BCA IG Newsletter

February 2003

EDITORIAL

Welcome to this latest issue of the IG newsletter. After a relatively quiet 2002, there are some exciting things happening in 2003. Firstly, some changes to the format of the Spring Meeting in York means that delegates can now register on a daily basis. This is good news for many industrial group members whose work commitments make it difficult to take a whole week off. There are lots of details in this newsletter; so please do have a look through the program and come along. This is a good opportunity to meet fellow diffractionists and also look at some of the latest developments in hardware/software in the commercial exhibition. Finally, please consider putting together something for a poster – you might win a prize! The meeting offers excellent training opportunities (less than £100 per day full board!) so recommend the meeting to your colleagues.

Also in this newsletter are details of the Autumn Forum. This promises to be a fantastic event with something for everyone and a chance to enjoy the capital for a couple of days; maybe longer.

Thanks to those who have kindly submitted articles for this newsletter. I am always looking for contributions.
Phil Holdway

Inside This Issue

Forthcoming Events 2003

BCA SPRING MEETING, YORK

15th – 17th April 2003

One-day registrations available.

Reduced fee if you register by 10th March.

Check Web Site for the latest information.
Copy the registration form on page 11.

Industrial Group AGM 16th April

BCA INDUSTRIAL GROUP AUTUMN FORUM

Provisional dates November 13th –14th
Birkbeck College, London

Preliminary details inside

Charity Registration Number: 284718

World Wide Web addresses:
BCA http://bca.cryst.bbk.ac.uk/BCA/index.htm
IG http://bca.cryst.bbk.ac.uk/BCA/IG/ig.htm

THANK YOU

Thanks to **RigakuMSC** for sponsoring the cost of distribution of this edition of the Industrial Group Newsletter.

INDUSTRIAL GROUP AUTUMN MEETING

SAMPLE PREPARATION

Hulme Hall
University of Manchester, 7th November 2002

Plus ca change - or an exile's return.

Despite having been a member of the economically-inactive-but-less-than-senior-citizen-age army for the best part of ten years, an Autumn Meeting in Manchester, my home town, was an unmissable opportunity! The Old Dining Room of Hulme Hall, where we were watched over by the portraits of distinguished clerical fellows, proved a most suitable venue for an informal but informative presentation of eight papers on Sample Preparation Techniques.

I had wondered how much would have changed since I was actively involved in xrd but I found that, although progress has led to further improvements in the hardware, many of the problems discussed were familiar and many of the usual suspects were present at the gathering! We heard of the techniques developed in response to sample preparation problems with materials gathered from tidal flats (Martin Gill, Museum of Natural History), from surfactants (Richard Morris, Huntsman Surface Sciences), from aero-engines (Colin Small, Rolls Royce) and from pharmaceutical materials (Chris Frampton, University of Southampton). In addition, the selection and preparation of samples for residual stress and texture measurements (Phil Holdway, QinetiQ) and the practical problems in contract analytical work (Ros Shwartz, London & Scandinavian Metallurgical) were reviewed. In a paper on lattice parameters and sample displacement, we were reminded of the need to consider possible sources of error arising from the nature of the sample (Mary Vickers, University of Cambridge).

A novel idea was the use of a torque wrench to ensure consistent compression of samples needing a binder – that seemed to be a good one!

Other papers reminded of the need for an understanding of what we are doing, why we do it the way we do and getting clear what we think we need to find out.

In fact, not a lot had changed: all the old things about consistent care, consistent technique, consistent alignment checks remain as true as ever.

During the Meeting, the latest in the series of Industrial Group Awards was given to Jo Jutson to mark her service to the BCA and her helpful involvement in the wider scientific community. Chris Frampton gave a short address, reviewing Jo's scientific career and contributions. (He omitted to note that this has included scientific presentations to her WI Branch: I wonder if this is unique among recipients of the Award?) Chris invited Jo to give her paper "From Spinel Analysis to Micro X-ray Diffraction - a Personal View of Applications and Advances" which she did with her characteristic modesty and clarity.

Note photograph removed to keep file size small!
Please view the online pictures at:

http://bca.cryst.bbk.ac.uk/bca/ig/reps02.htm#JJawrd

Jo Jutson receives her award from Chairman Professor Chris Frampton
(A spinel molecular model from Beevers Minature Models)

Summaries of all papers are in the IG section of the BCA website, through which the various speakers may be contacted. Congratulations are due to local organiser Judith Shackleton (Manchester Materials Science Centre) and thanks to the Group Committee for a stimulating, smoothly run Meeting.

Eric Kelly.
Penyffordd, Sir Fflint.

Editors note:
A UK supplier of Single Crystal Silicon for use as zero background substrates for PXRD samples was mentioned in discussion. Contact details are:

Crystal Scientific Limited
Lee Moor Business Park
Rennington
Alnwick
Northumberland
Tel: ++44 1665 572140
Fax: ++44 1665 572141
e-mail: sales@crystal-scientific.com

Technical Contact Dr Simon Cockerton

Quantitative Analysis

ROUND ROBIN – SAMPLES

The BCA Industrial Group is launching a quantitative analysis Round Robin.

Two samples are available:

1. Organic – a mixture of paracetamol and lactose.

2. Inorganic – a mixture of Rutile and Anatase.

The exercise will be discussed on the Thursday afternoon of the Spring meeting.

The choice of technique will be up to the individual participant.

Full details are not available as we go to press with this Newsletter. More details will be available on the IG web pages by the time you receive your copy.

Please volunteer to take part to test the accuracy of your quantitative analysis technique.

A prize of a bottle of champagne will be awarded to the participant whose result is closest to the theoretical composition for each sample. In the event of a tie the judges will consume the prize!

These exercises can provide useful insight into the best choice of technique for particular samples.
Please take part or at least come to the session at York and listen to the discussion.

The 2nd Pharmaceutical Powder X-ray Diffraction Symposium Concordville, Pennsylvania, USA 9th -12th December 2002

The symposium, which focuses on the use of XRPD for the analysis of pharmaceutical materials, comes three years after PPXRD-1 and the content reflected the dramatic rise in status of XRPD within pharmaceutical industry during that time. Structure determination for organics was the 'cutting edge' of PPXRD-1; at PPXRD-2, that baton was passed to high-throughput crystallisation and the screening and identification of polymorphic forms and salts.

The symposium opened with a day dedicated to the acquisition and analysis of XRPD data. Various representatives of academia, pharmaceutical industry, equipment manufacturers, analytical labs and database vendors covered the full spectrum of methods now available for examining pharmaceuticals as raw materials and as finished products. Whilst the emphasis was very much on 'in-house' laboratory XRPD (both 'standard' and 'high-throughput'), synchrotron radiation was also represented. The talks ended with a session on powder indexing and an evening poster session. It was clear by the end of the day that a great many ingenious hardware and software solutions have been developed to specifically target the problems currently occupying pre-formulation and formulation laboratories in industry.

The second day opened with a session on polymorph characterisation. Again, the speakers represented all interested parties and presented results from challenging problems, including determining the dynamics of dehydration in indapamide hemihydrate, 'clustering' of low-quality XRPD patterns for phase identification and characterisation of four polymorphs of fananserine. High-throughput crystallisation techniques that allowed up to ~300 crystallisations per day for a single lead compound were also clearly explained. The afternoon session focused-down on structure determination from powder data, and a wide range of examples including zopiclone, anhydrous caffeine (Z'=5) and a disordered form of ranitidine were presented, underlining the fact that structure solution is no longer strictly the preserve of the dedicated crystallographer.

The final day opened with a session on regulatory and patent issues. As is normal for such sessions, a number of well-known litigation cases including cefadroxil and ranitidine were discussed in order to highlight the differences between the scientific and legal arguments involved. Unsurprisingly, it was made quite clear that it was naïve to believe that 'scientific truth' was the be-all and end-all of any given litigation. The closing session, which changed tack to cover applications of small-angle X-ray scattering to pharmaceutical materials, jarred somewhat with the main thrust of the symposium, but nevertheless highlighted some interesting pharmaceutical themes, which will no doubt be built upon in future meetings.

The final day consisted of an instrument calibration and indexing workshop at the nearby ICDD Headquarters, which I was unfortunately not able to attend, but which benefited greatly from the contributions of expert tutors.

At the end of the symposium, one left with the impression that XRPD has a bright future within pharmaceutical industry. This

is a remarkable transformation for a technique that was, until quite recently, viewed as useful merely for fingerprinting or for providing a set of *d*-spacings for a table in a patent. The emergent key phrase 'high-throughput' is destined to resonate loudly in years to come. All speakers placed an emphasis on obtaining the best-possible XRPD data in order to make subsequent analysis of the data easier. It was clear that without high-quality data collection and data analysis, high-throughput XRPD will simply result in a massive collection of patterns that cannot be gainfully used.

The only disappointing aspect of the meeting was the relatively poor attendance. Some of this can be put down to the timing of the meeting and the existence of a similar analytical meeting held in the summer and an ACS meeting on polymorphism being held early in the 2003. For those who stayed away, this was an opportunity missed to hear exactly how 'solution seekers' and 'solution providers' are working closely together to push an analytical technique forward. The meeting was well organised and enhanced by an openness on the part of the speakers to discuss failures, as well as successes, and talks were (with only one exception) firmly rooted in science / technology and were refreshingly free of 'sales pitch' for hardware, software and services.

Kenneth Shankland
ISIS Facility
Rutherford Appleton Laboratory

What improvements or additions would you like to see to the Industrial Group Web pages?

What about a Web site of the month? Try: http://www.colorado.edu/physics/2000/xray/making_xrays.html
Send ideas to: djtaylor@lineone.net

Did you know?

1. The MEMBERSHIP section of the BCA website has been updated to include downloads of forms for Membership, Gift Aid, Donations, Bursaries and individual ECA membership.

2. You can keep the BCA informed of changes to your membership details by filling out an online form.

Articles Wanted:

Why not put pen to paper and write a short article for our next Newsletter. There are lots of examples to give you some ideas in our Hints & Tips section on the WEB.

We are also looking to expand the range of Industrial Applications of XRD on our WEB Site.
All we need are a few well chosen pictures and a few words. How about something on CEMENTS, MINERALS, MUSEUMS, PHOTOGRAPHY, DETERGENTS, PIGMENTS, POLYMERS

Newsletter Mailing

To keep cost down and to ensure that the newsletter gets to the appropriate people it is essential that we know your correct address. Also if there is a more appropriate contact in your organisation or if you no longer require a copy please let us know by contacting any of the committee officers.

The newsletter is also now posted on our WEB site (http://bca.cryst.bbk.ac.uk/BCA/IG/index.htm) . If you would like an e-mail notification of the WEB posting rather than a paper copy, then send an e-mail to djtaylor@lineone.net – with the title SUBSCRIBE WEB NEWS

Spring Meeting University of York, 15-17 April 2003

Industrial Group Highlights

The IG has put together a comprehensive programme based on membership feedback in our recent survey. You have the opportunity to get some real value for money (less than £100 per day full board!) training from world class experts for yourself, a trainee or student. There is something to keep you occupied over the full three days including a comprehensive exhibition featuring all the major suppliers, a real chance to update your product knowledge in a fast changing market place.

A Suggested Itinerary

Tuesday 15th April 2003

13:30 - 15:00 Plenary Session:ICCD's PDF-4 Databases: Search Indexes, Full Pattern Analysis and Data Mining.- John Faber-Principal Scientist ICDD.

15:30 - 16:00 IG Oral Poster Session

16:05 - 17:45 Introduction to Powder Diffraction

Evening - Posters and Exhibition.

Wednesday 16th April 2003

Morning 8:30 - 10:00 Workshop: Phase Identification Principles

10:30 - 12:00 Parallel Session : "High Throughput, Databases and Data Mining in Chemistry and Industry"

Afternoon13:00-14:30 and 15:30-16:30 - Workshop: Phase Identification Practice - including hands on PC session

14:30 - 15:00 Industrial Group AGM

Evening - Conference Dinner

Thursday 17th April 2003

All Day Sessions - Quantitative Phase Analysis

Lachlan Cranswick - Rietveld
Chris Dallman - Quantitative Phase Analysis using Classical PXRD Methods
David Middleton - Solid State NMR
Graham Buckton - Quantitative Analysis with Amorphous Materials
Claire Anderton - Spectroscopy

The afternoon will be devoted to discussion of the Quantitative Round Robin exercise.

SESSION DETAILS

Workshop - Phase Identification

Principal Instructor: John Faber ICDD

Organisers: Judith Shackleton & Dave Taylor

Session 1: Tuesday 16:05 - 17:45
Introduction to Powder Diffraction

Powder Diffraction is a widely used technique for identifying and quantifying phases of polycrystalline materials in industry and academe. This session introduces the Phase Identification and Quantitative workshops by explaining some of the fundamentals of the technique so that those who are not fully versed in the method will gain the necessary background to get maximum benefit from the following sessions. Even the more experienced will find this session useful - there is always something new to pick up from the expertise of our world renowned instructor!

Session 2: Wednesday 08:30 - 10:00 Phase Identification Principals

This break allows delegates to attend a relevant session on Databases & Data Mining

Continued on next page.

Spring Meeting University of York, 15-17 April 2003

Session 3: Wednesday 13:00 - 14:30 Phase Identification in Practice

Session 4: Wednesday 15:30 - 16:30 Phase Identification worked examples on PC's

This modular workshop spanning the first two days of the Spring Meeting has emphasis placed on the practical use of powder diffraction phase identification. The modules are linked to give a good grounding in best practice for phase identification and are suitable for both novice and experienced practitioners. You can choose to attend any of the modules that fit into your meeting schedule. For those who attend all the sessions an optional certificate of attendance is available for your professional development file.

The purpose of this workshop is to build proficiency in the interpretation of experimental powder data, especially in the application of the Powder Diffraction File (PDF) and new relational databases.

- The type of information the database contains.
- Its history.
- The way this information is organised.
- How data may be retrieved and interpreted.
- How to collect experimental data.
- How interpretation is affected by accuracy of experimental data.
- How to detect and understand common instrumental and specimen induced errors.
- How new relational databases extend search options.

Hands on work sessions will allow you to become familiar with the application of both printed and computer media to phase identification.

For further information, please contact:

Dave Taylor Tel: 01744 893108
E-mail: djtaylor@lineone.net
or
Judith Shackleton Tel: 0161 200 3581
E-mail: Judith.Shackleton@man.ac.uk
Manchester Materials Science Centre

Parallel Session : "High Throughput, Databases and Data Mining in Chemistry and Industry"

Wednesday 16 April 10:30 - 12:00
Chair: Sandy Blake (University of Nottingham)

10:30 David Rendle - (The Forensic Science Service, London Lab.)
Database Use in Forensic Analysis

11:00 Richard Storey - (Pfizer)
Automation of solid form screening procedures for the pharmaceutical industry and how to avoid the bottlenecks

11:30 Mariette Hellenbrandt - (FIZ Karlsruhe)
The Inorganic Crystal Structure Database (ICSD) - present and future

Workshop - Quantitative Phase Analysis

08:30 - 12:00 17th April 2003

Rietveld - Lachlan Cranswick - *to be confirmed*

Quantitative Phase Analysis using Classical PXRD Methods
Christopher I. Dallman
Pfizer Global R& D, Pharmaceutical R&D,
Ramsgate Road, Sandwich, Kent, CT13 9NJ

Powder X-Ray diffraction (PXRD) can be used to quantify the phase content of a crystalline mixture. Here the methods and limitations of the technique will be described with particular reference to pharmaceutical materials.

Continued on next page.

Spring Meeting University of York, 15-17 April 2003

NMR analysis of the structure and composition of organic solids.
David Middleton - *to be confirmed*
Department of Biomolecular Sciences,
UMIST.

This talk will give an overview of how cross-polarization magic-angle spinning NMR is used to determine the crystallographic and chemical purity of small molecules in the solid state. Also an illustration will be given of how solid-state NMR can contribute to structure solution from powders, and also of a new NMR approach for following the structural rearrangements of molecules during crystallisation.

Quantitative Analysis with Amorphous Materials - Graham Buckton - *to be confirmed*

Quantifying Crystalline Phases by Vibrational Spectroscopy
Clare L. Anderton
GlaxoSmithKline, Medicines Research Centre,

Vibrational spectroscopy (infrared and Raman) may be used to identify and characterise mixtures of crystalline forms. This talk will give an overview of the techniques, their applicability to quantitative analysis, and experimental applications.

Workshop - Quantitative Round Robin

13:30 - 15:00 17th April 2003

The afternoon will be devoted to discussion of the Quantitative Round Robin exercise.

Dr J.K.Cockcroft for the inorganic sample (a mixture of Rutile and Anatase TiO_2)

and Prof. C. S. Frampton for the organic sample (mixture of paracetamol and lactose).

For the latest details on this and other sessions check the IG web site!

Industrial Group AGM

The 20th ANNUAL GENERAL MEETING of the Industrial Group will be held at the University of York on 16thApril 2003 at 2:30pm.

Nominations are sought for Secretary/Treasurer and **four** committee members to serve for three years from April 2003.

Nominations, which shall be proposed by not less than two members of the Group and shall be accompanied by the written consent of the nominee, shall be sent to reach the Honorary Secretary of the Group not later than seven days before the Annual General Meeting.

Industrial Group Posters

Posters are invited for display at the Spring Meeting. As an extra incentive to your participation, in addition to the acclaim that your poster will no doubt bring, the Industrial Group are offering a magnificent prize of £50 for best poster.

Some guidelines follow for what we would prefer to see in our posters and our adjudicators will work from these.

Posters are encouraged that:

- are relevant to industry (including some background and value of the work to industry)
- have clear aims, results and conclusions
- concentrate on telling the story, rather than fine detail
- are not an advertisement for a commercial product

There will be an opportunity to give a brief oral presentation of the content of each poster.

For more information, contact:

Phil Holdway.

BCA INDUSTRIAL GROUP AUTUMN MEETING

Industrial Group Forum 2 -- Preliminary Details

London, Mid November 2003

The Industrial Group are pleased to announce a second Industrial Group Forum to be held over two days in London.

For more details keep an eye on the IG web pages.

To offer a contribution for this meeting please contact the individual session organiser. See the last page of this Newsletter for details.

DAY 1

Registration and Coffee

11:00 OPENING JOINT SESSION - NON AMBIENT XRD - (Contact Dave Taylor)

Lunch

Afternoon Parallel sessions: (P)Pharmaceutical SIG & (G)General Interest

(P) Non Ambient Applications:

(Contact Chris Frampton)

(G) Crystallography in Materials Science

(Contact Judith Shackleton)

Tea

(P) Polymorphism & Case Studies

(Contact Neil Feeder)

(G) Non Ambient Applications

(Contact Jeremy Cockcroft)

Evening programme:

We hope to arrange a special evening activity which will include:

Alun Bowen Lecture

DAY 2

Morning:

Joint Session:

Industrial Group Award and Lecture - Colin Small, Rolls Royce plc

Coffee

Parallel sessions:

(P) Amorphous Materials

(Contact Clare Anderton or Neil Feeder)

(G) Industrial Applications

(Contact Steve Norval)

LUNCH

Afternoon Joint session:

Instrumentation –

Past present and future

(Contact Jeremy Cockcroft)

15:30 Tea and Close

Expect to see a list of recommended local accommodation listed on the IG Web pages as details become available.

More details and registration form in the Summer Newsletter.

PANalytical XRD User Group Meeting

Millennium Point, Birmingham
22nd October 2002

A dozen or so intrepid diffractionists made their way through rush-hour traffic to the Millennium Point, Birmingham for the first 'PANalytical' XRD User Group meeting since its name change from Philips Analytical in September. The morning session opened with Users being brought up-to date with the current status of the company (Robin Aird). The new X'Celerator detector was then discussed by Martin Franson with an explanation of the 'Real Time Multiple Strip' technology that underpins its response. Paul O'Meara then went on to explain the new X'Pert Data Collector Software. There is now no need for the 'Organiser' software and X'Pert Highscore and Data Viewer now replace Graphics Identify. Uli Riedle explained the new XRDML file structure - more information about which can be found at www.XRDML.com. The session was concluded with John Benstead outlining what affects the lifetime of an X-ray tube. Essentially, it is due to filament material forming on the anode surface but it was interesting to find out what accelerates this – from temperature and water quality to tube power settings.

The afternoon session gave Users an opportunity to have their say. Contributors were David Gleeson (Nanomagnetics Ltd), Jeff Howe (Alcan Primary Metal), Sasha Babkevich (Oxford University), Mike Morris (Cork University) and Colin Small (Rolls Royce). Several of the speakers and others present, commented on the new X'Celerator detector. Users agreed that the detector greatly reduces acquisition time and one User said something along the lines of 'observations to date would indicate that 'PANalytical' is correct in its assertion that the detector is 100x quicker than other detectors' – how's that for a scientific response?

Now, let's be honest, the highlight of the day was the visit to the I-Max Cinema which PANalytical had kindly organised at the end of the meeting. We were supplied with 3D glasses. The film we saw was Cyberworld – a 3D animated film involving the hunt for a computer virus in electronic circuitry – I think! Anyway, we saw a collection of weird and wonderful 3D creatures and the film was quite impressive. However, as scientists, we were equally interested in the technology and more than one of us tried to observe the effects by rotating our 3D glasses, looking through one eye-piece only and looking the wrong way through the glasses! Overall, a very informative and entertaining day. Thank you PANalytical.

Mark Farnworth
Pilkington European Technical Centre

MEETING NOTICE:
1st Annual Biomaterials Workshop

17th March 2003 at Cranfield University.

Aimed at postgraduate students specifically researching calcium phosphates and other related biomaterials.

For more details see:
http://www.rmcs.cranfield.ac.uk/biomaterials

Note this is not a BCA Meeting.

THE SPRING MEETING REGISTRATION FORM (in PDF form) CAN BE DOWNLOADED SEPARATELY FROM THE WEB SITE.

The page is omitted in this downloaded document to minimise the size of the PDF file.

BCA INDUSTRIAL GROUP

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