

BCA IG Newsletter

September 2004

EDITORIAL

Welcome to this latest issue of the IG newsletter. In this issue we have our usual reports, including the excellent Spring Meeting, along with details of some exciting forthcoming events. Please do try and come along and support these. The Industrial Group Committee would welcome your comments and ideas for future meetings. The Industrial Group is organising a 'DIY Crystallography' meeting this autumn and need some contributions urgently. I'm sure that many of you have come up with novel solutions to problems with sample preparation, sample holders and even data analysis. Why not share it with others. As part of this meeting there will also be a session on Public Domain Software. If you know where to look, there is a lot of excellent software available, which can meet all your requirements. So please come along in November and find out more. Another worthwhile exercise is the low angle round robin and the instrument calibration test, details of which are in this newsletter. Both of these exercises will help provide you with information about the alignment of your x-ray diffractometer. Finally a big thank-you for all the contributions and as always, I would ask that you write a report for the newsletter if you attend any x-ray related meeting.

Phil Holdway

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Forthcoming Events 2004-2005

Registration available on the WEB.

Pharmaceutical SIG - AstraZeneca, Alderley Park

Tuesday 5th October 2004

See page 9 for list of speakers

Registration fees: £15:00 - £45.00

Contact: Dr Anne Kavanagh

Email: anne.kavanagh@astrazeneca.com

INDUSTRIAL GROUP AUTUMN MEETING 'DIY Crystallography'

Birkbeck College, London 4th November, 2004

Papers urgently required (see page 8)

Contact: Dr Jeremy Cockcroft.

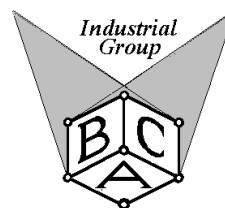
E-mail: cockcroft@img.cryst.bbk.ac.uk

BCA 2005 SPRING MEETING

Loughborough University 12th – 14th April 2005

Details on the web site and in the next newsletter

PLUS NEW XRF content (see page 9)



Charity Registration Number: 284718

World Wide Web addresses:

BCA <http://www.crystallography.org.uk>

IG <http://www.crystallography.org.uk/ig/ig.htm>

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BCA SPRING MEETING REPORTS

A Report on the SAS Programme of the BCA Spring Meeting held at UMIST April 2004

The SAS programme really started on Wednesday morning during the Instrument Calibration session with Dr. Manfred Kriechbaum from the Institute of Biophysics and X-Ray Structure Research, Austrian Academy of Sciences; Practical Aspects of SAXS in Industrial Research:

In this talk the technical features, set-up and application fields of a modern laboratory small-angle X-ray scattering camera in industrial research were presented. Emphasis was put on the nanostructural information content of SAXS in condensed heterophase (porous) systems; very common in industrial research and quality control, where SAXS can be an excellent complementary and even superior method (with respect to gas sorption methods) for the determination of inner surfaces and interfaces, as it 'sees' also enclosed pores or can be applied for wet samples. Industrial laboratories are also often faced with the task of high-throughput screening of multi-component formulations where the SAXS method can yield quickly nanostructural information when the SAXS camera is equipped with a flow-through sample cell attached to an Autosampler. Furthermore, there is also the possibility to combine SAXS simultaneously with another thermal or spectroscopic method (E.g. simultaneous SAXS-DSC measurement).

To start the first parallel SAS session it was Vladimir Kogan of PANalytical with a presentation entitled Comparative SAXS Measurements Performed by Different Techniques

Nowadays modern laboratory x-ray diffractometers are being considered as possible platforms to perform SAXS experiments, challenging the performance of dedicated systems. Vladimir presented the preliminary results of comparative SAXS measurements on polymers and bio-polymers, obtained using different experimental set-ups.

This was then followed by Manfred's second talk of the day; Time-Resolved SAXS Measurements at Synchrotrons:

The high-flux and brilliance of X-rays from modern 3rd

generation synchrotrons enable us to follow the nanostructural development, the supramolecular assembling or molecular mechanism of phase-transitions in situ and in real-time with a time-resolution in the millisecond time regime by small-angle X-ray scattering. As examples, a variety of such time-resolved experiments, mostly on biological samples, performed at the SAXS beamline at ELETTRA, Trieste, Italy, were presented. Among them T-jump and p-jump relaxation studies on phospholipid phase-transitions followed by time-resolved SAXS as well as individual set-ups and current limitations were discussed.

This was followed by Mary Vickers, with Solid Polymers to Particles in Solution, which covered topics as diverse as;

Oriented lamellae in blown polyethylene film,
Lamella repeat distances, tie molecules and mechanical properties,
Lyocell (Tencel) and voids,
Hair and breast cancer,
CaCO₃ in fuels & lubricants,
PbZrTiO₃ particles in suspension.

The final SAS session of the afternoon was from Andrew Harrison of Edinburgh University with "In-situ diffraction studies of microwave driven processes in materials chemistry and biology"

Andrew described how microwave heating is becoming increasingly important as a method of driving chemical synthesis and materials processes, both in solution, and in the solid state. He also highlighted the increasing concerns about the possible harmful effect that such radiation – as used in mobile 'phones - may have on biological tissue, over and above what might be expected from consideration of the likely heating effects at low exposure level. However, both aspects of this field are served by almost no direct experimental measurements of the nature of such effects. The team at Edinburgh University have developed several different types of microwave reactor that enable them to perform X-ray or neutron scattering measurements on powders, single crystals, colloidal and liquid crystal systems during microwave irradiation, and to measure temperature accurately and precisely. He described the principles of designing and operating such equipment, and described some of their work on colloidal growth, and the potential to use such

BCA Spring Meeting (continued)

methods to probe the possible effect microwave radiation may have on the structure of proteins and biological membranes.

Thursday 8th April saw the Small Angle session kicking off with Dmitri Svergun of the European Molecular Biology laboratory – Hamburg Outstation with Small Angle Scattering Studies of Biological Macromolecules in solution.

Small-angle scattering of X-rays and neutrons (SAS) is a fundamental tool in the study of biological macromolecules. SAS allows one to study the structure of native particles in nearly physiological solutions and to analyse structural changes in response to variations in external conditions. The scattering data bears information about the overall shape and internal structure at a resolution of 1-2 nm. The method is applicable to a broad range of sizes, from individual macromolecules to multi-domain proteins and large macromolecular assemblies. Recent progress in instrumentation and data analysis significantly enhances resolution and reliability of structural models provided by the technique and makes SAS a useful complementary tool to high-resolution methods, in particular, in large-scale structural studies.

Advanced methods to analyse X-ray and neutron scattering data from solutions of biological macromolecules were presented including: ab initio low resolution shape determination; modelling of quaternary structure by rigid body refinement; ab initio analysis of medium-angle data to obtain domain structure from X-ray data; the use of specific deuteration combined with contrast variation in neutron scattering to construct detailed inhomogeneous structural models; addition of missing loops and domains to high resolution protein models; quantitative analysis of equilibrium mixtures of oligomeric proteins. Practical applications of the methods were illustrated by recent examples.

This was then followed by a chap called Richard Morris from Huntsman Surface Sciences, Oldbury with; "He Scrubs Up Well – Doesn't He?" or, New Aspects of Expanded Lamellar Surfactants – Aqueous, Sugar Based Exfoliates.

Currently there are two types of body scrub available; aqueous and non-aqueous. Aqueous scrubs contain insoluble abrasives e.g. shells, seeds or pumice and

are sold as pastes, gels or thickened emulsions. Non-aqueous scrubs contain soluble abrasives i.e. sugar and salt. Oils, glycols and silicones are used in place of water. The disadvantages of these products are that the aqueous scrubs are viscous, difficult to manufacture and the insoluble abrasives may lead to bacterial contamination. The non-aqueous scrubs are pastes, which have an oily or greasy feel, difficult to spread and are expensive. However, it is now possible to produce water based sugar scrubs! It has been discovered that surfactants can be made to form liquid crystals in saturated sugar solution.

We can see from the SAXS data that the inter-lamellar spacing is in the Region of 500 Å. These liquid crystals can support additional solid particles of sugar. The solid sugar crystals are suspended indefinitely and act as an exfoliate. The presentation demonstrated the application of SAXS in the development of new surfactant technology and Richard also gave a brief demonstration of how to make your own sugar scrub from everyday chemicals found in the kitchen.

Markus Winter from Bruker then followed with SAXS from multi-functional XRD instruments; NanoSTAR - The Universe of Nanostructure Analysis that described the new Bruker XRD instrument.

Richard Heenan of ISIS at RAL delivered the final morning session; SANS – Practicalities and prospects

Small angle neutron scattering, SANS, remains a powerful tool in many areas of science, despite many advances in X-ray instrumentation. Accelerator based, pulsed neutron sources, such as ISIS, are the way forward for the future of neutron scattering. Major new pulsed sources are under construction in the USA and Japan and a second target station, optimised for cold neutrons, is to be built at ISIS which will enable a new world class SANS facility within the UK. The advantages of pulsed source SANS were outlined and examples given of SANS science from both LOQ at ISIS and the continuous source D22 instrument at the ILL reactor in Grenoble. SANS contrast variation, using deuterated components enables unique information to be obtained on increasingly complex systems. Complex sample environment such as pressure cells, flow and shear cells and stop-flow techniques are readily available to map system behaviour rather than simply measure single structures.

Thursday afternoon saw Pete Laity from Cambridge

BCA Spring Meeting (continued)

with Scattering from segmented Co-polymers – a reinterpretation of SAXS data;

Small-angle X-ray scattering (SAXS) has been widely used to study the microphase-separated morphology exhibited by polyurethanes, which arises through the immiscibility of 'hard' and 'soft' chain segments. The results are often analysed on the basis of a lamellar model, which appears to be justified on the basis of the expected volume fractions of 'hard' and 'soft' microdomains.

Peter's recent work has re-examined the SAXS data from a series of segmented co-poly (ether-urethane)s, using a number of alternative morphological hypotheses. The results suggest that a lamellar interpretation might not be the best model for polyurethane systems. On the contrary, the scattering data obtained from the co-poly(ether-urethane)s under various experimental conditions could be reproduced using 'globular' scattering models of the Zernike-Prins or Percus-Yevick types. This suggested relatively small volume fractions of hard segment microdomains and a significant persistence of segmental mixing. Analysing the SAXS data by curve-fitting these models has revealed considerable new insight into the morphological response to deformation in these materials and indicated possible links to mechanical and thermal behaviour.

Next up was Mark Farnworth from Pilkington Glass with X-ray reflectivity in the glass industry;

Mark described how the techniques of XRPD, GAXRD, pole figures and X-ray Reflectivity measurements can be used to examine samples from all stages of glass manufacture, from the crystalline raw materials to the amorphous final product. GAXRD is used to examine thin coatings on the glass surface of hydrophobic coatings, which disperse water. Refractory materials are examined to determine how much non-crystalline material is present and the amounts of quartz, cristobalite and tridymite. Also, multi-layer stack coatings are examined E.g. Ti ZrO₂ Ag Si.

Texture maps can be produced which show the degree of texture in the silver layer of the coatings and the thickness, density, top and bottom roughness of each individual layer can be measured.

Finally, Richard Clapperton of Huntsman Surface Sciences Oldbury gave a fascinating insight into the

world of detergent research with: SAXS Interpretation of Deflocculated Vesicles - The Route to Superconcentrated Detergents

Richard described how liquid detergents contain concentrated solutions of surfactants and their phase behaviour determines the key physical properties of the detergent, such as viscosity and storage stability. Whilst the latter can be measured through techniques such as rheometry, and phase can be identified by optical microscopy, there are few tools available to the formulator to characterise the liquid on a molecular level. Small Angle X-ray Scattering (SAXS) is a fast, versatile technique that enables the finer details of surfactant phases to be determined. This includes confirmation of phase type and measurement of phase dimensions, such as water layer thickness between surfactant bilayers. The effect of additives on phase structure can be monitored by SAXS, thus assisting product development. The challenge of achieving superconcentrated detergents has been greatly simplified by SAXS characterisation.

This concluded the SAS sessions and everyone agreed that it had been an enjoyable and successful event.

Richard Morris June 2004

****SPONSORS WANTED****

The Industrial Group is always looking for new sponsors for the group newsletter.

Potential sponsors, whether individuals or companies, should contact the editor or any member of the Industrial Group Committee for further information.

Sponsors will have an acknowledgement on the front page of the newsletter and if relevant, the company logo and web address.

Ind Group E-mail Mailing list – Online registration.

The IG sends about six E-mail notices each year to anyone interested (You don't even need to be a BCA member!). These inform of Newsletter postings and the various meetings we organise each year. You can now register for our E-mail list online - follow the link from the IG home page. There is an opportunity to be removed from the list with each mailing.

Instrument Calibration Session “How to be a star”

Introduction - *Dave Taylor and Jim Kaduk*

The opening introductory session was shared by Dave Taylor and Jim Kaduk (the new ICDD Chairman). Jim explained the criteria for a star quality pattern in the ICDD powder diffraction file. Dave went on to cover the standards available for instrument testing including non-ambient calibration and gave a reminder of the lists maintained on the IG web pages. The rapid expansion of the PDF is a significant factor in driving us towards better instrument alignment to ensure that phase identification software pulls up the right matches with the push to tighter search windows. The need for testing was demonstrated with examples from the IG Instrument Sensitivity Round Robin and a new spreadsheet will be available soon to automate checks. The basics of instrument alignment were then covered. Dave also announced details of a new low angle Round Robin based on a Ag Behenate film being launched by the IG this summer. Jim rounded off by explaining the procedures for submitting “star” quality patterns to ICDD for all those new compounds currently missing from the PDF database.

Aspects of calibration in SAXS - *Manfred Kriechbaum*

Manfred covered the difficulties of calibration for SAXS measurements with nothing in the way of absolute standards. Rat-tail (tendon collagen) with a spacing of approximately 67nm is used for checks but it is not very stable especially to moisture. He also gave an overview of the role of SAXS at the Austrian Academy of Sciences.

The European XRPD Standard - *Steve Norval*

Steve explained the background to this standard. For XRPD, there are different instruments, techniques and applications. It is not the easiest technique to standardise. The motivations for putting together a ‘standards’ document include good practice, a point of reference for producing acceptable data and demonstrating competence. The title of the document is ‘X-ray Diffraction from Polycrystalline and Amorphous Materials’. It has been put together by a working group of a technical committee of the European Commission for Standardisation. ‘General Principles’ and ‘Procedures’ documents were released in 2003 under the following codings: BSEN 13925-1 and BSEN 13925-2 respectively. The aim is to release a third document ‘Instruments’ this year - BSEN 13925-3. Beyond that, documents on ‘Reference

Materials’ and ‘Terminology’ will be released. Steve gave an overview of the contents of the ‘Instruments’ document. It covers issues such as types, components, calibration and testing. For the control of a diffractometer there is component selection, component configuration, alignment and testing. Procedures have been established for calibration and instrument alignment/verification.

Lined-Up or Spot-On? The Ups and Downs of Diffractometer Alignment - *Martin Vickers*

Martin gave a very useful and practical guide to the alignment of the equipment at Birkbeck College which includes both transmission and reflection geometry. Good alignment produces accurate 2-theta positions, maximises intensity and produces nice peak shapes. Useful tricks and a well thought out approach, designed especially to assist Bruker Users in the tricky art of alignment, were well received.

Stress Instruments - *Judith Shackleton*

Judith gave a good overview of the approach required for residual stress analysis and stressed the importance of accurate results when measurements rely on very small changes in high angle measurements. Additional complications arise with the complex shapes of the components for analysis. Stress is calculated from Hook’s Law [Young’s Modulus – Stress/Strain] using the $\sin^2\psi$ method. In effect, the crystallographic planes act as an atomic scale strain gauge. A stress-free standard is not required and the method is easy to carry out, however, the method is only sensitive to the top few tens of microns of a surface. She explained how an algorithm developed in conjunction with Rolls Royce was being applied to peened samples.

Calibration for Silver Halides - *David Beveridge*

David explained the problems associated with the photographic industry and the difficulties of resolving phases which requires the very precise measurements of a few lines at moderately high angles. Calibrations are required for peak position, width and peak profile. Peak widths are often variable and depend upon chemical variability, strain and grain size. Often in house standards are required as calibration aids. The session ended with the chair **Jeremy Cockcroft** thanking the speakers for their contribution to an interesting session.

Mark Farnworth
Pilkington European Technical Centre

PANalytical User Symposium – 28th/29th April 2004

The meeting was held at Dunchurch Park, Nr. Rugby – a former manor set in 30 acres of picturesque woodland and grassed areas. The symposium combined a series of technical presentations on the X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) techniques with the UK launch of the Axios XRF Spectrometer.

Robin Aird (PANalytical Ltd) described the new X'Pert Highscore Plus XRD software available from PANalytical. Robin said the software is a complete powder pattern analysis tool, covering phase identification, crystallographic analysis and Rietveld calculations. All of the common and many of the less common data formats can be imported into the software and it supports the internationally recognised CIF format for the publication of XRD data. The Reporting interface has MS Word compatibility.

Leain Grimsley (British Geological Survey), spoke about her work in the sampling and analysis of soil in the Tavistock district of Devon. The PANalytical MiniPal 2 energy dispersive (ED) XRF instrument was used to measure As and Zn in soil. The MiniPal showed good accuracy at the 0.1% level. Portable XRF's can analyse up to 40 samples per day

Chris Staddon (Nottingham University) described his work on 'Spintronic' materials which consist of alternating layers of low temperature and high temperature GaAs and GaMnAs. A Materials Research Diffractometer (MRD) is used to obtain reciprocal space maps and to obtain strain measurements. Chris said that strain has been seen to relax as the coating thickness goes above a critical limit. Annealing the coatings produces changes in the XRD pattern which provides insights into diffusion mechanisms.

Rob Foster (Exposure control section, H&S Laboratories) described the XRF metal rubbing technique in which diamond coated 'lapping'

paper is used to obtain specimens from metal surfaces. The technique is non-destructive, can be used on any size of item and does not require the metal item to be taken to the lab. Rob said that 0.003mg of Cr, Ni, Mn and Fe could be determined within 1% of certified values.

Richard Morris (Huntsman Surface Sciences) talked about a sugar based structured surfactant system. It is a novel aqueous delivery system that comprises surfactant, carbohydrate and water. Sugar induced 'Lamellar' sheets are formed with a spacing in excess of 500 angstroms. Richard explained how SAXS is used to measure the bi-layer spacing, to 'tune' formulations and to determine thermal stability. These systems suspend solid particles and allow the system to flow as a normal liquid.

Cyril Marchant (PANalytical Ltd) explained that instrument breakdowns and performance problems fall mainly into three categories: vacuum, water and software, with the majority of situations relating to the first two. He gave a number of basic 'ground rules' to reduce running costs. Ensure that the gas bottles (e.g. He and Ar) don't run completely empty before they are replaced. Check gas bottles for leaks at regulators and unions (use soapy water and watch for bubbles). Ramp up the kV and mA when the tube is cold and don't switch it off from high power (ramp it down slowly). Use sleep and dose channels when the instrument is not being used and tube 'breeding' to run up slowly. The water supply needs to be set-up correctly and maintained. Periodically top-up a circulating water system and check external water filters. Solid samples should be clean and dry.

Martijn Fransen (PANalytical Ltd) spoke about non-ambient measurements using the X'Pert Pro instrument. Martijn addressed some issues concerning non-ambient chambers. Sample height should be measured before heating and corrected 'on the fly' or afterwards. Parallel beam optics are better than focusing optics since sample height can move over 1 mm without a

PANalytical User Symposium (continued)

change of d-spacing. A d-spacing can be measured as a function of temperature and compared with literature values or a temperature calibration point defined where a known phase transition takes place.

Philip Russell(PANalytical Ltd) discussed detection limits of difference XRF instruments. WDXRF instruments (broadband excitation and selective detection) provide the best detection limits, generally for light elements especially those lighter than zinc. Detection limits for 'sequential systems' (those that scan elements one at a time) are a function of the total number of elements detected. EDXRF instruments (selective excitation and broadband detection) have best limit of detection for elements with atomic number >40 (Nb and higher). Practical limit is a function of the complexity of the system – e.g. presence/absence of major elements in the vicinity of the elements of interest.

Mark Russell(Queens University) described his work on Portland cements. He uses an Anton Paar Humidity chamber to control the temperature and moisture environment of his cement samples. The hydration characteristics of new and blended cements are determined which are then used to predict reactions and design new mixes.

Heather Harrison(British Geological Survey) described the use of the Epsilon 5 polarised EDXRF for the measurement of soil samples in the Merseyside and North Wales areas. The high excitation voltage gives good results for high Z elements. 300 samples per week are measured and have produced a geographical map containing 35,000 samples!

Reg Nichols(PANalytical Ltd) announced the UK launch of the Axios-Advanced spectrometer, an instrument equipped for 'ultimate precision and demanding light element applications'. He said it delivers unmatched performance incorporating numerous cutting-edge technologies such as the SST-Max tube.

Phil Russell (PANalytical Ltd) gave Users more detail about the instrument. It contains a Rh-anode SST-Max tube which provides tube stability all the way up to 160mA improving analytical performance in the light element range. There is an integrated sample changer and a choice of loading mechanics which can be used to analyse up to 168 samples at a time. It can measure and analyse a variety of samples including solid pieces, pressed and loose powders, fused beads, liquids, foils, granules and thin films. There is also He flushing and a dust collection device. The software includes SuperQ 4, IQ+, Pro-trace, EP-multi, EDS and SPC. Phil said that with a minimum of training, most day-to-day operation is a push-button exercise.

Mark Farnworth

Pilkington European Technical Centre

Please note: a longer version of this report can be found on the IG website:

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. Don't forget, if you attend a conference, please send in an article about it.

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

A Low Angle Instrument Calibration Round Robin

The BCA Industrial Group is pleased to announce a NEW low angle calibration round robin.

The round robin is being co-ordinated for the Industrial Group by Dave Taylor.

The sample being used is a thin Silver Behenate film on a Polyethylene terephthalate (PET) substrate. It will be distributed as a 30mm square specimen and can be easily cut to size with scissors to fit your specimen holder. Participants in the round robin will need to measure the specimen from 1 degree to 40 degrees 2 theta for Copper Radiation or the equivalents for other wavelengths. The instrument conditions are left to participants with a recommendation of a minimum of 0.01 degree steps with a 1 second per step time interval and taking just over an hour. A template will be supplied for reporting results, which will include instrument parameters and the measured data as an angles, intensity - x,y list.

Each participant will be given a unique number to protect their identity in the final report but allow them to see how their data compared with the rest. It is expected that information on angular displacement, peak intensities and line profiles will form the basis of the data evaluation. All participants can keep their test specimen as a valuable instrument calibration resource. Subject to the results of this UK Round Robin the exercise may be rolled out world-wide and the test specimen may become a certified reference material.

Why use a Silver Behenate film?

The material orients on the substrate to give multiple orders of the basal Bragg reflection. The fact that it is a reproducibly deposited film will allow direct comparison of the intensity and line profile of each instrument with no contribution from the sample or its preparation.

It is a silver carboxylate, $C_{22}H_{43}O_2 \cdot Ag$ and crystallises as a dimer

Crystallographic information of the bulk Silver Behenate:

Triclinic Unit Cell ($Z = 2$)

$a = 4.71 \text{ \AA}$, $b = 4.15 \text{ \AA}$, $c = 60.30 \text{ \AA}$

$\alpha = 104.5^\circ$, $\beta = 93.2^\circ$, $\gamma = 75.6^\circ$

$\rho_{\text{meas}} = 1.30 \text{ g/cm}^3$, $\rho_{\text{cal}} = 1.35 \text{ g/cm}^3$

Sign up on line to take part now!

The BCA gratefully acknowledge the support of Tom Blanton at Eastman Kodak Company, USA, for supplying the material used in this round robin exercise.

Call for Papers

**Industrial Group Autumn Meeting,
DIY Crystallography – Thursday 4th
November 2004, Birkbeck College,
London.**

Offers of short presentations of 5 - 20 minutes are urgently required for this informal meeting which will encourage a sharing of ideas. Lots of useful ideas are implemented in labs around the country to make something work better, be more efficient or save money. We are looking for short talks describing low cost innovations that can be shared with the powder diffraction community. We expect to cover hardware, modifications, specimen holders, public domain software, etc. The list is only limited by your ingenuity.

To offer or discuss a possible contribution please contact:

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Watch the WEB for programme and a registration form.

INDUSTRIAL GROUP AWARD

It is the intention of the BCA Industrial group to make an Industrial Crystallography Award to suitable UK crystallographers working in industry or in academic institutions. The Award will be given in recognition of a sustained contribution to industrial crystallography including crystallographic and diffraction work of all kinds.

The Committee of the Industrial group will make the final decision concerning the Industrial crystallography Award. They intend the Award to take a form that fittingly marks the contribution made by the recipient.

Nominations for this Award are invited now and should be sent to the Secretary of the Industrial group. Besides the name and affiliation of the person proposed, nominations should state briefly why she or he merits the Award, giving a brief account (ideally not more than one sheet of A4) of her or his crystallographic work and its industrial significance. If desired, the proposer may suggest the form that the award should take.

Future Meetings – Further Details

Pharmaceutical SIG - 5th October 2004, AstraZeneca - Alderley Park.

Alphabetical list of speakers:

Rebecca Booth (AstraZeneca) – 'Hydrate or anhydrate: that is the question'

Ed Collier (UMIST) – 'A crystallisation/crystal engineering approach to aid salt selection'

Chris Gilmore (University of Glasgow) - 'The hunt for polymorphs using PXRD'

Chris Hunter (University of Sheffield) - 'A 1H NMR study of crystal nucleation in solution'

Gareth Lewis (AstraZeneca) - 'In-silico procedures for phase identification and quantitative phase analysis'

Doug Minick (GSK) - 'Ab initio vibrational circular dichroism (VCD) and optical rotation: new tools for the determination of absolute configuration'

Colin Pulham (University of Edinburgh) - 'High-pressure recrystallisation - a new method of screening for polymorphs and solvates'

Fees : £15:00 - £45.00

Please register for this meeting using our on-line Registration Form

Contact:

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NEW!! XRF Sessions at the 2005 Spring Meeting

Loughborough, 12-14 April 2005

The BCA is pleased to announce the introduction of parallel X-ray Fluorescence (XRF) sessions to run alongside the usual crystallography content. XRF has been introduced after discussion with exhibitors and will be organised by the Industrial Group. This will provide a much needed forum for users of all manufacturers XRF equipment and will cover both energy dispersive (EDXRF) and wavelength dispersive (WDXRF). The scope of the commercial exhibition is extended to include XRF equipment and supplies.

Call for Papers

Offers of 15-25 min presentations are most welcome. Planned sessions include: XRF Exhibitors - state of the art equipment and consumables, Awkward samples (workshop), Liquid samples, Applications of WDXRF and EDXRF, Standards & Calibration and Light Element Analysis (Li-F or wavelength >1.3nm). To offer a contribution, please fill out our web form.

Please tell your friends and colleagues!

Please help us to spread the word about the XRF content at the 2005 Spring meeting by letting your friends and colleagues know. The XRF community is not covered by our current mailing list so we do need your help!

Register your interest

Do you want to be kept informed about the XRF programme and be sent registration details? If so then please fill out your details on a form on our web site and we will keep you updated.

Check the IG WEB pages for specific programme information.

Industrial Group Committee

2004-2005

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INSTRUMENT CALIBRATION AND INTENSITY TESTING

A Spreadsheet to evaluate a Standard Corundum Diffractometer Scan

A spreadsheet using macros has been developed from the Intensity Round robin which will allow you to test the performance of your diffractometer using a single scan and check that it is giving the equivalent of ICDD PDF star quality data.

The spreadsheet has been designed to read the proprietary ICDD .REF file format for use as an evaluation tool prior to pattern submission to ICDD. However, it is equally suited for use in any laboratory using manually pasted data and file conversion routines.

You will need a standard Corundum sample. NIST SRM1976 plate is the preferred standard (it avoids sample preparation induced errors including preferred orientation) but you can also use SRM676 powder. The use corundum powder from another source is possible, just use the SRM676 option during processing.

Bruker users will find a spreadsheet (scanplot101.xls) developed by Steve Norval provides a useful bridge and other file converters are planned. You may want to check out the available data conversion options on the CCP14site. ConvX with output as an ASCII file which can be read into a new spreadsheet works well.

The spreadsheet is available by sending an E-mail to Dave Taylor (djtaylor@lineone.net) with the title RR Spreadsheet. You will be notified of significant updates and file converters.

More information is available in a PDF file on the Industrial Group website.

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