paul Raithby

BCA Council 2008

COUNCIL MEMBERS

President (2009)
Prof. Paul R. Raithby
Department of Chemistry
University of Bath
Claverton Down
BATH BA2 7AY
Tel: 01225 383183
p.r.raithby@bath.ac.uk

Vice President (2010)
Prof. Alexander J. Blake
School of Chemistry,
University of Nottingham
University Park
NOTTINGHAM NG7 2RD
Tel. 0115 9513488
A.J.Blake@nottingham.ac.uk

Secretary (2010)
Dr Georgina Rosair
School of EPS - Chemistry
Perkin Building
Heriot-Watt University
EDINBURGH EH14 4AS
Tel: 0131 451 8036/4241
g.m.rosair@hw.ac.uk

Treasurer (2008)
Dr Harry R. Powell
MRC Laboratory of
Molecular Biology
MRC Centre, Hills Road
CAMBRIDGE CB2 2QH
Tel: (01223) 402423
hrp1000@cam.ac.uk

ORDINARY MEMBERS

Dr Richard Cooper (2009)
oXray Ltd
Oxford Centre for Innovation
OXFORD OX2 0JX
Tel: 01865 811107
Richard.cooper@ oxray.com

Dr Elspeth F. Garman (2009)
Laboratory of Molecular Biophysics
Rex Richards Building
University of Oxford
South Parks Road
OXFORD OX1 3QU
Tel: 01865 275398
elspeth@biop.ox.ac.uk

Prof. William Clegg (2009)
School of Natural Sciences (Chemistry),
University of Newcastle,
NEWCASTLE UPON TYNE
NE1 7RU
Tel: 0191 222 6649
W.Clegg@newcastle.ac.uk

GROUP REPRESENTATIVES

Biological Structures
Dr Darren Thompson
Department of Biochemistry
University of Sussex
BRIGHTON BN1 9QG
Tel: 01273 876631
D.Thompson@sussex.ac.uk

Chemical Crystallography
Dr Jonathan Charmant
School of Chemistry,
University of Bristol,
BRISTOL BS8 1TS
Tel. (0117) 928 8253
jon.charmant@bris.ac.uk

Industrial
Dr David Beveridge
Harman Technology - ILFORD Photo
Town Lane, Mobberley,
KNUTSFORD WA16 7JL
Tel: 01565 650000
David.Beveridge@ harmantechology.com

Physical Crystallography
Dr Matt Tucker
STFC Rutherford Appleton Laboratory
DIDCOT OX11 0QX
Tel: 01235 445581
M.G.Tucker@rl.ac.uk

Young Crystallographers
Ms. Susanne Huth
School of Chemistry
University of Southampton
SOUTHAMPTON SO17 1BJ
Tel: 023 8059 4132
s.huth@soton.ac.uk

CO-OPTED MEMBERS

Prof. Garry Taylor
Centre for Biomolecular Sciences
North Haugh
University of St Andrews
ST ANDREWS KY16 9ST
Tel: 01334 4677301
glt@st-and.ac.uk

Prof. Paul Fewster
PANalytical Research Centre
Sussex Innovation Centre
BRIGHTON BN1 9SB
Tel: 01273 704422
paul.fewsterpanalytical.com

EX-OFFICIO MEMBERS

Education Coordinator
Dr Michael R. Probert
Department of Chemistry
Durham University
Science Site, South Road
DURHAM DH1 3LE
Tel: 0191 334 2004
m.r.probert@durham.ac.uk

Editor
"Crystallography News"
Prof Carl H. Schwalbe
School of Life & Health Sciences
Aston University
BIRMINGHAM B4 7ET
Tel: 0121 204 4009
c.h.schwalbe@aston.ac.uk

Webmaster
Dr Jeremy Karl Cockcroft
Department of Chemistry
University College London
LONDON WC1H 0AJ
Tel: 020 7679 5802
cockcroft@ img.cryst.bbk.ac.uk

GROUP CHAIRMEN

Biological Structures Group
Dr Andrea Hadfield
Department of Biochemistry
University of Bristol
BRISTOL BS8 1TD
Tel: 0117 331 2151
a.t.hadfield@bristol.ac.uk

Chemical Crystallography Group
Dr Richard Cooper
oXray Ltd
Oxford Centre for Innovation
OXFORD OX2 0JX
Tel: 01865 811107
Richard.cooper@oxray.com

Industrial Group
Dr Anne Kavanagh
AstraZeneca
MACCLESFIELD, SK10 2NA
Tel. 01625 517454
Anne.Kavanagh@ astrazeneca.com

Physical Crystallography Group
Prof. David Keen
ISIS Facility, Rutherford Appleton Laboratory
Harwell Science and Innovation Campus
DIDCOT Oxfordshire
OX11 0QX
Tel: 01235 446556
d.a.keen@rl.ac.uk

Full committee details on the BCA website **www.crystallography.org.uk**
Spring Meeting Registration and Subscriptions: **www.crystallography-meetings.org.uk**

From the Editor

I start my column with two pieces of very happy news, both involving our President. Paul Raithby has won the prestigious Structural Chemistry Award from the Royal Society of Chemistry. The citation reads, "Awarded for his wide-ranging structural studies of organometallic and organic materials, including influential contributions to the use of synchrotron radiation in structural chemistry, leading contributions in organometallic polymers and ground-breaking developments in excited-state crystallography." For a long time I regretfully believed the famous comment by Nobel laureate **Leopold Ruzicka** that "a crystal is a chemical cemetery." Thanks to the efforts of a small number of bold crystallographers including Paul who harnessed leading-edge laser and synchrotron technology, it is becoming evident that a crystal subjected to the appropriate experimental conditions can turn into a precision engineering works. Paul has communicated another piece of good news: Station I19 at the Diamond synchrotron has recently received its first beam. More details are given elsewhere in this issue. Paul also reports sad news: the death of **Andrew Lang**, who was not only a brilliant and innovative crystallographer but also an active loyal member of the BCA.

You could be forgiven for thinking that this issue of *Crystallography News* has been mixed up with the travel section of your Sunday newspaper. As I write this, I have already attended the meeting of the American Crystallographic Association in Knoxville, Tennessee. You will see my description of the meeting in this issue. I am looking forward to the Congress of the International Union of Crystallography in Osaka, Japan. IUCr Congress venues provide the theme for the Puzzle Corner this time. I am sorry to have missed the meeting of the Slovenian-Croatian Crystallographic Association, charmingly abbreviated "Slo-Cro", in Ptuj, Slovenia. I would have loved to send my friends a postcard from Ptuj.

Although the ACA meeting was held in Knoxville, most of the organization was done by people from the Oak Ridge National Laboratory nearby. This facility is a classic example of swords into ploughshares. Its first reactor was built during World War II to develop the atomic bomb. After the war **Clifford Shull**, **Ernest Wollan** and colleagues used it for neutron diffraction. Together with **George Bacon** and coworkers at Harwell they developed this technique into a practical means for settling previously unanswerable questions about hydrogen atom location. Subsequently Oak Ridge became famous for its crystallographic software. A suite of programs prefixed "OR" covered the main stages of crystal structure analysis. Most of their algorithms have since been swallowed up by more modern programs, but ORTEP is so good that it is still in common use. Its author, **Carroll Johnson**, was cheerfully present at the ACA meeting. Neutron diffraction continues to prosper at Oak Ridge. A highlight of the meeting was a tour of the Spallation Neutron Source (SNS) now being run up to full power and having its instrumentation installed and commissioned. I am delighted to include an article by **Jason Hodges** and **Al Ekkebus** describing this powerful new facility and how to apply for access to it. The SNS is also available to non-U.S. scientists with a sufficiently strong case.

Meanwhile, neutron science in the UK has also been taking great strides forward. The first neutrons have been created at the ISIS Second Target Station, TS-2. The initial suite of instruments on TS-2 includes a new SANS instrument, SANS2D. The exciting details of this first experiment appear in this issue.

Also featured on the cover are two landmarks not far from the site of the forthcoming Osaka meeting. To show how well Japan preserves its historical heritage while embracing the most modern technology we have images of Osaka Castle and the SPring-8 synchrotron.

Closer to home, plans for next year's Spring Meeting of the BCA at Loughborough are taking shape nicely. An outline containing the names of enough distinguished speakers to whet your appetite appears in this issue, and full details will be published in the December issue and on the Web.

I draw your attention to one item in particular in the Future Meetings section. The dates for the 2009 European Crystallographic Meeting in the fascinating city of Istanbul, Turkey, have been changed to August 16-21 to avoid a clash with the rescheduled Formula I race. This is undoubtedly a good thing: hotel rooms should be somewhat cheaper, and it should stop us crystallographers comparing sluggish Formula I cars inching their way around the track with our zippy electrons whizzing around our synchrotrons.

Of course all this travel now leaves one with a guilty conscience about the carbon footprint. For the Knoxville meeting the ACA asked for a voluntary contribution of $4 per person to offset the carbon dioxide emissions. This seems amazingly cheap to me, but we crystallographers are never wrong about numbers… are we? My guilty conscience, prompted by an enquiry from **Louise Male**, extended to our use of paper in producing Crystallography News. I was pleased to learn from **John Tangny** at **William Anderson Printers** that the material is Elemental Chlorine Free (ECF), is made from pulp sourced from fully sustainable forests and follows the Forest Stewardship Council guidelines. Formal registration under the FSC scheme is currently being undertaken. More information about the Forest Stewardship Council is available on their website, www.fsc.org.

Carl Schwalbe

Puzzle Corner

THIS time the puzzle is a forwards, backwards, vertical and diagonal word search set by Joan Schwalbe. Find the International Union of Crystallography and all the venues (cities and countries; one country is abbreviated) where its Congress has taken place. How many times does Osaka occur?

For the answers to the previous puzzle corner please see p.20

P	K	Z	G	E	N	E	O	F	L	O	R	E	N	C	E	L	L	B	D
S	O	F	V	E	S	M	Q	S	E	G	D	I	R	B	M	A	C	I	F
H	O	L	R	L	R	S	O	W	A	H	E	Y	Q	O	W	S	V	O	M
A	R	K	A	A	P	M	E	S	A	K	S	O	A	Y	F	D	I	L	O
M	B	O	N	N	N	C	A	T	C	T	A	M	E	C	I	N	F	O	N
B	Y	S	J	O	D	C	I	N	A	O	S	W	O	G	S	A	L	G	T
U	N	N	O	I	N	U	E	L	Y	T	W	H	P	F	I	L	B	Y	R
R	O	C	S	T	W	E	L	V	E	P	S	C	Y	L	J	R	O	M	E
G	T	M	A	A	F	O	D	R	H	O	W	D	A	D	Z	E	E	O	A
O	S	A	K	N	G	B	D	E	N	T	I	R	E	M	R	H	E	S	L
I	S	Y	A	R	X	A	V	A	W	H	T	R	Q	T	S	T	P	A	G
O	X	Y	A	E	M	G	P	I	C	S	Z	M	S	Y	I	E	C	K	U
T	W	P	O	T	T	A	W	A	U	Q	E	P	H	S	R	N	D	A	R
O	H	B	V	N	J	E	N	A	C	E	R	P	E	T	U	Z	U	X	D
Y	L	A	T	I	H	A	E	L	O	M	L	O	H	K	C	O	T	S	W
K	X	U	A	E	D	R	O	B	Y	Z	A	T	G	N	I	J	I	E	B
I	P	Q	F	A	N	O	R	T	U	E	N	I	T	H	G	R	F	E	O
F	U	N	I	T	E	D	K	I	N	G	D	O	M	A	A	P	J	W	N
G	E	N	E	V	A	K	A	S	O	M	A	R	A	P	E	G	I	F	D
G	U	R	C	H	I	N	A	T	O	M	V	E	T	W	A	S	R	A	W

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:

David Massey | BCA Administrative Office
Northern Networking Events Ltd
1 Tennant Avenue, College Milton South
East Kilbride G74 5NA
Phone 01355 244966 Fax 01355 249959
e-mail **bca@glasconf.demon.co.uk**

Corporate Members

Alpha Biotech
Astex Technology
Bruker AXS Ltd
Cambridge Crystallographic Data Centre
Chemical Computing Group
CrystalMaker Software Ltd
Hampton Research
Incoatec GmbH
International Centre for Diffraction Data
Malvern Instruments Ltd
Marresearch GmbH
Molecular Dimensions Ltd
Oxford Cryosystems
Oxford Diffraction
PANalytical
Rigaku MSC
STFC
Thermo Fisher Scientific

AGM Loughborough 2009

BCA Annual Spring Meeting University of Loughborough 21st-23rd April 2009 "Dynamic Crystallography"

THE main meeting will follow the successful format of recent years and run from 11:30 on Tuesday 21st April to 13:30 on Thursday 23rd April. The overall theme of the meeting is "Dynamic Crystallography" and there are 20 cutting-edge symposia (titles below) around this theme. The meeting will see the return of a parallel XRF stream for the third time of what is becoming a regular two year cycle. The programme will be of general interest and combined with some dedicated XRF stands in the commercial exhibition makes the meeting one not to be missed for anyone involved in XRF or considering a purchase. We've allocated slots in each symposium for contributed talks, and details on how to submit abstracts are given below.

In addition to the main meeting two **satellite meetings** will also take place:

- The Young Crystallographers Meeting will run on Monday afternoon and Tuesday morning.
- A meeting to mark the retirement of Dr **Frank Allen** as Scientific Director of the Cambridge Crystallographic Data Centre (http://www.ccdc.cam.ac.uk/) will run on Thursday afternoon until Friday lunchtime.

There is also a hands-on computer workshop on the dSNAP program (http://www.chem.gla.ac.uk/snap/dSNAP_index.html) for multivariate analysis of structural data.

Simon Parsons, Chair, Programme Committee
S.Parsons@ed.ac.uk

Links

Loughborough University:
http://www.lboro.ac.uk/

Travel to Loughborough University:
http://www.crystallography-meetings.org.uk/arrival.htm

Conference Web Site:
http://www.crystallography-meetings.org.uk/

Speakers:
http://www.crystallography-meetings.org.uk/Speakers.htm

Plenary Speakers

Teaching Plenary (PCG):
Martin Dove (University of Cambridge)
Dynamics from Diffraction: Information Beyond the Atomic Displacement Factor.

Lonsdale Lecture:
David Watkin (University of Oxford)
Crystallography - Technology, Science or a Black Art?

Biological Structures Group:
Venki Ramakrishnan (MRC, Cambridge)
Insights into translation from crystallography of functional complexes of the ribosome.

XRF:
David Lowe (United Kingdom Accreditation Service)
Method Validation to Achieve ISO 17025 Accreditation,

Selected Speakers

Ann Chippendale (Reading)
Robert Feidenhans'l (Copenhagen)
A**ndrew Goodwin** (University of Cambridge)
Philippe Guionneau (Institut de Chimie de la Matière Condensée de Bordeaux and University of Bordeaux)
Robert Hammond (Leeds)
Madeleine Helliwell (Manchester)
Frank Leusen (IPI)
A breakthrough in crystal structure prediction
Hazel Sparks (University of Durham)
[2+2] cycloaddition reactions in the spotlight
Christina Streli (TU Wien)

Programme Committee

Professor **Simon Parsons** (Chair of Programme Committee)
Professor **Paul Raithby** (BCA President)
Professor **Sandy Blake** (BCA Vice President)
Dr **Harry Powell** (BCA Treasurer)
Mrs **Gill Moore** (Northern Networking)
Mr **David Massey** (Northern Networking)
Dr **John McGeehan** (BSG)
Dr **Pierre Rizkallah** (BSG)
Dr **Andrew Bond** (CCG)
Dr **Amber Thompson** (CCG)
Mr **Chris Staddon** (IG)
Dr **David Taylor** (IG)
Dr **David Allan** (PCG)
Dr **Andrew Goodwin** (PCG)

Dr **Simon Coles** (YC)
Ms **Helen Maynard** (YC)

Abstract Submission

There are slots reserved in each symposium for contributed talks. Please submit abstracts for consideration using the Word template available from **http://www.crystallography-meetings.org.uk/abstracts.htm** by 29th September 2008. Applicants will be informed by the end of October whether their talk has been accepted.

As always the poster session will be one of the main for opportunities for scientific discussion. The deadline for poster abstracts is 2nd February 2009.

Please ensure that all abstracts are submitted using the template available from **http://www.crystallography-meetings.org.uk/abstracts.htm**. Please do not use old versions of the template from previous years.

Frank Allen Symposium BCA Loughborough 2009

Frank Allen will retire from the position of Executive Director at the Cambridge Crystallographic Data Centre (CCDC) on 30 September 2008. Frank has been a central pillar of crystallography in Cambridge since 1970. He joined **Olga Kennard**'s group following undergraduate and graduate studies at Imperial College, London with **Don Rogers** and postdoctoral work with **Jim Trotter** at the University of British Columbia in Vancouver, Canada. Initially the work in Cambridge involved structure analysis of organic crystals but work within the embryo crystallographic database soon became the major interest.

He has been involved in most major developments at the CCDC. He has written (& run!) software to check entries and build the Cambridge Structural Database (CSD) & played a major part in releases of the CSD during the 1970s and 1980s. He has written numerous software components which underpinned the first programs used to search the CSD - CONNSER, BIBSER and ultimately QUEST which was first released worldwide in 1989. A few key Allen-Motherwell routines are still invoked by today's ConQuest program!

Frank always realized the potential afforded by the accumulation of structural results within the CSD and his research places strong emphasis on the application of the accumulated data in the CSD and the results that can be obtained from them. Frank has co-supervised many PhD students through his collaborations with structural chemistry groups worldwide and has authored or co-authored in excess of two hundred publications.

Frank became a Fellow of the Royal Society of Chemistry in 1992, was awarded the RSC Silver Medal and Prize for Structural Chemistry in 1994 and was Vice-President of the British Crystallographic Association (BCA) between 1997 and 2001. Frank became the Editor of Acta Crystallographica, Section B in 1994 and served in this role for eight years until the Geneva Congress in 2002. He was a Council Member of the European Crystallographic Association between 1997 and 2001 and is a Visiting Professor of Chemistry at the University of Bristol. Frank won the prestigious Hermann Skolnik Award of the American Chemical Society's Division of Chemical Information in 2003.

Frank is known to generations of crystallographers & chemists around the world through his international activities, his commitment both to our community & to science and by his approachable, affable and unflappable nature. The speakers invited to participate in this symposium will reflect the range of Frank's important and unique contribution to crystallography, structural science and informatics.

Owen Johnson, CCDC

Title	Group(s)	Chair
Developments in Instrumentation and Technology	BSG CCG	**Gwyndaf Evans**
Radiation Damage	BSG	**Elspeth Garman**
Reactions in Macromolecular Crystals-1	BSG	**John McGeehan**
Reactions in Macromolecular Crystals-2	BSG	**Arwen Pearson**
Structure and Dynamics in Metalloproteins	BSG	**Peter Moody**
Data Acquisition for Time Resolved Studies	BSG	**Ilma Schlichting**
Computation, Graphics and Animation	BSG	**Venki Ramakrishnan**
Computational Crystallography	CCG PCG	**Richard Cooper**
Dynamic Techniques	CCG/YC	**Lynn Thomas**
Reactivity in Crystals (1)	CCG	**Andrew Bond**
Reactivity in Crystals (2)	CCG	**Alex Griffin**
Temperature-Dependent Crystallography	CCG	**Andres Goeta**
Environmental Applications -1	IG XRF/XRD	**Richard Morris**
Monitoring crystals, crystallization and transformations 1	IG/BACG	**Nick Blagden (BACG)**
Monitoring crystals, crystallization and transformations 2 [Joint with BACG]	IG/BACG	**Alison Burke (IG)**
Understanding API Phase Transitions	IG	**Brett Cooper**
Crystallography in the Pharmaceutical Pipeline	IG	**Matt Johnson**
Multiferroics	PCG	**Andrew Wills / Peter Hatton**
Crystallography Near the Edge	PCG	**Matt Tucker / Dave Allan**
Hydrogen Storage	PCG	**Dave Keen / Ivana Evans**
Dynamics in Framework Structures	PCG	**Andrew Goodwin / Matt Tucker**
XRF: General Session [Two Sessions]	XRF	**David Beveridge**
New Developments in Instrumentation and TXRF	XRF	**Margaret West**
Environmental Applications-2	XRF	**Dave Taylor**
Trace Analysis	XRF	**Mark Ingham**
Method Validation	XRF	**Ros Schwarz**
Portable Instruments	XRF	**Margaret West**

Timetable, AGM Loughborou

	Day 0 Monday 20 April	Day 1 TUESDAY 21 April				Day 2 WEDNE	
9:00		Lecture Theatre 1				Lecture T	
9:15		Young Crystallographers 4				Plenary IG	
9:30							
9:45						Coffee 9.	
10:00							
10:15						Parallel session	
10:30		Registration/exhibition 10.30-11.30				Lecture Theatre 1	Lecture Theatre 2
10:45						Reactivity in Crystals-1 (CCG)	Reactions in Macromolecular Crystals-1 (BSG)
11:00				Lecture Theatre 4			
11:15				XRF General (XRF)			
11:30		Lecture Theatre 1					
11:45		Lonsdale Lecture 11.30-12.30					BSG AGM 11.45-12.30
12:00							
12:15							
12:30		Lunch/exhibition/registration 12.30-13.30				CCG AGM 12.30-1.15	Lunch/AG
12:45							
13:00	Lecture Theatre 1						
13:15	Young Crystallographers 1						
13:30		Sessions 13.30-15.00				Sessions 1	
13:45		Lecture Theatre 1	Lecture Theatre 2	Lecture Theatre 3	Lecture Theatre 4	Lecture Theatre 1	Lecture Theatre 2
14:00		Computational Crystallography (CCG/PCG)	Developments in Instrumentation and Technology (BSG/CCG)	Multiferroics (PSG)	XRF General (XRF)	Reactivity in Crystals-2 (CCG)	Reactions in Macromolecular Crystals-2 (BSG)
14:15							
14:30							
14:45						CCDC Prize Lecture	
15:00	Coffee 15.00-15.30	Coffee 15.00-15.30				Coffee 15	
15:15							
15:30	Lecture Theatre 1	Sessions 15.30-17.00				Sessions 1	
15:45	Young Crystallographers 2	Lecture Theatre 1	Lecture Theatre 2	Computer Rm	Lecture Theatre 4	Lecture Theatre 1	Lecture Theatre 2
16:00		Dynamic Techniques (CCG/YC)	Radiation Damage (BSG)	dSNAP Workshop	New Developments in Instrumentation and TXRF (XRF)	Temperature-Dependent Crystallography (CCG)	Structure and Dynamics in Metalloproteins (BSG)
16:15							
16:30							
16:45							
17:00	Break	Break 15 minutes				Break 15 minutes	
17:15		Lecture Theatre 1			Lecture Theatre 4	Lecture Theatre 1	
17:30	Lecture Theatre 1	Exhibitors Forum 17.15-18.45			Exhibitors' forum (XRF)	Plenary (BSG)	
17:45	Young Crystallographers 3						
18:00						Lecture T	
18:15						BCA AGM	
18:30							
18:45		Dinner, exhibition and posters					
19:00	Young Crystallographers' dinner					Comfo	
19:15							
19:30							
19:45							
20:00						Conference dinne	
20:15							
20:30							
20:45							
21:00							

ugh 2009

ESDAY 22 April		Day 3 THURSDAY 23 April				Day 4 FRIDAY 24 April
Theatre 1		Lecture Theatre 1			Lecture Theatre 4	Frank Allen Symposium
(XRF/XRD)		Teaching Plenary (PCG) Martin Dove			XRF Keynote Method validation	
45-10.15		Coffee 9.45-10.15				
ns 10.15-11.45		Sessions 10.15-11.45				
Lecture Theatre 3	Lecture Theatre 4	Lecture Theatre 1	Lecture Theatre 2	Lecture Theatre 3	Lecture Theatre 4	
Crystallography Near the Edge (PCG)	Environmental Applications (Joint XRF/XRD)	Hydrogen Storage (PCG)	Data Acquisition for Time-Resolved Studies (BSG)	Understanding API Phase Transitions (IG)	Method validation (XRF)	
PCG AGM 11.45-12.30	IG AGM 11.45-12.30	Break 15 minutes				
		Sessions 12.00-13.30				
		Lecture Theatre 1	Lecture Theatre 2	Lecture Theatre 3	Lecture Theatre 4	
GMs/exhibition 11.45-13.30		Dynamics in Framework Structures (PCG)	Computation, Graphics and Animation (BSG)	Crystallography in the Pharmaceutical Pipeline (IG)	Portable Instruments (XRF)	
3.30-15.00		Close 13.30				
Lecture Theatre 3	Lecture Theatre 4	Frank Allen Symposium				
Monitoring Crystals, Crystallisation and Transformations-1 (IG/BACG)	Environmental Applications of XRF					
.00-15.30						
5.30-17.00						
Lecture Theatre 3	Lecture Theatre 4					
IG/YC Prize Lecture Monitoring Crystals, Crystallisation and Transformations-2	Trace Analysis (XRF)					
Theatre 1						
18.00-19.00						
ort time						
er 19.30 for 20.00						

Beamline I19 at Diamond

STATION I19, the dedicated 'small molecule' single crystal X-ray diffraction beamline, at Diamond successfully received synchrotron radiation into the optics hutch for the first time on the morning of Wednesday, 18th June, 2008. A screen dump showing the first beam at the first beamline diagnostic unit is shown in Fig. 1.

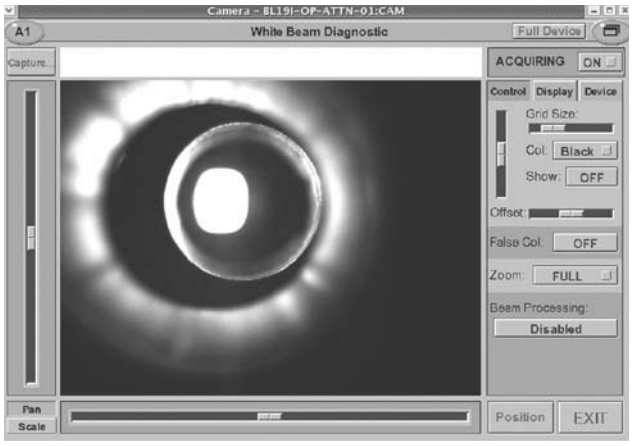


Fig. 1

The first light on the beamline represents a major milestone in the development of the station and the team at Diamond, some of whom are shown in Fig. 2, are to be congratulated on achieving this early success.

Fig. 2

Commissioning of the station will continue over the summer and early autumn, and it is hoped to welcome the first users during the late autumn or early winter period.

The next deadline for the structural science community is 1st October, 2008, which is the last date for the submission of proposals for beamtime for the allocation period from April to September, 2009. I19 should be fully operational for this allocation period and potential academic users are encouraged to submit proposals via the Diamond website:

http://www.diamond.ac.uk/ForUsers

Access for industrial users is expected to begin in late 2009.

More information about beamline I19 can be found via the web link:

http://www.diamond.ac.uk/I19

and potential users are encouraged to visit this site to see the range of experiments that can be undertaken.

Paul Raithby,
Coordinator of the User Group

David Allan,
Principal Beamline Scientist for I19

Harriott Nowell,
Beamline Scientist for I19

ISIS Second Target Station

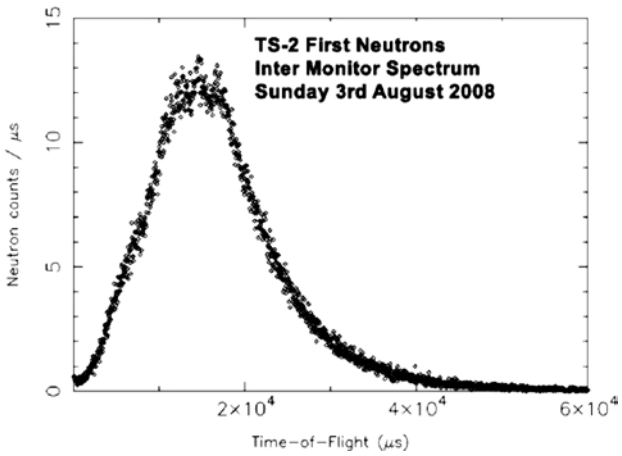
First Neutrons Created at the ISIS Second Target Station

THE UK's ISIS Second Target Station Project moved a major step closer to completion when the first neutrons were created in the ISIS Second Target Station. After five years of planning and construction, the first neutrons were detected by the Inter instrument at 1308 BST on Sunday, 3 August 2008.

"The first neutrons met all of our technical performance predictions and creating them is a significant milestone in the life of the facility and in the completion of the project," said Dr **Andrew Taylor**, Director of ISIS. "The Second Target Station builds on the success and expertise we have developed over the past 20 years at ISIS and allows us to move further into the areas of soft matter, advanced materials and bioscience. We will be carrying out fundamental research that will shape the technological advances of tomorrow."

The £145 million Second Target Station Project began construction in 2003. It will double the capacity and substantially increase the capability of the facilities already available at ISIS, which serves an international community of over 2,000 scientists.

Log files posted on the Web capture the excitement of this event:



After several hours work this morning scanning the shutter, the beamline suddenly came into alignment at 13:08 and neutrons were measured. So many neutrons flooded down the beamline that the gas tube detector (which had been temporarily removed from its shielding housing and taped to the sample table) went off scale. There are a lot of very relieved and jubilant people in the Inter control room (many who slept fitfully during the night wondering how to get the instrument working). More data is being collected in order to analyse the neutron spectrum and compare to calculations.

After further beamline checks, and some waiting for the accelerator, this is the first data to be published from the Inter instrument. Data fitting indicates that the intensities agree with calculations and that the distribution is consistent with the moderator temperature.

Cambridge Structural Database User Survey 2008

THE Cambridge Structural Database (CSD) currently contains crystal structure information for over 450,000 organic and metal-organic compounds determined by X-ray and neutron diffraction.

The number of structures added per year has increased from just a few hundred in 1965, when the CCDC was founded, to over 30,000 in 2007. This annual increase is set to continue rising.

In order to improve our database editing procedures and the overall information content of the CSD, it is important for us to have a clear view of:

- Which information fields in the CSD are most important to users?
- Can any of the current information be better expressed?
- Are there additional information items that should be added?

Therefore, the CCDC is carrying out a comprehensive and detailed CSD user survey. The outcomes of this survey will inform decisions about data content and how to maximise the use of the CSD and its benefit to users. We would like to gather opinions from all types of CSD users, from occasional users to those scientists for whom the CSD is a central part of their research activities. Since the CSD is used in a very wide variety of scientific disciplines, we also wish to obtain opinions from the broadest possible spectrum of our user base.

The closing date for return of the survey is 31 December 2008.

Everyone who completes the survey before this date will be entered into a prize draw.

The survey can be accessed at http://www.ccdc.cam.ac.uk/products/csd/user_survey/ and thank you for taking part.

The Spallation Neutron Source

The Spallation Neutron Source - a new resource for crystallographers

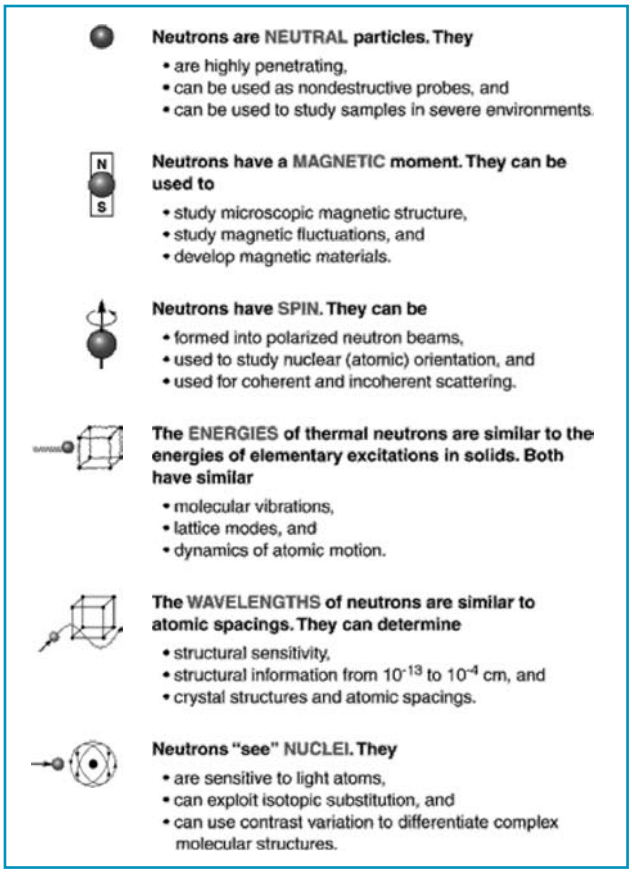
THE Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL) is developing an instrument suite that will provide crystallographers with increased capabilities. SNS is an accelerator-based neutron source that will provide the most intense pulsed neutron beams in the world for scientific research and industrial development. Completed in 2006, SNS is gradually ramping up power and is now up to 500kW in regular operation with the goal of achieving full beam power of 1.4 MW, Undergoing a rapid phase of expansion, the user program currently includes three SNS instruments, increasing to six by end of the September 2008 - February 2009 user cycle and to ten in late 2009.

How does SNS work?

Negatively charged hydrogen ions are produced in millisecond pulses at 60 Hz by an ion source and injected into a linear accelerator which accelerates them to very high energies, about 1 GeV, and near the speed of light, about 0.9c. The ions pass through a diamond film which strips off the pair of electrons, producing a stream of protons. These protons are compressed into microsecond bunches and directed to a heavy metal target, a rapidly circulating stream of 1.3 m^3 of mercury in the case of SNS. The resulting collision of protons with the mercury nuclei produces neutrons. These high energy neutrons are slowed to thermal energy by a series of moderators of water and liquid hydrogen, with the goal of producing a high flux of neutrons in the range of about 1 eV. These neutrons are guided to instruments where they are produce diffraction patterns when the neutrons strike the experimental sample.

The importance of neutron scattering to the scientific community was recognized by the awarding of the 1994 Nobel Prize for Physics to Clifford Shull and Bertram Brockhouse. Shull pioneered the use of neutron scattering at Oak Ridge to decipher the structure of materials, and Brockhouse found ways to use it in his Canadian laboratory to learn about the motions of atoms in materials. Neutron scattering is a powerful tool for determining how atoms are arranged in individual crystals. Moreover, neutron scattering can reveal the changes that occur in crystal structure as the material is exposed to changing pressures, temperatures, or other environmental variables.

Properties of neutrons useful for crystallographers



The high intensity neutron beams available at SNS will greatly expand the range of feasible study in material science. It will be possible to study much smaller samples, such as multilayer thin-film structures. How physical properties of materials are influenced by the reduced size in various dimensions of new materials' building blocks (e.g., nanoparticles, nanofibers, multilayer thin films) is a growing area of interest because such understanding offers a new avenue for tailoring material properties. Neutron scattering will impact this area, as well as the related and newly developing areas of self-assembly of complex crystals and processing of biomimetic structures. Several instruments at SNS will be of great interest to crystallographers.

Powder Diffractometer (POWGEN)

[Contact **Jason Hodges**, hodgesj@ornl.gov]

POWGEN is a versatile diffractometer designed to study polycrystalline materials and will enables users to collect typical Rietveld statistics in ~2 minutes from a 0.5-cm^3 sample with ~0.15% resolution at short d-spacings and <1% resolution for nearly all d-spacings of interest. Usually data collection will be performed over the d-spacing range 0.4 - 4 Å but adjustment of the phase of the bandwidth choppers in this instrument also

allows collection of diffraction data for d-spacings as large as 66 Å. The third-generation conceptual design of POWGEN and time-event mode of collecting data, allows users freedom to trade intensity for resolution at time of analysis and when using cycling sample environment equipment analyze data stroboscopically. This overall provides greater flexibility than most existing neutron diffractometers. In addition, this standard tool provides faster and higher precision than other diffractometers in the United States.

Scientific studies at this instrument encompass a wide range of novel materials. These include, but are not limited to, structural studies of such as high-Tc superconductors, metal-insulator phase transitions, charge and orbital ordering transitions, magnetic materials, zeolite, aluminophosphate and metal-organic frameworks; metals and semiconductors; dielectrics, ferroelectrics, and thermoelectrics; and ab-initio structure solutions of polycrystalline materials such as pharmaceutical compounds. In addition, POWGEN is capable of acquiring refineable data sets in rapid data collection mode, making it an ideal instrument for parametric studies and time-resolved in situ studies of the electrochemistry of catalysts, ceramic membranes, hydrogen storage materials, and charging and discharging of battery materials. POWGEN will begin commissioning in late 2008 and join the general user program in 2009.

Single Crystal Diffractometer (TOPAZ)

[Contact **Christina Hoffmann**, hoffmancm@ornl.gov]

TOPAZ will address problems and greatly expand the range of materials explored in chemistry, earth sciences, materials science and engineering, solid-state physics, and biology. This instrument is designed to perform elastic scattering experiments under controlled environmental conditions to probe material structures and responses. Enabling to measure the same single crystal sample with neutron as with X-ray diffraction was the guiding design principle of TOPAZ.

Data will be collected on samples of 0.1 mm^3 or less. Resolution is such that an average unit cell size of [50 x 50 x 50] $Å^3$ for compounds of moderate complexity can be easily accommodated. This includes inorganic large and porous framework and guest-host materials, metal (in-) organic cluster and molecular compounds, and organic arrangements of interest to biology and medical applications. Materials to be investigated using TOPAZ include functional materials of the high Tc superconductor perovskite structure family. Potential future high-density, three dimensional storage materials on molecular basis, some known as single molecule magnets have raised much interest but could mostly only be studied in polycrystalline samples with neutron diffraction. Also of importance are catalytic precursors, metal hydride materials for potential hydrogen storage applications, and organometallics. Neutron single crystal diffraction is uniquely positioned to decipher the pathways and bonding of hydrogen in presence of heavy metal ions as it is sensitive to both classes of elements in comparable quantity. Simultaneously, the magnetic ordering and structure can be investigated exploring the fact that neutrons carry a magnetic moment. This allows magnetic and spin density studies in magnetically interesting materials. TOPAZ will begin commissioning in 2009.

Macromolecular Neutron Diffractometer (MaNDi)

[Contact: **Leighton Coates**, coatesl@ornl.gov]

The SNS macromolecular diffractometer (MaNDi) will be a state of the art high-resolution macromolecular crystal diffractometer. Optimized for rapid data collection from large structures, MaNDi will achieve 1.5 Å resolution from crystal volumes between 0.1-1.0 mm^3 with lattice repeats in the order of 150 Å. The instrument will use a decoupled hydrogen moderator for optimal resolution and separation of Bragg peaks. The design utilizes a 30 m flight path and a variable wavelength bandwidth of 2.16 Å to accommodate different types of experiments. This bandwidth variation is achieved by the use of three disc choppers in the incident flight path. With larger crystals (> 1 mm^3), it will be possible to obtain useful data in the resolution range 2.0-2.5 Å for unit-cell repeats of up to 300 Å, a revolution in neutron macromolecular crystallography (NMC). Simulations predict experimental duration times of between 1 and 7 days, which will revolutionize NMC for applications in the field of structural biology, enzymology and computational chemistry. MaNDi will begin commissioning in 2012.

Access to ORNL neutron scattering facilities

Research proposals for experimental access to the Spallation Neutron Source and High Flux Isotope Reactor are being received from a worldwide research community. At least 75% of the beam time of each instrument is available through the general user program; the external peer review procedures for all users take as their starting point the criteria proposed by the International Union of Pure and Applied Physics with the paramount criterion being scientific merit. All user proposals will have a feasibility and safety review carried out by facility staff. There are two proposal review cycles annually; each cycle includes proposals for both HFIR and SNS instruments.

Several instruments, in addition to those noted previously, may be of interest to the crystallographer: the high pressure diffractometer SNAP and the VULCAN engineering diffractometer at SNS, the Neutron Powder Diffractometer and the Neutron Residual Stress Mapping Facility at HFIR. Additional details about the ORNL neutron scattering program, including descriptions of instrument capabilities and experimental proposal submission process, are available at http://neutrons.ornl.gov. Please contact neutronusers@ornl.gov or call 865-241-3675 for more information.

Jason Hodges and Al Ekkebus,
Neutron Scattering Science Division, Oak Ridge National Laboratory

ACA President Marvin Hackert (center) and his wife Bretna (left) listen to an explanation of the POWGEN3 powder diffractometer by Ashfia Huq (right) during the ACA tour of SNS. (Photo credit: Curtis Boles, ORNL)

Andrew Richard Lang FRS

Andrew Richard Lang FRS 1924-2008

DISTINGUISHED for his pioneering studies in X-ray diffraction physics, especially for his developments of the techniques of X-ray topography, Andrew Lang will be greatly missed by many for his detailed knowledge of crystal physics. His topographic techniques image one- and two-dimensional imperfections in crystals: such as dislocations, stacking faults, growth-sector boundaries and ferromagnetic domains.

The projection topograph is often called the 'Lang method'. For the past fifty years, Lang's methods have been widely used in the assessment of crystals for the electronics, diamond and other industries. An example of an application using synchrotron X radiation was his measuring and mapping of the relative lattice constant across a large (5 mm) synthetic diamond, to an accuracy better than one part per million.

Lang had studied many types of X-ray diffraction phenomena: X-ray moiré fringes, the first direct observation of a refractive index for X rays greater than unity, and Borrmann-Lehmann fringes. His most important discovery in this category (with Norio Kato in 1959) was that of Pendellösung fringes in wedge-shaped perfect crystals. The fringe spacing provides a determination of absolute structure amplitudes.

He had also made significant discoveries using other techniques, especially electron microscopy and cathodoluminescence, separately and in combination with X-ray topography; and he had also studied a wide range of crystalline materials, including metals, semiconductors, quartz and diamond. His first published paper (1947) was on the crystal structure of a crossed-chain potassium soap.

Andrew Lang's work exhibited sustained innovativeness, craftsmanship in experimentation, and perceptiveness and thoroughness in the analysis of experiments. The topographic images which he produced were of the very highest quality and were often exceedingly beautiful.

Andrew Lang was born in 1924 at St Annes-on-Sea, England. He obtained a First-Class Honours London External BSc in Physics at Exeter in 1944, a London External MSc in 1947 and a Cambridge PhD in 1953. He had worked in industrial research in England (Lever Brothers and Unilever Ltd) and in USA (Philips Laboratories, Irvington-on-Hudson, NY). He taught at Harvard University (1953-1959). He had been at the University of Bristol ever since 1960, where he had been promoted to Professor of Physics in 1979.

He had been Chairman of the American Crystallographic Association Apparatus & Standards Sub-Committee (1957-59); Consultant, Smithsonian Astrophysical Laboratory (1957-59); Associate Editor, Journal of Crystal Growth (1966-90); and Member of the UK SERC Synchrotron Radiation Facility Committee (1976-78). In 1964, he was awarded the Charles Vernon Boys Prize of the Institute of Physics and the Physical Society. He was elected a Fellow of the Royal Society in 1975.

Moreton Moore

WE are saddened to learn of the untimely death of Andrew Parkin on August 28th.

Along with making imaginative contributions to crystallography, Andy was one of the foundersof the Young Crystallographers Group. A full obituary will appear in the December issue of Crystallography News.

ACA Meeting Knoxville

THE stern warning "Possession of firearms in this building is prohibited" on the entrance doors of the Knoxville Convention Center was an immediate reminder that we were no longer in the genteel surroundings of York. Once inside, however, we found ourselves in a spacious environment that featured interesting works of art. We enjoyed pleasant views outside to a campus that included a pond and a cascade created for a World's Fair in 1982, a grove of magnificent magnolias laden with huge creamy-white blossoms, and a statue of Rachmaninov, who gave his last-ever concert at the University of Tennessee nearby.

As usual, there was a substantial British presence at this meeting. The pre-conference workshop on "Magnetic Structure Analysis by Neutron Diffraction Techniques" was introduced by **Andrew Wills**, while that on "Wise Use of Dose: Structure Solvability vs. Structure Integrity" featured no less than three presentations by **Elspeth Garman**. The anchor General Interest session at the end of the meeting, where exciting big-name speakers are thrown in to discourage early departures, included a lecture on "Cluster Analysis in Crystallography" by **Chris Gilmore**. In between we had major contributions from Diamond and ISIS scientists **David Allan**, **Elizabeth Duke** and **Richard Ibberson**.

The unifying theme of the Transactions Symposium on macromolecular crystallography, "Structure/ Function Studies", should have conveyed a sense of familiarity to anyone who had attended the BCA meeting in York. Providing a sign of the health and confidence of our scientific discipline, the ACA chose to put the emphasis on "Complementary Methods", teaming X-ray crystallography with neutron crystallography, single crystal spectroscopy, small angle scattering and NMR.

As befits a meeting with so much input from Oak Ridge, one of the sessions on the first day covered "Structure and Dynamics of Hydrogen Bonded Systems." **Tom Koetzle** gave the initial overview. Tom started with a salute to the early pioneering work on neutron diffraction at Oak Ridge and Harwell which elucidated the hydrogen bonding in ice and KDP. He described how the more intense beams available from high flux reactors and pulsed sources enabled larger systems such as sugars, amino acids and even some proteins to be studied; and he pointed out the exciting prospects for research using still more powerful sources like the new SNS at Oak Ridge. Tom was kind enough to mention my poster describing a neutron diffraction study of andrographolide. As a result I had more customers than usual and felt less like a forlorn Big Issue seller on a bad day. The next talk, by **David Allan**, showed how pressure changes can be used to change the hierarchy of intermolecular forces in molecular crystals. The information obtainable from difficult in situ X-ray diffraction studies, often inadequate to establish hydrogen atom locations, can be augmented with high-pressure neutron powder-diffraction data. **Gerard Harbison** introduced the additional techniques of solid-state NMR and quantum chemical methods, detailing the measurable parameters that probe the state of a strong hydrogen bond and demonstrating the validity of calculations provided that sufficient terms are taken into consideration.

After a welcome coffee break, where an unusually wide variety of tea bags catered for all types of tea drinkers, this session resumed with a focus on dynamics. **George Reiter** presented the results of a Deep Inelastic Neutron Scattering (DINS) study on a superprotonic conductor to analyse the Born-Oppenheimer potential surface. Such work was only possible on the Vesuvio instrument at ISIS, although a new instrument is coming at Oak Ridge. Srinivasan Iyengar tamed the ferociously difficult problem of calculating dynamics for systems ranging in size up to soybean lipoxygenase 1 by developing a quantum wavepacket molecular dynamics method. **Matthew Hudson** investigated a highly anisotropic proton conductor by neutron diffraction and INS spectra of single crystals, the latter making good use of the TOSCA spectrometer at ISIS. Finally, **Paula Piccoli** reported the structure of a dichloride hexahydrate cube from neutron diffraction data, clearing up an ambiguity left unresolved by the X-ray structure and providing insight into the solvation of halide ions.

In the afternoon session on "Solid State Transformations and Reactions" **Bruce Foxman** gave a characteristically thought-provoking lecture. He began by scolding some recent entrants to the field for muddling the terminology that had been carefully defined by the original investigators. He distinguished topotaxy (the orientation of the product crystal correlates with that of the reactant), topochemical reaction (the chemical nature of the product is governed by the structure of the reactant crystal), and Lonsdale twinning (daughter crystals are in a twin relationship to the parent). He went on to discuss and give recent examples for four types of solid state reactions: (i) one-phase topotactic reactions, (ii) topotactic reactions involving two or more phases, (iii) single crystal to polycrystalline product, (iv) single crystal to amorphous product. **Ross Angel** moved the emphasis from reactions to structural phase transitions, in which the space-group symmetry of one phase is a subgroup of the symmetry of the other. **Art Schultz** presented neutron diffraction results on a finely balanced

structural phase transition in which isotopic H/D substitution is sufficient to alter the Jahn-Teller distortion and trigger the transition.

As is typical also at BCA meetings, the session on "Challenges in Industrial Crystallography" covered a fascinating spectrum of topics from tungsten ores, microcrystalline materials from the petroleum industry and cements through to drug design. It included one contribution by **Scott Misture** of a methodological nature, showing how productive the charge flipping approach can be in providing at least partial structures from low-quality powder diffraction data on inorganic structures. Inevitably, as a pharmaceutical crystallographer I was most stimulated by the three lectures on drug design. **Ravi Kurumbail** presented work on MAPKAP-2 kinase, which initially appears to be an attractive target because it has an important role in inflammatory disease and it readily yields well-formed crystals. However, the diffraction resolution has not extended beyond 3.0 Å. Although many protein constructs have been tried, yielding such oddities as fcc symmetry with Z = 96, none have improved the resolution. Even so, it has been possible to obtain useful electron density maps that facilitated the design of inhibitors with enhanced affinity and specificity. **Melissa Harris** presented experiences from three projects where obstacles had to be overcome in protein expression, purification and crystallisation. Finally, **John Spurlino** described the use of crystallography as a screening tool. This approach starts with small drug-like fragments which can easily seek out a binding site in high-throughput experiments. For those fragments that bind well, structural information enables them to be elaborated or linked in order to produce candidate drugs.

On the final morning attention was again directed beyond the narrow confines of crystallography in a session on "How Structures are Used by Others." **Jane Richardson** began by listing many ways in which macromolecular structures are used and how best to make them accessible for each use. Thus teaching is facilitated by copious annotation of the PDB files and provision of links to graphical material. Biomedical research depends on a proper match between chain identifiers and residue numbers in publications and those in PDB files; it can be thrown into confusion by the arbitrary assignment of "mystery density" as water. Comparison of structures, e.g. before and after ligand binding, depends critically on consistent interpretation of features in both structures. As a starting point for building related structures in fields such as cryo electron microscopy, SAXS, molecular replacement and homology modeling it is important that highly mobile or uncertain parts of the structure can be identified and not relied upon. Computational biologists want the opposite: every atom present, as a "best guess" with suitable sterics and geometry even if unseen. Finally, methods developers want experimental structure factors and phases to be deposited, ready for the testing and application of improved techniques in the future. MolProbity is available (http://molprobity.biochem.duke.edu) as a tool for validation of protein and nucleic acid structures. **Roland Dunbrack** reported comprehensive statistical analyses of structural features in the PDB: side chain rotamers and multi-conformational side chains, backbone-dependent rotamer libraries and adjacent-residue-dependent Ramachandran maps, and biologically relevant interfaces. The methods developed will be made available to the crystallographic community. After the coffee break there were three talks on specific application areas: **Nick Grishin** on dramatic changes of protein structure in evolution, **Donald Hamelberg** on slow conformational switches in proteins, and **Jeffrey Gray** on the computational prediction of protein complexes.

Participants departed in an optimistic frame of mind. Next year's meeting will take place in Toronto in its more usual late July time slot (July 25-30).

Carl Schwalbe

York 2008

Young Crystallographers Meeting - 7-8th April 2008

THE Young Crystallographers meeting was held on 7 - 8th April 2008, prior to the Spring Meeting of the British Crystallographic Association (BCA) at the University of York. Since a general overview of this meeting has appeared in the June issue, here is a considered view of two presentations that attracted special interest.

High Pressure Structural Studies of Energetic Materials

Iain Oswald, University of Edinburgh

Iain began by saying that energetic materials release heat and / or gaseous products at a high rate on a certain stimulus such as heat or shock. These materials can be classified as explosives, propellants etc. Their crystal form determines characteristics such as detonation velocity and reactivity. During operation the extreme conditions may lead to phase transition.

Iain used both x-ray and neutron diffraction to perform high pressure studies on two widely used nitramine-based explosives HMX and RDX. He discussed high pressure studies of the beta polymorph (one of four) of HMX. At high pressure there are phase transitions and changes in molecular geometries. Ian went on to describe his work on RDX - a widely used military explosive containing nitro groups. RDX has been used since World War 2 and has two known polymorphs at ambient pressure. Previous X-ray studies had identified an alpha to gamma phase transition in which its space group, Pbca, didn't change. The RDX was analysed up a pressure of ~8GPa. The phase transition of α - γ was observed at ~4GPa and the structure of the γ-form was determined for the first time at 5.1GPa. Powder neutron diffraction showed that all the d-spacings contract upon application of high pressure, and that Pbca is not the correct space group. Attention turned to single crystal X-ray work. The space group was found to be $Pca2_1$ and there are two molecules with different conformations.

Information on the structure of such phases can lead to safer composition of explosives that are less sensitive to accidental initiation.

John Kaniuka and **Mark Farnworth**
Pilkington Group Limited

Evidence for a New Phase of Methane

Helen Maynard, University of Edinburgh

Methane is the most abundant organic molecule in our universe and is found at high pressures in the outer planets and satellites of our solar system. However, neither of the known high pressure forms of methane has been completely solved. Helen used a diamond anvil cell to perform high pressure x-ray crystallography and optically observed a phase change during single crystal growth at ~370K and 8GPa. The single crystal set-up of 9.5, SRS, Daresbury was modified to allow in situ high pressure and high temperature data collection. However, a breakdown meant that data collection was not possible. The implications of a new phase of methane would suggest that it accounts for 10-15% of Neptune's mass below the upper H-He layer.

John Kaniuka

Industrial Group sessions

PRELIMINARY reports on these sessions by bursary recipients have appeared in the June issue. Definitive accounts in greater depth are published here.

APPLIED CRYSTALLOGRAPHY SHOWCASE 9TH APRIL 2008

Industrial Group Young Crystallographers Prize Talk High Pressure Structural Studies of Energetic Materials

Iain Oswald, University of Edinburgh
(Summarised above)

Direct Correlation between Ferrite Microstructure and Electrical Resistivity

Judith Shackleton, University of Manchester

Ferrites are used for transformer cores and operate at ever increasing frequencies. Magnesium-zinc ferrites are toroid shaped. Their density is 93% of theoretical. Secondary Electron Microscopy (SEM) - Energy Dispersive

Spectroscopy (EDS) shows only a single phase with less zinc at the surface. Judith went on to show the results obtained from Tomographic Energy Dispersive Imaging (TEDDI) which produces 3D scans of the lattice parameters calculated using the Le Bail full pattern method (TOPAS software).

The lattice parameters were found to be larger on the outside of the samples. Resistivity measurements were also carried out and the variation in resistivity found to be about 10 ohm/cm over a length of approximately 800 microns. This is a similar length scale to that found by TEDDI.

Protein Structures in Drug Discovery - from Fragments to Macromolecules

Judit Debreczeni, Astra Zeneca

Judit began by saying protein crystallography is one of the most powerful experimental techniques to aid the design and development of drug molecules in the pharmaceutical industry. It has been able to adapt to the ever-changing needs of the business, shifting focus on different drug targets and approaches. High throughput environments produce automation of as many steps as possible to solve structure. Robots are used for crystallisation and also for the crystallography e.g. crystal screening and data collection.

High impact areas are studies for protein-ligand, protein-fragment and protein-protein interactions. Crystallography has become a widely used technique to identify small building blocks, fragments of potential drug compounds.

Applications of X-ray Diffraction and Mapping in the Glass Industry

Mark Farnworth, Pilkington Group Limited

Mark began by saying that glass is not traditionally thought of as a good material for analysis by XRD. However, the glass making raw materials and the refractory materials that make up parts of the Float Glass Process are crystalline and their nature needs to be understood. In addition, many of the ultra-thin coatings deposited onto glass are also crystalline. Mark described how the non-crystalline (or glassy) content of refractory material is determined using mixtures of fused silica and quartz.

Thin layers of material deposited onto glass can be very crystallographically oriented (texture). Mark showed how he uses pole-figure measurements to determine the strength of Ag(111) columnar orientation in thin silver layers. In conclusion, Mark described how XRD maps are obtained from 10cm x 10cm coated glass plates. For a selected Bragg reflection, maps of peak area (a function of texture and layer thickness), peak position (a function of strain) and peak FWHM (a function of crystallite size) are measured.

Application of X-ray Diffraction and other Analytical Techniques to Pharmaceuticals

Suzanne Harte, SAFC Pharmorphix

Suzanne explained why it is necessary to have a thorough understanding of the solid state properties of potential drug candidates. A wide range of complementary analytical techniques are used. By utilizing thermal, optical, spectroscopic and diffraction methods it is possible to gain a much greater comprehension of pharmaceuticals than can be achieved from one isolated technique. When a material first arrives, spectroscopic Nuclear Magnetic Resonance (NMR) is used. High Performance Liquid Chromotography (HPLC) assesses purity and Aqueous Thermodynamic Solubility. A D8 GADDS instrument is used as a screening tool. Suzanne explained that it is a reliable method for identifying different forms of materials.

Application of X-ray Diffraction Within Forensic Science

David Rendle (ICDD), Visiting Fellow (Cranfield Forensic Institute)

David began by saying that XRD has been used in forensic science laboratories around the world since before the middle of the last century. Its use sixty years ago was generally restricted to basic qualitative analysis. Modern day applications have the added advantages of the availability of high speed computer-controlled diffractometers and data processing software. Intelligence gathering - the recording and collation of detailed analytical results in a database - whilst time-consuming and tedious, is essential to the forensic scientist for estimating the significance of the results and for indicating trends over a period of time. David concluded by saying the analysis of street drug seizures by XRD yields not just the identity of the drug, but also the identities of the adulterants or excipients used to dilute the drug.

Small is Smart - Wed 9th April

Powder Diffraction of Nanomaterials

Steve Norval, Intertek MSG

Steve began by saying that, for the most part, techniques for phase identification, quantification, microstructure, in-situ monitoring etc are well established and were applied for decades before “nano” became fashionable. Heterogeneous catalysts are good examples of nanomaterials that have been around longer than X-ray Diffraction. Adsorbants and pigments were also being used before the nano-revolution. For a material to be described as ‘nano’ the crystal size in at least one direction is generally considered to be less than 100 nanometres.

Stephen presented information about rutile titania, commonly used as a pigment in many materials. Crystallite size distributions determine its physical nature; for example, 'white' pigment is usually ¼ micron in size whereas for sun-block (to scatter UV light) the pigment size is approximately 25 nm. Nano-composite clays have enhanced mechanical properties and usually contain montmorillonite clays.

X-ray In-Plane Scattering of GaN Nano-columns

Christopher Staddon, University of Nottingham

Gallium nitride is a wide band gap semi-conductor and is used in blue emission lasers, high density storage media and high power FET's. Chris said that a major problem with this material is the lack of suitable substrates, which in turn results in high defect densities in the material and hence less than ideal devices. To avoid this problem, isolated GaN nano-rods are grown by molecular beam epitaxy (MBE) on sapphire substrates using aluminium nitride buffer layers of appropriate thickness. Chris said that he has used in-plane scattering, to determine the mean value of the in-plane lateral dimensions, orientation and quality of the GaN nano-columns. The experimental data was collected from two independent in-plane reflections - (200) and (300). A bi-modal size distribution was discovered.

Solving Crystal Structures of Zeolites using Powder Diffraction and Electron Crystallography

Chris Gilmore, University of Glasgow

Chris discussed zeolite structures and the challenges involved in solving them, particularly in early stages of synthesis and characterisation when samples are poorly characterised. The crystallite sizes are often so small that peak broadening makes the powder patterns hard to deconvolute. Chris has used density building functions and density histogram matching techniques to solve a number of structures ab-initio using both electron diffraction data in 2- and 3- dimensions and powder data.

A low resolution structure is first generated using structure factors and more direct methods. They are analysed using density building functions. The most promising phase sets are subjected to entropy maximisation and then likelihood and density histograms are used to select the optimal phase sets.

Big is Beautiful - Wed 9th April

Analysis of Weld Residual Stresses in Prototype Engineering Components and Structures using Pulsed Neutrons

Supriyo Ganguly, Open University, Milton Keynes

Suprivo began by saying weld stresses can result in fatigue so there is a requirement to investigate damage tolerance. This has been carried out through the WELDES project. Welding is probably more cost effective than traditional riveting. The factors which control fatigue initiation and crack growth are well known but it is important to understand the actual crack growth mechanism. Two aluminium alloys have been investigated: 2024-T351 and 7150-T651. Wing structures are machined and welded. The size and measured density define the appropriate residual stress measurement that is used. Time-of-Flight Diffraction with a 14-40 millisec range and Pulsed Neutron Strain Scanning are techniques that have been used.

Residual Stress Measurements of gas turbines components by X-Ray Diffraction

George Bibby, Rolls Royce

Residual Stress measurements are made to effect the quality control in material supply. They are used to assess the 'equivalence' for process change in manufacturing and investigate the condition of material after rig tests and service running. George discussed the method of manufacture of cast / wrought discs and the architecture of gas turbines. They have analysed low pressure compressor blades, high pressure turbine discs and engine mounts. Components can be immersed in hydrofluoric acid to remove surface material prior to analysis. Sources of error include 'psi splitting' due to the presence of 'out of phase' shear forces and shadowing due to material on the sample that impinges the diffraction cones. George concluded by saying that providing the methodology is right then X-ray diffraction is a very satisfactory method for measuring residual stress.

JEEP: The Joint Engineering, Environmental and Processing Beamline at Diamond Light Source

Michael Drakopoulos, Diamond Light Source Limited

Advances in engineering disciplines are often driven by development of new materials, but also by incremental progress of existing technology. In both cases, reliability and reproducibility of results are key. Michael informed everyone that Diamond Light Source is constructing a beamline dedicated to Engineering Science to become operational by October 2009. It will be the first engineering beamline in the UK. The beamline will focus on diffraction and imaging-based methods for the study of structural properties of engineering materials in static and dynamic conditions. Imaging and tomography establish a picture of structural properties on the microscopic scale. To penetrate deep into engineering materials, JEEP will provide synchrotron radiation at X-ray energies between 50keV and 115keV (0.28 to 0.08 angstroms). The attenuation length for iron is a few millimetres; at 3x the attenuation length the signals are still strong so measurement through 1cm of steel will be possible.

Industrial Group Plenary Lecture
- Wed 9th April

X-ray Diffraction on Mars

Rob Delhez, Delft University of Technology

Rob began by saying that space research is driven by curiosity or scientific interest in many fields. If there is life in space, we need to find organic molecules. Humankind has found 150 molecular species in the space environment, 230 including isotopes. Of these, 50 species have been found in comets. For meteorites approximately 3% of the mass is organic of which 20% is soluble and 80% is kerogene. Unlike the Earth, Mars has no tectonic plates and no magnetic field. Its surface temperature is 0 to -15C during the day and -85C at night. Meteorites have provided evidence for minerals, gases and organics. Mars Landers have told us about the atmosphere and temperatures. Orbiters have provided pictures and 3D images (visible, ultra-violet, infra-red, spectroscopy, magnetic) of the surface. They have provided evidence of landslides, dust storms and clouds.

A Mars Science Lab will contain X-ray Diffraction and X-ray Fluorescence instruments. The mission has several goals including the search for life, climate and geology investigation and human exploration. A paper was published - Powder Diffraction 20(2) June 2005, 128-133. The XRD instrument has a micro-focus X-ray tube with a cobalt anode producing a spot size of 50 micrometres. There is also the ExoMars program which has objectives for entry, descent, landing, surface mobility via a rover and access to the sub-surface using a drill which will penetrate down to 2m.

Projects are also planned to investigate organic materials. Some minerals/structures are formed by algae e.g. banded iron formation (BIF) and stromatolites (calcium carbonates). Others can host organic molecules e.g. iron hydroxides and clay minerals. Some can absorb positively charged species onto their internal surfaces e.g. amino acids. Rob went on to explain some of the requirements of a Mars XRD instrument. For 'space qualification', it would need to withstand accelerations up to 40g, a vacuum of 10-14 bar and it would need to be sterilised before launch. Camera-type diffraction is required and the diffractometer would need to have no moving parts. For the X-ray source, there are two options: a radioactive source where radiation protection would need to be built in or a tube where high voltage risks would need to be considered. A 2D position and energy sensitive detector is required. The presentation was excellent and illustrated a very practical application of X-ray Diffraction at the forefront of science and technology.

Mark Farnworth, Pilkington Group Limited

PDF versions of the full Autumn meeting reports can be downloaded from the Industrial Group's web site at:-
http://bca.cryst.bbk.ac.uk/bca/ig/ig.htm

Puzzle Corner...
...JUNE ANSWER

THE winner this time is Derry Jones. In the historic photo Mike Glazer had already identified the four men at the back from left to right as: R. C. (Bob) Evans, W. H. (Will) Taylor, J. D. Bernal and W. Wooster. Derry suggested that the female crystallographer was **Dorothy Crowfoo**t (married to Hodgkin in 1937), although he felt that the hair didn't look exactly right. Nobody has come up with a positive identification for the two in the front row.

The distinguished crystallographers with their correct job titles and a contribution to crystallography are:

William H. Bragg *Professor of Mathematics and Experimental Physics, Adelaide Univ.* Ionisation spectrometer

Auguste Bravais *Professor of Physics, Ecole Polytechnique* Space lattices

Georges Friedel *Director, Ecole Nationale des Mines* Friedel's Law, I(h k l) = I(-h -k -l)

Herbert A. Hauptman *Researcher, U.S. Naval Research Laboratory* Direct methods

Dorothy M. Hodgkin *Chancellor, Bristol University* Penicillin and insulin structures

William N. Lipscomb *Clarinetist, Minneapolis Symphony Orchestra* Boron hydride structures

William Hallowes Miller *Professor of Mineralogy, University of Cambridge* Miller indices h k l

Louis Pasteur *Dean of Science, Lille University* Separation of crystals of left- and right-handed enantiomers

Arthur L. Patterson
Senior Member, Institute for Cancer Research Interatomic vector synthesis

Neils Stensen
Vicar Apostolic of the North Interfacial angles in crystals of a given substance are constant

The only one without a job title is Robert O. Gould. Many of you may know that Bob is a part-time vicar who helped to feed poor people in Edinburgh while he was editing Crystallography News, and relative to most parts of Britain Edinburgh is the North; but Bob is far too modest to take on a grandiose title like Vicar Apostolic of the North.

News from the Groups

News from the Groups

CCG AUTUMN MEETING 2008:
"New Methods in Chemical Crystallography"
Sponsored by **Oxford Diffraction**
Wednesday 12 November 11.00 - 17.00,
Newcastle University.

This meeting will feature a range of speakers presenting current research in X-ray crystallographic techniques. The topics include: application of charge-flipping solution methods to modulated crystal structure data; parametric diffraction studies; combining X-ray structural data and computational chemistry; and modelling diffuse X-ray scattering from disordered materials. Confirmed speakers include:

Dr Trixie Wagner (Novartis Pharma AG, Switzerland)
"Advancing into higher dimensions - a practical approach to modulated structures".

Dr Lynne Thomas (University of Glasgow)
"Bragg scattering and beyond...Getting more from diffraction patterns".

Dr Natalie Fey (University of Bristol) "*Building knowledge bases from structural data".*

Industrial Group of the BCA: Pharmaceutical Special Interest Group Autumn Meeting

THE Pharmaceutical Special Interest Group Autumn meeting will be held at AstraZeneca, Charnwood Site, Loughborough, on the 5th November 2008.

Pharmaceutical SIG - 5th November 2008, chaired by **Anne Kavanagh**, **Brett Cooper** and **Matthew Johnson**.

Speakers at this meeting will be:

Robert Docherty (Pfizer)
The Future Application of Computational Methods in Solid Form Selection.

Graeme Day (University of Cambridge)
Discovering and Understanding New Crystal Forms: Guiding Experiments by Crystal Structure Prediction and Modelling.

Frank Leusen (University of Bradford)
A Major Advance in Crystal Structure Prediction.

Xue Wang (University of Leeds)
Crystal Morphology: Measurement, Modelling and Closed-loop Control.

Amy Robertson (AstraZeneca)
Process Analytical Technology Applications in Crystallisation Development.

Claire Thompson (GlaxoSmithKline)
Pharmaceuticals in a State of Disorder - But How Much?

Matt Tucker (ISIS)
Looking Beyond the Bragg Peaks with Total Scattering.

Travel directions by air, public transport and road, see:
http://bca.cryst.bbk.ac.uk/bca/ig/Trav08AM.htm

ORGANISERS:
Dr. **Matthew Johnson**: matthew.6.johnson@gsk.com
Dr. **V. Brett Cooper**: brettcooperhome@btinternet.com

Full details can be found at:
http://bca.cryst.bbk.ac.uk/bca/ig/meet08AM.htm

Report - XRD and MINERALS

British Geological Survey, Keyworth, Nottingham, 15th May 2008

BACK in the splendid BGS meeting auditorium, after enjoying Martin Gill's Birthday party celebrations the night before, the audience (not all of whom were at the party) was treated to a morning of presentations concerning industrial applications of X-ray Diffraction that included a wine tasting session.

The first speaker was **Caroline Kirk** from the Department of Chemistry, Loughborough University and the Department of Mineralogy, Natural History Museum, London and her presentation was entitled;
Structural Studies and High Temperature Properties of Bismuth Vanadate Sillenites; Man-made Minerals.

Caroline explained that phase formation in the bismuth-rich end of the Bi_2O_3-V_2O_5 system had been investigated and an extensive solid solution of sillenite related phases found four temperature dependant polymorphs. These materials are metastable and have unusual high temperature behaviour not observed in other sillenite phases.

High temperature X-ray diffraction, differential thermal analysis and impedance spectroscopy were used to characterise their high temperature properties. Structural studies of this family of bismuth vanadate sillenite materials were performed using neutron diffraction data to try and correlate the metastable nature of these materials to their structure. The structure of these materials is related to the sillenite structure, but a new model was required to take into account Bi3+ partially occupying the tetrahedral site along with V5+ and the non-stoichiometric nature of the oxygen lattice.

It was found using DTA and impedance spectroscopy that conductivity also varied with changes in temperature. X-ray diffraction experiments with increasing temperatures show that at 750°C the system transforms from BCC to FCC and on cooling from FCC to BCC at 650°C.

On cooling there are 2 BCC phases, which are metastable - why? Perhaps there is a problem with the structure model.

From neutron diffraction, high temperature XRD and DTA studies it was proposed that the disordered nature of the oxygen lattice is the key to their metastable nature and different cations are being investigated.

The next speaker was **Stephen Cairn**s from the WestCHEM, Department of Chemistry, University of Glasgow Department of Chemistry, Loughborough University and the Department of Mineralogy, Natural History Museum and his presentation was entitled;
Synthesis and Structural Studies of the Ettringite Group of Minerals.

Stephen discussed the importance of the understanding of the structure of etteringite and thuamasite in the application of civil engineering, specifically in the complex reactions of concrete. The formation of ettringite in setting concrete leads eventually to the formation of cracks; a famous example of this being the collapse of the Kepong Bridge in Malaysia.

The ettringite group of minerals have the general formula Ca6X2(SO4)3(OH)12.26H2O, where X = Al, Cr, Fe and Si and the sulphate group can be substituted by carbonate or borate groups. Although the minerals are compositionally closely related, structurally they are different. Ettringite and thaumasite are the most important members of the group as they form in cement pastes, mortars and concretes. This has major implications for the construction industry and further study into the structure of these compounds is required to address this issue.

A systematic study of the structural chemistry of ettringite, thaumasite and their related phases is being undertaken using powder x-ray diffraction, single-crystal x-ray diffraction (for which a novel method of growing single crystals was developed) and neutron diffraction of both natural and synthetic s amples. A detailed phase diagram exploration is one of the aims of the group's programme and includes analysis of synthetic analogues of these mineral phases, so that migration of metal ions from the old to new structures can be studied and investigations into potential solid solutions that may exist between different members of the ettringite group.

The third speaker of the morning was **Eric Ferrage** from Laboratoire HydrASA, Université de Poitiers who presented:
Investigation of Smectite Structure Heterogeneities: an XRD Profile Modelling Approach.

Eric explained that smectite clay minerals in sedimentary rocks play an important role in water mobility and retention in soil or in waste repositories because of its high ability for cation and water retention. As a function of relative humidity and under non-saturated conditions, smectite shows a stepwise hydration behaviour corresponding to the intercalation of 0, 1 or 2 discrete sheets of water molecules in its inter-layers.

However, heterogeneities of charge location (between octahedral and tetrahedral sheets) and/or of charge amount (from one interlayer to the other or within a given interlayer) most often lead to the coexistence of different hydration states within smectite crystals. In addition strong positional disorder of interlayer water and cations has most often to be considered when attempting to reveal the structure of such disordered layered systems.

Isothermal X-ray diffraction (XRD) patterns were collected vs. time and the experimental structures calculated were compared to the standard models.

The following speaker was **Helen Maynard** from School of Physics, Centre for Science at Extreme Conditions, SUPA, University of Edinburgh and she presented:
New 'minerals' of the outer solar system - The high-pressure Crystallography of Methane.

Very enthusiastically, Helen explained that here on Earth silicates dominate our mineralogy, but for both the Uranian and Neptunian systems a different, perhaps simpler, chemistry exists between H_2O, NH_3 and CH_4. Within the interior 'hot ice' layer of these bodies it is thought that interactions between these molecules account for the anomalous magnetic fields generated.

Methane's dissociation reactions could also be key to understanding what drives the planet's energy supplies. However studies of at these extremes of pressure (up to 25Gpa - 1Gpa at the bottom of Earth's oceans) and temperature have thrown up inconstancies and the way forward is perhaps to understand how the constitute materials behave at room temperature.

In the suite of outer solar system 'minerals', H_2O, NH_3 to CH_4, methane is the sole organic member and the only one with no hydrogen bonds. Van der Waals and stearic repulsive interactions instead determine its solid structure and high-pressure studies can probe the interplay between these forces. The methane tetrahedron exhibits orientational disorder in many phases and the high-pressure behaviour of methane has been discussed in terms of a 'bad rare gas' model, leading to an assumption that at higher pressures it will adopt structures related the hexagonal closed-packed structure.

Studies have shown that this simplistic view of the molecule was incorrect and that the phase diagram is very complex, with eight distinct solid structures.

Using a diamond anvil cell to replicate the extreme high pressures required it was discovered that phase B of solid methane has 28,000 molecules per unit cell and so it is proposed that a new suite of minerals exist in the outer solar system.

The final and intoxicating speaker of the morning was **Jenny Hugget** of Petroclays with her fascinating presentation entitled the geology of wine - complete with samples - hurrah!

As a moderate but enthusiastic consumer of wine I never realised how important geology is to the growth of the vine (never gave it much thought really, until today). I shall pay much closer attention in future - anyway over to Jenny. In between each section and to illustrate each point of the presentation the audience was asked to participate in tasting wines from various geographical regions (for obvious scientific reasons, you understand).

The role of underlying rock in viticulture is at least four-fold. It influences the soil type that forms over it, it permits penetration of vine roots to varying degrees depending upon the nature of the rock, it controls the geomorphology (slope) and it assists or hinders drainage of rainwater. However, as in many wine making areas the link between geology and wine quality is tenuous, some examples of spurious linking of geology and wine that have occurred were also considered. One famous quote describes terrain as being: "Of great importance to the wine maker, of very little importance to the drinker".

So is it only a matter of climate? Or are geology and terroir important when growing grapes for wine? Terroir is defined as a delimited area with its own characteristic geology, climate and methods of viticulture. Experts have said differing things about this concept:

- Where there is a perceived marketing advantage in associating a wine with soil in a specific region the terroir concept is being exploited (Australian wine expert: Busby, 1825).
- The influence of the surrogates geology and landscape on wine rather than through the properties of the soil itself (French wine consultant Pomerol, 1989).
- A concept that originated in France. It is difficult to think of another country where it could have started, since it has features so characteristic of second-class French thinkers, a combination of the obvious (e.g. the quality of a plant depends where you grow it) and the mystical. (Hancock, 1999).
- Most scientists admit they cannot express quantitatively the relationship between terroir and the characteristics of wine produced from that terroir (Australian soil scientist: White, 2003).

The concept of terroir is implicit in the fanciful tendency to associate wine flavours with aspects of the soil or bedrock. Perhaps the most widely stated is the supposed "flinty" character of Chablis wine. To a geologist it is difficult to imagine how a material as insoluble in normal groundwater as flint could contribute to the flavour of any wine, let alone what the flavour of anything so hard and insoluble could be. Equally fanciful is the suggestion that flint, schist and slate-bearing soils impart a "gunflint" character to Riesling. Geology influences aspect and slope, which in turn will influence microclimate and drainage. At high latitudes sunny slopes have always been preferred for viticulture. In southern England where the angle of the sun is 62° at noon on midsummer's day a vineyard on a 30° slope will receive 8% more radiation than a vineyard on level ground. This may not sound much, but by October, during the final ripening, the difference is 30%.

Geological controls on soil chemistry affect wine quality. Vines require all the usual plant nutrients (mainly N, P, K, Mg, Fe), present in well-maintained soil on any rock. Too much of any of them is a bad thing for vines. However in the Morgon appellation within Beaujolais the particular intensity of the wine is attributed to a vein of Mn ore that runs through the district. Iron availability is important. In limestone the high pH of the pore fluid reduces the solubility of Fe, with

the result that the Fe released by weathering becomes less available to plants. This is less of a problem for white varieties (which need less Fe) than red, and in the best limestones for viticulture there are sources of Fe. In the Chalk of Champagne the source of Fe is pyrite and in the Tuffeau of the Loire it is glauconite. Nutrient deficiency is controlled by geology in Bourgueil, where most of the Cabernet Franc is grown on alluvial sands and conglomerates with a low Fe content that leads to chlorosis. The better vineyards are on middle Turonian Tuffeau Blanc.

Geological controls on soil and wine quality may be summarized as follows:

- "The parent rock type has little direct influence on wine quality" Wine Science, Jackson (1994).
- The proportion of pebbles influences 1) drainage (good drainage is important) 2) warmth (absorbing heat by day, releasing it by night).
- The proportion of clay influences fertility and water retention (some is good).

However, the importance of geology to wine quality has sometimes been exaggerated.

- Chardonnay generally does best on alkaline soils (marls, clay with limestone).
- In 1923 Premier Cru Chablis was recognised as being grown on Kimmeridgian limestone, while Petit Chablis could be grown on any other soil within the appellation.
- This was strongly opposed by some growers who said that orientation and altitude are as important and that some quality Chablis had always been grown on Portlandian limestone ….
- 1976 the reference to Kimmeridgian limestone was dropped from the definition of quality Chablis, while aspect and microclimate were added.

The importance of solid rock porosity and permeability is considerable.

- Vines derive most of their nourishment from a depth extending down to 0.6 m, but will, most of the time rely on water from down as far as 2 m for transpiration.
- Only during periods of drought will they draw significant water from >2 m. At these times high porosity and low permeability will be an advantage.

But is viticulture more important than soil or geology?

- Riesling is grown on schist in Alsace, but nearby in Germany it is grown on slate.
- Alsatian Riesling is typically both more full-bodied and drier than German Riesling.
- Is this due to viticultural differences or subtle differences in soil / climate?
- In Alsace the wine is generally kept longer in barrel than it is in Germany.
- And Alsatian soils are typically more calcareous and clay-rich than the German slate soils.

All I can say is that I'm off to purchase a few cases of samples. Isn't geology fascinating?

Richard C.E. Morris,
Morris Analytical X-ray

Afternoon Session

Chaired by the programme organiser - **Martin Gill**, Natural History Museum

After lunch **Stephen Hillier** from the Macaulay Institute gave a presentation on the second survey of the National Soils Inventory of Scotland (NSIS). This included an overview of the importance of soil to the ecosystem and how it is affected by its mineralogy. Scottish soils differ from the rest of the UK in that they have a higher organic content, lower pH and are lower in nutrients. The minerals present are predominately quartz, feldspar, carbonates, iron oxides and clay minerals. These influence the soil properties owing to the nutrients they contain, their binding abilities, trace elements present, etc. The NSIS-2 is focusing on the variability of these properties on a local scale. Testing being carried out is bulk density, porosity, biodiversity, NIR, FTIR and XRD.

The soil samples are initially McCrone milled and then spray dried prior to powder diffraction analysis. Previous work has shown that side loading the samples in to the XRD slide results in preferred orientation some of which can be attributed to the operator. The spray drying on the other hand results in spherical "droplets" that pack randomly and are not influenced by the operator. This is seen by the consistency of the pattern when the same sample is prepared and analysed by different technicians. Full pattern matching is being carried out but this requires availability of the RIR data. The background is not modelled and an internal standard is not being used so as not to introduce dilution effects. The Macaulay Institute holds the National Soils Archive for Scotland with >40,000 air dried samples collected over 60 years.

Andrew Hardy from the University of Exeter followed on with a talk about cosmeceuticals. He explained that these are compounds that combine both pharmaceutical properties with a cosmetic role. Their first recorded use is believed to have been by the ancient Egypticians. A bivalve shell dated from 1300BC has been found containing traces of a green compound believed to be copper based. It is believed that cosmeceuticals are crushed minerals but there is so little sample left of these ancient materials that isotopic analysis is planned to determine if they are in fact naturally occurring or man-made. Modern day women in the Middle East still continue to use Kohls as cosmeceuticals which they do not realise can contain heavy metals such as As, Pb and Hg. So beware if you are given any of these as a gift!

A detailed history of chasing the blue mixed valence of iron sulphate was presented by **David Beveridge**. Its existence was first reported in the literature in 1843 by Barreswill. It was then again mentioned in 1863 by Lefort. However, David warned us, it is important to take 19th century reports with a "pinch of salt". In 1979 it was again reported in the literature by Steger. David has attempted to recreate this mixed valence state of the iron sulphate and was successful. However, it is only very transitory and lasted approximately 10 minutes before it decomposed in to a rhomboclase structure. Everything then stopped for tea!

After a short break **Alison Pawley** discussed synchrotron studies of the structure of hydrous phyllosilicates (HPs) at high pressure and temperature. HPs are important in the Earth's mantle. They occur at subduction zones and take part in recycling water back in to the mantle. This water then affects the flow rheology of the mantle. HPs are soft and highly anisotropic. An example is talc, a magnesium silicate hydrate, which exhibits weak stable sliding behaviour. By increasing the pressure talc transforms in to a 10 angstrom phase that is likely to be involved in the transport of water in to the mantle to depths of up to 200km.

High pressure IR on the synchrotron ring using a beam diameter of approximately 10um showed that the frequency of OH stretching in talc increases as pressure is increased. This suggested there is no H bonding present in the structure. For pyrophyllite there was a decrease in frequency with increasing pressure suggesting the presence of hydrogen bonding. For both talc and pyrophyllite increasing the temperature led to a decrease in the frequency of OH stretching. A deuterated sample of the 10 angstrom phase has been made in the lab. Studies on this material have not resolved the structure and it has led to a realisation that the structure is more complicated than at first believed. The data is still being looked at.

There is an international study taking place on crystalline silica. The reasons for this and the use of XRD in this study was presented by **Peter Stacey** in a presentation entitled accuracy in analysis - how much crystalline quartz do you think you have?

Crystalline silica causes silicosis therefore there is legislation related to its measurement. Samples of dust are taken from the air and collected on mini filters. These are then placed directly in an Xpert Pro MRD. The CRM used in this work, NIST 1878, ran out and was replaced in 1999 with NIST 1878a. Comparisons between the 2 CRMs indicated the crystalline phase in 1878a was 4% lower than certified. NIST then withdrew 1878a and re-issued it at 97% crystalline silica. This lead to people being very confused about the CRM and its value. Therefore, it was decided that this was an ideal opportunity to organise a comparative study. There are a number of participating labs from France, Canada, Spain, the UK and the USA. The measurement procedure was developed by the French. Al or Ag peak is used for depth and absorption corrections. 7 "standards" have been tested from a number of countries. A number of different issues were raised and it was noted that as the particulate size increased the level of agreement between the labs decreased. Further analytical techniques were also considered including specific surface area by nitrogen BET and IR by KBr disc. From this work it emerged that there was a good correlation between the XRD results and those of SSA but not with IR. Further developments planned include using Rietveld refinement on occupational hygiene samples. The final talk of the day was given by **Melanie Sapsford** on analysis of non-marine evaporitic deposits of the Wadi Natrun in Egypt. Examination of the geological and archaeological samples helps to determine the role that sodium salts have played in ancient Egypt and how these relate to the changing nature of the Wadi lakes. The Wadi Natrun is a valley 30 miles long and 50 km from Cairo. It contains a series of ephemeral evaporitic lakes of a non-marine origin. The number of lakes has changed with time and there are currently 12 lakes at present. The focus of Melanie's work has been samples from Lake Fazda. This is believed to have been around since Roman times based on the archaeological remains of glass manufacture found from that period. Chemical analysis of samples has included XRD, ICP and ion chromatography. The samples appear to contain high levels of burkeite, halite, some sulphate groups and some trona but none contain natron. Natron salts are known to have been used in the mummification process and it was hoped to find out which lake these salts have come from - obviously not Fazda.

Alison Burke,
Huntsman Pigments

Meeting Report - May 2008, BGS, Keyworth

Morning Session

Dave Taylor, BCA Industrial Group
Welcome

DAVE provided the welcome for the day from the Industrial Group of the BCA for this second joint XRF meeting with the RSC Atomic Spectroscopy Group. He thanked all the sponsors for their kind contributions, the BGS for use of the excellent facilities and Mark Ingham and his collegues for their help with set up and local arrangements.

Chair, Morning Session
Andy Scothern, RSC

The first talk of the morning was by **Nick Marsh** (Leicester University), on the preparation and presentation of mineral samples for XRF analysis. The usual requirement is a fine powder (but if you have to crush the sample, is the technique nondestructive?) and there are several stages to this. Drying should be undertaken with care: volatile elements such as mercury can be lost, as can essential water from hydrated minerals. Crushing and milling is very dependent on mineral properties such as hardness, brittleness, cleavage and density, all but the last of which can be anisotropic. The final stage is to convert the powder into a form suitable for analysis. This may be a loose powder, a pressed pellet, a fused bead, even a dust on a filter. These all have their particular characteristics. It is always worth thinking about critical thickness (or thinness, in the case of dust on a filter). Mineralogical effects - for example, when one grain type is very different in its absorption of X-rays to others - can be very significant in pressed pellet samples. A fused bead will get round this difficulty. All in all, it was an interesting and thought-provoking talk.

The second speaker of the session was **John Mansell** (Omya UK Ltd), who spoke about the analysis of limestones by

EDXRF. This study included elements from major to trace levels, and included samples from a large number of quarries, so that the provenance of unknown samples could be established. The XRF method was very much quicker than previous methods (ICP or DCP-based) which required preliminary dissolution of the sample. Not quite all elements could be analysed - in particular, the sensitivity needed for Cd is really beyond the instrument, and Ba is difficult because of line overlaps.

Margaret West (West Analytical Solutions Ltd) spoke next, describing the background to a new BSI standard for glass-making sands. She described the parameters of the specification for a glass-making sand, and how the new version of BS2975 had been put together by the Society for Glass Technology. This is in two parts: sampling and testing; and chemical analysis. A wide range of physical properties is specified, which need to be tested. There is a detailed description of a sampling protocol, because sampling (surprise, surprise!) is one of the trickiest problems in the analysis. Grinding media are discussed, and the preparation of samples for presentation to the XRF. For some purposes, wet chemical analysis (inevitably involving HF digestions) is unavoidable, but mostly XRF is a lot easier. Standards and reference materials are, of course, needed, and a range has been made available by the Society for Glass Technology in conjunction with BAS Ltd.

The final talk in the morning session was by **John Boyle** (University of Liverpool), who described the analysis of lake sediment cores by EDXRF. These samples can be very variable, and a general approach is needed. Many elements must be determined, often in a small sample (less than 1 g - the cores may have to be divided among many researchers). Particle size is rarely a problem because it is normally very small anyway. Matrix effects are definitely a problem, but one for which corrections can usually be made. Background measurements can be awkward, as the peaks are close together, but there are ways around this difficulty. Line overlaps are another problem, but again one which can be overcome with care.

This closed the morning session, after which we enjoyed an excellent buffet lunch while we looked at the manufacturers' exhibits.

David Beveridge
Harman Technology limited

Afternoon Sessions

Chair, Afternoon Session 1
Ros Schwarz (Oxford Instruments Analytical)

Neil Eatherington, (British Geological Survey, Nottingham)
When Sample preparation is not an option

Neil acknowledged that for the majority of applications XRFS requires sample preparation to get the best from the technique, but what happens when preparation is not an option? In-situ samples together with historic or valuable samples require sensitive handling and cannot be destroyed. The problems with in-situ analysis stem from the moisture content of samples together with particle size and mineralogical effects.

Drying of samples was discussed with attention drawn to the problems of using a microwave oven (elevated temperatures of up to 200°C) and the loss of volatile analytes from environmental and soil samples. Conventional drying at 40°C was ruled out because of the time taken to achieve a dry sample (<5% moisture) measured in days rather than minutes.

Several case studies were presented featuring marine sediment cores, panned stream concentrates and in-situ analysis. Data was compared between handheld XRFS costing ~ £35k and specialist equipment such as an ITRAX core scanner costing ~£200k with the conclusion that the handheld was useful as a screening tool and has some success at quantification. Overall the correlations between fully prepared samples and in-situ samples were generally good.

Prof **Graham Martin**, (Victoria and Albert Museum, London)
Heritage and XRFS -unlikely companions?

This was a different presentation where Graham avoided posting the usual spectra, graphs and data but instead concentrated on the application of ED-XRF within the heritage sector. Graham began with an overview of the Victoria and Albert Museum and its associated institutes and explained that a large challenge was communicating the applications to his colleagues with arts based backgrounds.

The sheer scale and diversity of the exhibits and potential sampling sites were discussed together with the fact that invasive and chemical techniques were offered as a last resort but usually not at all. The analytical equipment used was presented from the Tracor DuBois pigment analyser from 1983 to the present equipment of the Bruker ArtTax, a portable (in 3 cases), open-sourced analyser mounted on a highly flexible mechanical arm that requires high operator skill levels.

The types of sampling were explored detailing pigments from an unused artist palette from 1880 that effectively provided standards for pigments through to illuminated manuscripts, ceramics and more recently the exploration of polymers in more contempory exhibits.

Health and safety aspects were also considered by demonstrating the elevated arsenic contamination from the process of taxidermy to the contamination of mercury in felt hats. Indeed it was suggested that if one hat was thrown into a reservoir it had the potential to poison one million litres of water at present WHO levels of mercury contamination.

Owen Butler, (HSL, Buxton) &
Elke Adriaenssens,(VMM, Antwerp, Belgium)
Analysis of heavy metals in suspended airborne particles collected on filter papers by XRF techniques

Owen began by summarising the work of the HSL at the Buxton site which has favourable scientific and engineering facilities. As an agency of the Health and Safety Executive, part of the governments Department of Work and Pensions the analytical services unit undertakes wide ranging tests and is involved with regulatory and investigative measurements, method development, proficiency testing and some commercial work. The workplace risk to the inhalation of aerosol and particulates

down to sub-micron size and subsequent sampling problems were discussed. In particular was noted the problem caused by ergonomic work practices of sampling to mimic inhalation. Technical innovations such as a unique design of filter cups to accept varying diameter papers from 13-48 mm, together with the problems of moving from WD-XRFS to ED-XRFS analysis and the subsequent bias introduced with the smaller beam size were presented. A new model for sampling particles, the Minisampler employing a 13 mm filter located adjacent to the mouth was demonstrated and validated using a model and automated welding rig. Future interest lies in the analysis of nano-particles with spot sizes down to that of a pinhead.

Elke introduced her work with VMM, the Belgium equivalent of the EA, reporting on air and water quality and the general state of the environment. Air quality was measured in a network of automatic and semiautomatic stations for a variety of parameters including PM10 and PM2.5 particulate fractions. The automatic stations provide continuous real-time data available on the website **www.vmm.be**.

Two populations of heavy metal data for 2007 were described, urban/rural and industrial. The rural/urban mean and maximum values were low whilst the industrial values were high requiring a wide ranging application to process more than 7500 samples annually. Technical problems were overcome by the attention to measuring conditions and the lack of commercially available standards overcome by a method of in-house standard preparation. The calibration was described as secondary and validated by ICP-MS or AAS techniques.

Elke went on to describe the equipment used for heavy metal determinations commenting on the inflexibility of using two X-ray tubes for the WD-XRFS technique. A change of equipment to a PANalytical Epsilon 5 ED-(P)-XRFS with a Gd tube allowed for the analysis of As, Cd, Cr, Cu, Mn, Ni, Pb, Sb and Zn at 30 minutes per sample. The advantage of the ED analysis was a lower detection level, meeting BS EN 14902 requirements except for Cd. Validation between ED-(P)-XRFS and ICP-MS showed good correlation for As, Cd, Ni and Pb at higher concentrations. For Ni and Pb the correlation is still acceptable at lower concentrations, for Cd and As the correlation is not good in the low concentration range.

Simon Carter
BGS

Afternoon Session 2

Chair, **Dave Taylor**

Richard Meeres, (Bureau of Analysed Samples Ltd).
A History of the Development of Certified Reference Materials

This presentation discussed the history of the company, from its inception in 1912 as Ridsdale & Co to the Bureau of Analysed Samples (BAS) in 1935. In 1950, the Honorary Advisory Committee (HAC) was created to provide an unbiased wealth of experience, make recommendations, evaluate results and approve Certified Reference Materials (CRMs). The HAC has met twice a year since its creation, and BAS also organises Triennial Meetings of UK Co-operating Analysts to gather comments and ideas for the future. The types of CRMs produced consist of iron & steels, non-ferrous metals & alloys, iron & non-ferrous ores, ferro-alloys, slags & refractories and ceramic materials & minerals.

Alison Burke, (Huntsman Pigments).
Routine Analysis of TiO_2 by WD XRF

Huntsman Pigments has seven factories around the world which together produce approximately half a million tonnes of titanium dioxide each year. Around 60% is used in coatings (paint, etc) with the remainder being used in products such as plastics, inks, toothpaste and cosmetics. Sample types requiring analysis are the final packed pigment, calciner discharge, in-process samples and ores. Several techniques are used for elemental determination; however XRF provides a simple, rapid and easy to use method.

The Harmonisation Project of Analysis was initiated in 1998 in order to ensure inter-site reproducibility precision targets for each XRF element. Initially blind testing was carried out every six months to establish levels of agreement using z-score statistics. Now established harmonisation is maintained by the project sharing monthly validation for all seven laboratories, carrying out annual blind tests, hosting telecons every two months, providing operator training and international compliance audits.

Andy Scothern, (Saint-Gobain Gypsum)
XRF Analysis in the Gypsum Industry

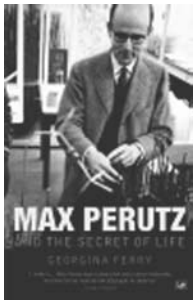
Gypsum markets consist largely of construction (37%), renovation (29%), household (19%) and industry (15%). Saint-Gobain Gypsum (SGG) currently employs in excess of 200,000 people, with two out of three working outside France. Research and Development centres are based mainly in France, but also in Germany, the United States and South America. The Technical Centre is based in East Leake and concentrates on process research, product research and product development.

Gypsum consists of several phases - gypsum ($CaSO_4.2H_2O$), hemi-hydrate ($CaSO_4.1/2\ H_2O$), and both soluble and insoluble forms of anhydrite ($CaSO_4$). By applying temperature to this material, it phases through gypsum to hemi-hydrate to soluble anhydrite finally to insoluble anhydrite and back again by the addition of water. The reduction of water, and therefore the reduction in heat required for the final products are crucial and SGG is trying to find ways to implement this.

Samples are dried overnight at 40°C in order to remove any free water and to convert any soluble anhydrite to hemi-hydrate. Proximate analysis was used in the past to record the weight loss on heating; however, thermogravimetric (TGA) monitoring is now used. Problems with this technique include the inability to measure insoluble anhydrite, as there is no water left to lose, and difficulties when clays or additives are present. WD-XRF is now used for total SO_3 content in anhydrite as it is more accurate than TGA, assuming all SO_3 is present as sulphate. Other work in progress included analysis of cement boards, semi-quantitative analysis on powders and liquids and trace metals in dusts.

Leian Grimsley, BGS

Books



Max Perutz and the Secret of Life

Georgina Ferry

Chatto and Windus 2007
Price £25 (hardback)
ISBN 9780701176952, xii + 352 pages , Paperback by Pimlico, price £12.99.

IN 1936, Max Perutz (1914-2002), a chemist from Vienna with little knowledge of either crystallography or biology, plunged, partly by chance (he had hoped to work with the Nobel prizewinning chemist Sir Gowland Hopkins) into crystal-structure analysis at a world centre of physics. In the Cavendish Laboratory in Cambridge, he became a self-funded (from his affluent Austrian parents) student of the charismatic J. Desmond Bernal, who was investigating the structure of biological molecules. As a junior researcher, Perutz had first to coax a continuously evacuated gas X-ray tube to take photographs of rhodonite mineral crystals from slag. Referees to both his mineralogical publications in 1937 and 1939 criticized his limited attention to detail at this stage.

Following the death in 1937 of the head of the Cavendish Laboratory, Lord Rutherford (whom Perutz had not yet met, despite working there for a year), senior physics chairs in England were reshuffled, with Bernal moving to Birkbeck. While remaining an admirer of Bernal, Perutz opted to continue his PhD at Cambridge. W.L.Bragg's arrival as head of the Cavendish in 1938 confirmed Perutz's immersion in crystallography. He had already determined to study the structure of haemoglobin, which was to become his life's work, inspired by a conversation with the biochemist Felix Haurowitz in Prague.

Although critical of Perutz's early X-ray reports, Bragg became a staunch supporter of protein studies, especially in the setting up of the MRC Research Unit on the Molecular Structure of Biological Systems in 1948 and in negotiations leading to the MRC Laboratory of Molecular Biology (LMB), eventually opened in a new building in 1962. This was a few months before Perutz and John Kendrew were awarded the Chemistry Nobel Prize for studies of globular proteins. The LMB which Perutz created and chaired (rather than directed), 1962-1979, gained an incredible twelve Nobel prizes.

In 2002, Crystallography News published (No 81, 24-26) an affectionate and knowledgeable obituary of Perutz (from The Independent) by David M. Blow; Blow subsequently wrote the Royal Society Biographical Memoir, 50 227-256 (2004). Now Perutz is the subject of a full authorised biography. Georgina Ferry is not a scientist but her biography of Dorothy Hodgkin (1998), utilizing a vast collection of archival material, was well received and introduced her to describing crystallographic analysis of biological systems. Shortly before Perutz died in hospital from a rare skin cancer, he asked Ferry to be his biographer. His fears as to the paucity of records proved unfounded and Ferry managed to trace much of his correspondence (retained by recipients, including his devoted wife Gisela) as well as calling on the recollections of an array of relatives, friends, and molecular biologists and other scientists. She tells the story of Perutz's skill, insight and persistence over decades investigating the structure of haemoglobin, his part in founding molecular biology, and the emergence and flowering of the LMB. Her book embraces the happy Viennese childhood and early life, and the struggles for financial support and scientific recognition through to the travel, talks, writing and continued research of later years. Thus one is made aware of Perutz's devotion to human rights, his good manners, and his intellectual honesty (which did not baulk at frankness).

Perutz enjoyed the written word and took semi-popular scientific writing seriously, beginning with an article on proteins, the machines of life, in 1944. His first academic book Proteins and nucleic acids (Elsevier, 1962) was rather overshadowed by James D. Watson's The molecular biology of the gene in 1965. Science is not a quiet life (World Scientific, 1997) was a kind of scientific biography, telling of the disappointments, controversies and successes in the haemoglobin saga through introductory commentaries on the reprinted published papers together with a chapter for the uninitiated, optimistically entitled 'Crystallography without tears'. His more general books on science, scientists and science policy contained chapters mostly based on essay book reviews. Within I wish I'd made you angry earlier (Oxford UP, 1998) the article 'Enemy alien' records how Perutz, having been attracted to Cambridge for research in 1936, became in 1939 a refugee, and then in 1940 endured a harrowing time of internment in the Isle of Man and Canada. JM Thomas, in the Crystallography News report (No 83, 27-30, 2002) comments on the almost poetic way in which Perutz begins this chapter.

In the same year, 1962, that Perutz received his Nobel Prize for Chemistry, Maurice Wilkins at King's College and Francis Crick (a colleague of Perutz's from 1949 in the early days of the MRC Unit within the Cavendish) and James Watson at Cambridge were awarded the prize for Physiology or Medicine for the DNA structure. However

it was the publication of Watson's very personal (Ferry calls it explosive) account, The double helix (Athenaeum, 1968) with its portrayal of scientific competition, which embarrassed Perutz. This was over his disclosure to Crick and Watson about fine-focus and other X-ray photographs of the B-form of DNA, taken by Rosalind Franklin, whose group tended to work in isolation. Franklin was on good terms with Crick and his wife for the rest of her life - she died in 1958 - and may not have realised fully how vital her data were to Crick and Watson.

Franklin's friend Anne Sayre (wife of crystallographer David Sayre) was one of the first, in Rosalind Franklin and DNA (Norton, 1975), to amend some of Watson's inferences and set out the interactions between the model builders at Cambridge and the more experimental workers at King's between November 1951 and February 1953. The many other assessments include Brenda Maddox's full biography, Rosalind Franklin: the dark lady (HarperCollins, 2003) and Watson Fuller's article in Nature, 424, 876 (2003). While Franklin's contribution to the DNA story is now recognised (though not by a share in a Nobel), Perutz's long letter to Science of 1969 (164, 1537-1539) shows that he was conscious that it might have been improper to pass on to Crick in February 1953 unpublished data from a King's MRC report. (In his letter, Watson admitted that readers of his book might 'badly misconstrue' Perutz's actions). Others including Sir John Randall at King's, judged Perutz more harshly. Ferry handles this sensitive episode tactfully, including a quote from Wilkins that 'none of us behaved quite impeccably'.

Perutz engaged in many 'haemoglobin battles' about the stereochemistry of the co-operative mechanism and Ferry recounts disputes in the 1980s with Robert Shulman and Chien Ho. For a warm and generous person he had some surprisingly acrimonious conflicts and suffered the insecurities and envies that afflict many effective scientists. He was also a hypochondriac, perpetually anxious about his diet, having contacted coeliac disease, and perhaps cultivated eccentricity.

Aside from the structure of proteins, Perutz was happily married and for long indulged in his love of mountaineering and, until prevented by back trouble, skiing. Indeed in the late 1930s and the 1940s he was able to combine these interests with crystallography in both wartime and peacetime investigations such that he gained a reputation as a glaciologist.

Ferry's painstaking but enjoyably readable account of Perutz and haemoglobin is accompanied by photographs of the chief characters, a glossary, and an unfussy paged collection of over 40 pages of notes. Among the qualities admired and practised by Perutz were good writing, respect for other people, persistence in searching for the truth, and ability to communicate science interestingly to non-specialists. On these criteria, I think Perutz would be content with Ferry's version of the secrets of his life.

Derry W Jones

Abstract Submission for AGM Loughborough 2009

THERE are slots reserved in each symposium for contributed talks. Please submit abstracts for consideration using the word template (available from the conference website) by Friday 24 October. Applicants will be informed by 21 November whether their talk has been accepted.

As always, the poster session will be one of the main fora for scientific discussion. We'll also be offering poster contributors the opportunity to display graphical abstracts on screens throughout the exhibition. More details on this to follow. The deadline for poster abstracts will be in early February.

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.

1-5 September 2008
7th International Workshop on Polarised Neutrons in Condensed Matter Investigations, Tokai, Japan.
http://61.199.198.53/pncmi_html/

1-9 September 2008
EMBO Practical Course on Protein Expression, Purification and Crystallisation: EPC6, Hamburg, Germany.
http://www.embl-hamburg.de/workshops/2008/PEPC6/

1-12 September 2008
12th JCNS Laboratory Course - Neutron Scattering, Jülich/Garching, Germany.
http://www.fz-juelich.de/iff/wns_lab_now

3–5 September 2008
5th CCP4 Protein Structure Workshop, University of Cumbria in Carlisle, UK.
http://www.chem.gla.ac.uk/research/groups/protein/gala/

7-12 September 2008
BCA/CCP4 Summer School in Macromolecular Crystallography, Oxford.
http://www.biop.ox.ac.uk/www/bcasummer2008.html

8-9 September 2008
6th International NCCR Symposium on New Trends in Structural Biology, Zurich, Switzerland.
http://www.structuralbiology.uzh.ch/symposium2008.asp.

8-10 September 2008
12th International Conference on Experimental Mineralogy, Petrology and Geochemistry EMPG-XII, University of Innsbruck, Austria.
http://www.uibk.ac.at/mineralogie/empg2008/index.html

8-10 September 2008
9th International Congress for Applied Mineralogy, Brisbane, Australia.
http://www.icam2008.com/

9-14 September 2008
WATOC-08 World Association of Theoretical and Computation Chemists. Sydney, Australia.
http://www.ch.ic.ac.uk/watoc

14-20 September 2008
EMBO Practical Course - X-ray crystal structure determination of macromolecules, Saint Aubin, France. http://cwp.embo.org/pc08-26

15-17 September 2008
XTOP2008 - 9th Biennial Conference on High Resolution X-Ray Diffraction and Imaging. Linz, Austria.
http://www.hlphys.jku.at/xtop2008/xtop2008.html

15-17 September 2008
German Neutron Scattering Conference 2008Technische Universität München, Garching, Germany.
http://www.frm2.tum.de

15-19 September 2008
2008 E-MRS Fall Meeting, Warsaw, Poland.
http://www.e-mrs.org/meetings/fall2008/

18-22 September 2008
11-th European Powder Diffraction Conference - Warsaw, Poland.
http://www.epdic-11.eu

20-24 September 2008
ICSG 2008: International Conference on Structural Genomics, Oxford.
http:// www.spine2.eu/ISGO

22 September - 3 October 2008
IX School of Neutron Scattering Francesco Paolo Ricci, Santa Margherita di Pula (CA)- Sardinia, Italy.
http://www.fis.uniroma3.it/sns_fpr/index.htm

23-24 September 2008
Translating Co-crystals Properties, Screening and Design into Commercial Success, Amsterdam, Netherlands.
http://www.iqpc.com/nl/cocrystals/2020

24-26 September 2008
Biomolecular Dynamics and Protein-Water Interactions, Munich, Germany.
http://biodynamics.physik.tu-muenchen.de/main.htm

29 September - 3 October 2008
Ninth International School on the Crystallography of Biological Macromolecules, Como Italy.
http://www.crystallographyschool.org.

9-10 October 2008
7th ANKA Users Meeting, Karlsruhe, Germany.
http://ankaweb.fzk.de/conferences/users_meeting_2008/first_page.html

13-19 October 2008
NSCMI-2008 XX International Workshop on Neutron Scattering in Condensed Matter Investigations Gatchina, Russia.
http://rno.pnpi.spb.ru/infoconf.php

15-17 October 2008
Modern Trends in Neutron Scattering Instrumentation. München, Germany.
http://www.jcns.info/Workshop/

19-23 October 2008
Biological Physics at Large Facilities from Molecule to Cell Grenoble, France.
http://www.ill.eu/news-events/workshops-events/biological-physics-at-large-facilities-from-molecule-to-cell/home/

19-26 October 2008
EMBO Practical Course on Solution Scattering, EMBL, Hamburg Outstation, Germany.
http://www.embl-hamburg.de/workshops/2008/embo/

20-24 October 2008
7th NCCR Practical Course and EMBN Summer School - Membrane Protein Crystallisation, Basel, Switzerland.
http://www.structuralbiology.uzh.ch/membranecourse2008.asp

23-24 October 2008
Workshop on the Scientific Opportunities in France for a 4th Generation Intermediate Energy Light Source, Institut Henri Poincaré, Paris, France.
http://www.synchrotron-soleil.fr/ArcEnCiel

3-5 November 2008
NOBUGS 2008 Collaboration for Developers of Computer Techniques in Scientific Instrumentation, Sydney, Australia. http://www.nbi.ansto.gov.au/

5 November 2008
Industrial Group of the BCA: Pharmaceutical Special Interest Group Autumn Meeting, AstraZeneca, Loughborough.
http://bca.cryst.bbk.ac.uk/bca/ig/meet08AM.htm

9-14 November 2008
EMBO World Lecture Course - Recent Developments in Macromolecular Crystallography Pune, India.
http://cwp.embo.org/wpc08-02/index.html

12 November 2008
Chemical Crystallography Group Autumn Meeting 2008: New Methods in Chemical Crystallography, Newcastle University.
http://img.cryst.bbk.ac.uk/BCA/ccg/ccg.html

16-20 November 2008
SARX2008: Latin American Seminary of Analysis by X-Ray Techniques, Cabo Frio, RJ, Brazil.
http://www.lin.ufrj.br/sarx2008/

17-20 November 2008
14th International Conference on Thin Films. Ghent, Belgium.
http://www.ICTF14.UGent.be

1-4 December 2008
GISAXS (Grazing Incidence Small Angle X-ray Scattering) Symposium, Boston MA USA.
http://www.mrs.org/s_mrs/bin.asp?CID=13669&DID=208259&DOC=FILE.PDF

9-12 March 2009
17th Annual Meeting of the German Crystallographic Society, Hannover, Germany.
http://www.conventus.de/dgk2009

21st-23rd April 2009
BCA Annual Spring Meeting: Dynamic Crystallography, University of Loughborough.
http://www.crystallography-meetings.org.uk/

4-14 June 2009
High Pressure Crystallography: from Novel Experimental Approaches to Applications in Cutting-Edge Technologies. Erice, Italy.
http://crystalerice.org/erice2009/2009.htm

22-24 June 2009
ICNX-2009, International Conference on Neutron and X-Ray Scattering Kuala Lumpur, Malaysia.
http://icsd.ill.fr/ICNX2009.pdf

22-26 June 2009
Goldschmidt 2009 'Challenges to our Volatile Planet', Davos, Switzerland.
http://www.goldschmidt2009.org/

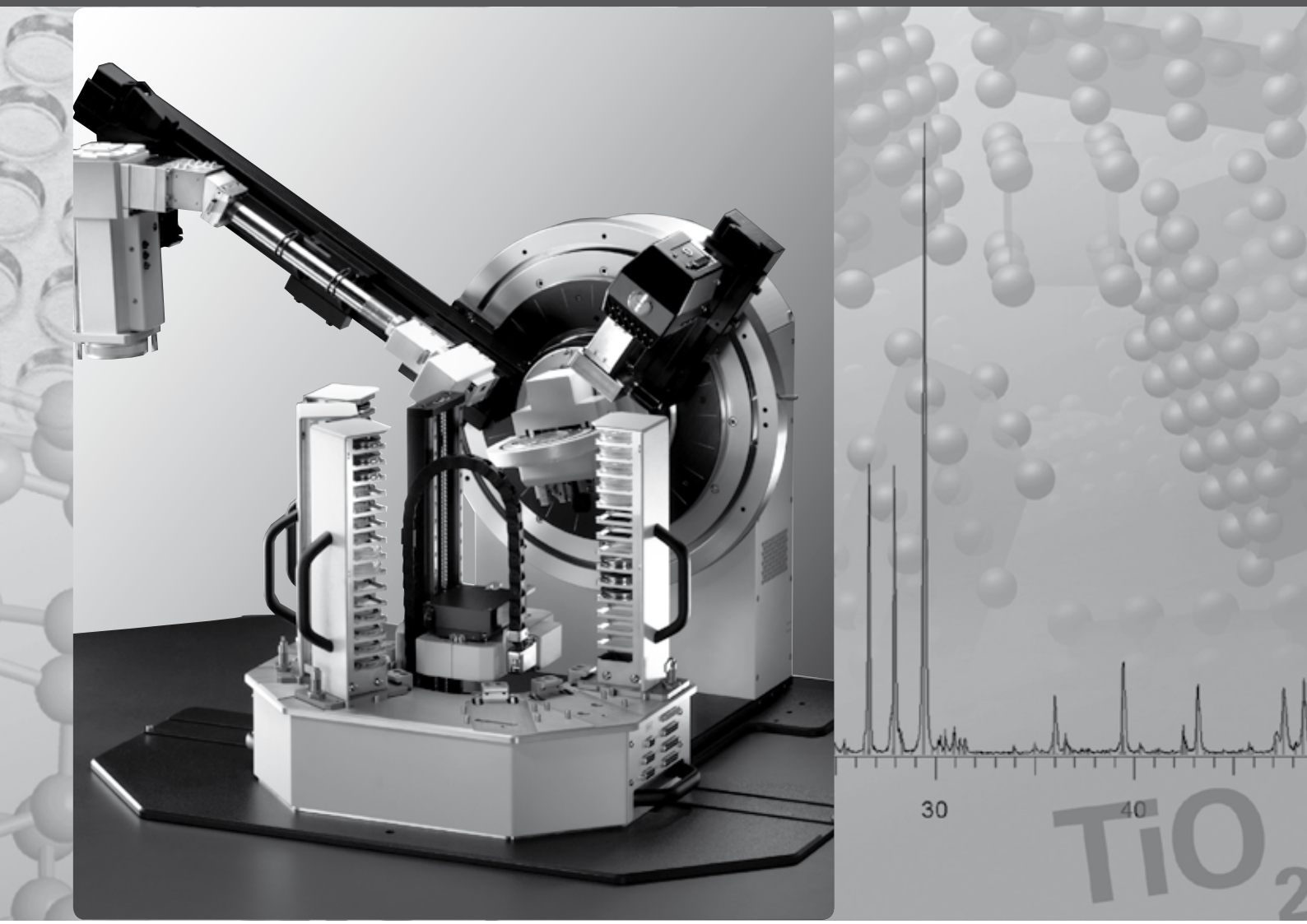
25-30 July 2009
Annual Meeting of the American Crystallographic Association 2009. Toronto, ON, Canada.
http://www.amercrystalassn.org/meetingspg_list/futuremeetings.html

2-7 August 2009
SAGAMORE: Charge Spin and Electron Density, Santa Fe NM USA.
http://www.sagamoreXVI.org

14-16 August 2009
Symmetry and Crystallography in Turkish Art and Culture: Satellite Conference of ECM-25, Istanbul, Turkey.
http://www.lcm3b.uhp-nancy.fr/mathcryst/istanbul2009.htm

16-21 August 2009
25th European Crystallographic Meeting, Istanbul, Turkey.
http://www.ecm25.org

Bruker AXS



D8 DIFFRACTION SOLUTIONS – Manage your Analytical Challenges

D8 DIFFRACTION SOLUTIONS stands for continual progress in all X-ray core technologies: X-ray sources, X-ray optics, sample handling, detectors and software – all on a common platform. Two innovations for higher data quality and sample throughput enhance the D8 capabilities:

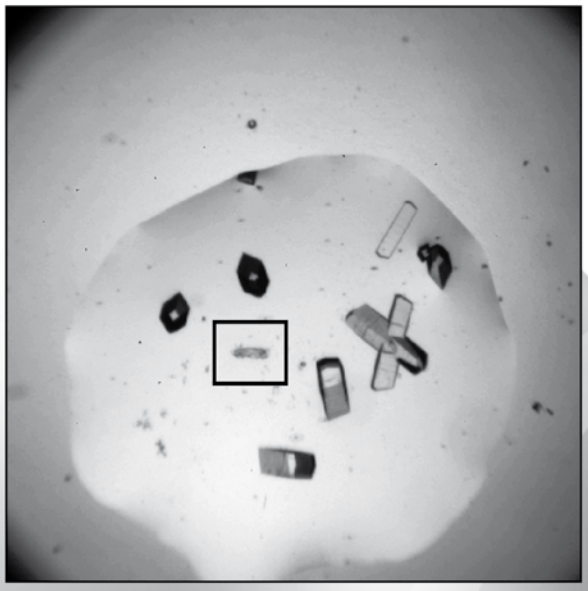
- Auto Changer for large sample batches
  - Effective instrument usage without user intervention
  - Reflection and transmission investigations without conversion
- LynxEye™ and VÅNTEC-1™ linear detectors
  - Large 3° 2Theta coverage for 150 times faster measurements
  - Huge 12° 2Theta coverage for 1 second Snap-Shot recording of kinematic processes

Contact us for more details and system demonstration! www.bruker-axs.com

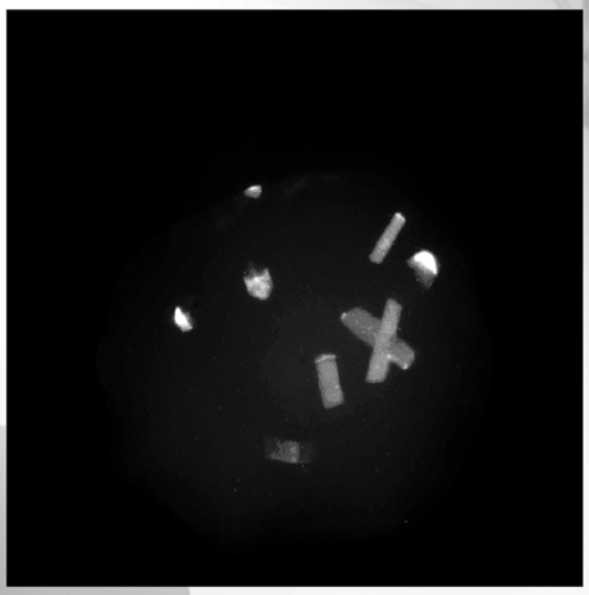
See what U'V been missing!

Leading With Innovation

Desktop Minstrel™ UV crystal imaging for protein crystallography



Visible light image



Ultraviolet light image

Screen your crystals quickly, accurately and automatically with both visible and UV light

www.Rigaku.com/automation

Rigaku

Rigaku Americas Corporation phone: (281) 362-2300 e-mail: info@Rigaku.com
Rigaku Europe phone: +[44] 1732 763367