



e-newsletter

Issue 4, 2011

Australian X-ray Analytical Association

President's Address

Dear AXAA Members and Readers,

A quick look back through the AXAA activity calendar reveals that 2011 has been a very busy year. AXAA became an incorporated body, formed a new National Council, hosted the 2011 Workshops, Conference, and Exhibition event, and overhauled our membership rules. Our Newsletter got a facelift and a new section, thanks to Newsletter Editor Dr. Nathan Webster – congratulations on your first full year of Editorial work.

This year saw major achievements for many members - Mark Raven won the prestigious 5th Reynolds Cup, Prof. Brian O'Connor became a Distinguished Fellow of the International Centre for Diffraction Data (ICDD), and I was a finalist for the Eureka Peoples' Choice award. Several of our student affiliates received awards from the ICDD to attend the Denver X-Ray conference this year too. Congratulations to all.

Sadly, this year also sees the passing of Prof. Bob Snyder, who will be greatly missed, read on in this issue for a tribute to him.

Don't be fooled into thinking that AXAA activities are slowing over the festive season, even this month our members reached out into our regional community via the web – read on in this issue for "XRF Training Success for OTML Staff in Papua New Guinea through the Internet XRF Course".

Next year will see the return of our regular AXAA activities – the student seminar days and technical meetings, important events run in states with active membership. 2012 will see one of each event run in at least two states.

Finally, best wishes from the AXAA National Council for a safe and enjoyable festive season to you and your families.

Happy holidays!

Vanessa Peterson
National Council President

Editorial

We will continue our "Matters for Scatterers" section of the newsletter in 2012 – if you know of any researchers, especially students or early career scientists, who could have their work X-posed in this forum please let one of the Council members know.

A big thank you to all of those people who have contributed articles, reviews, events, company news and advertisements to the newsletter throughout 2011.

Nathan Webster
Newsletter Editor

XRF Training Success for OTML Staff in Papua New Guinea through the *Internet XRF Course*

Eight staff from Ok Tedi Mining Limited (OTML) in Tabubil, which is situated the Western Province of Papua New Guinea (PNG), have completed Advanced Level certification for the *Internet XRF Course* directed by Dr. Brian O'Connor of Perth. The Advanced Level course is designed for people capable of developing new XRF methods.



OTML staff with their certificates in recognition of completing the Internet XRF Course. They are – STANDING from the left: Steven Meibo, Wilfred Ted, Nou Ako and Jack Apipnok. SEATED from the left: Hillary Turnamur, Gloria Samiak, Doris Turbarat and G'Selle Parua.

In addition to Brian O'Connor running the course via the internet, strong local support was provided by Brian Evans, OTML Development Chemist and Gabriel Saulep, Chief Chemist at the OTML Mill Laboratory.

Gaining a course completion award for the Advanced Level course requires participants to gain a high mark in the assignments set for all 11 modules of the course. Those gaining a very high mark were awarded Advanced Level certification 'With Distinction'. The awards were presented at Tabubil in December 2011. The OTML staff completing the course are shown in the photograph with their certificates.

The exceptional efforts of Steven Meibo, G'Selle Parua, Doris Turbarat and Hillary Turnamur were rewarded through the presentation of advanced level certificates 'With Distinction'.

The training exercise was a major milestone for OTML in achieving world-class XRF capability under Brian Evans' leadership.

Brian O'Connor

fun was his period leading ICDD. He was an ultra enthusiast on the things that mattered to him – his family, his legion of friends and his science.

Bob and his dear wife Sheila travelled frequently to the most distant points of the globe, on all continents, to support local conferences and workshops. We were most fortunate that he especially valued coming to AXAA National Conferences. He loved Australia, and our reds became his favourites, particularly our Shiraz wines with Bob pronouncing 'shiraz' with such obvious relish. I was especially fortunate to share a bottle of Grange with Bob some years ago. It was a great moment when Bob first sipped the great red.

Bob's contributions to science are recorded in the ICDD obituary. One of many significant achievements cited in the tribute is the book "Introduction to X-ray Powder Diffractometry" that Bob co-authored with close friend Ron Jenkins. The book is a must for any X-ray diffraction laboratory.

Farewell Bob.

Brian O'Connor
Former AXAA President

Vale Professor Bob Snyder (1941-2011)



Bob Snyder, the American X-ray powder diffraction dynamo who contributed so much to the advancement of analytical science, passed away on September 1st, 2011 from complications due to cancer. It is exceedingly difficult to accept that he has left us. As the saying goes, 'they threw away the mould when they made Bob Snyder'. He was in so many ways a unique and wonderful human being whose commitment to others and to science knew no bounds.

AXAA Members are referred to the remarkable tribute at <http://www.icdd.com/profile/whatsnew/bobsnyder.pdf> which was crafted so lovingly by Tim Fawcett, Executive Director of ICDD.

I had worked closely with Bob on the ICDD Board when he was Chairman. What an excitement and time of sheer

Book Review

"UNDERSTANDING XRF SPECTROMETRY"

by James Willis and Andrew R Duncan (published by PANalytical B.V., The Netherlands, 2008).

No-one that I know of has made a career of XRF analysis voluntarily: most analysts have been more or less strong-armed into the field because "you know something about X-rays". It is not, to my knowledge, routinely taught in analytical chemistry courses at Universities to any depth and, indeed, is viewed with some suspicion by the Chemistry fraternity because it does not involve liquids and volumetric flasks.

Therefore, those of us who have been given this career change have not only had to come to grips with analytical requirements of XRF but also X-ray physics and a whole slew of instrumentation that frankly looks like a bunch of washing machines. This we have had to do quickly and on the fly.

The books written by Willis and Duncan are an invaluable aid to all those in the XRF field of analysis as they not only cover all aspects of this technique, but do so concisely and with ample examples.

The volumes cover topics from X-ray physics, to spectrometers (both wavelength and energy dispersive varieties) and also the mathematics of quantitative analysis. This latter topic covers absorption corrections

and the various models in existence for their calculation, statistical analysis, errors..... the list goes on.

In addition to this, there are extensive chapters on sampling and sample preparation. Because the detail, especially in the instrumentation and quantitative analysis sections, is quite general these volumes are suitable for users of all the different XRF manufacturers.

Both volumes have the same X-ray tables which include the wavelengths and photon energies for the principal X-ray spectral lines, the mass absorption coefficients for various wavelengths, absorption edge jump ratios, fluorescence yields and the ever-useful oxide-element (and vice versa) conversion factors. The fact that these Tables are in one book is good enough reason alone to consider their purchase.

These books would have been a great help in the early days of my XRF career. Having also attended the course given by James Willis in conjunction with their publication, I now know what I should have known some time ago, but didn't have time to find out.

My advice is, if you are serious about making XRF your career choice, is to invest in these publications as they will certainly enhance your ability to make the correct decisions about managing your XRF analysis.

Volumes I and II are available in hard cover from PANalytical, contact scott.gilroy@panalytical.com

Dr. Sharon Ness
XRF/XRD Specialist
Sietronics Pty Ltd

Membership Matters

We remind readers that AXAA-Inc membership is for the 3-year period starting from the last AXAA National Conference (existing membership will be re-approved in 2014). Membership is free. Candidates should provide their CV and a short sentence about what they intend to do in the organisation, and a nomination letter from an existing AXAA member (if possible). Please send these to National Council Secretary Natasha Wright. The council votes on membership at periods no longer than 6 months.

Upcoming Events, Deadlines

1. Internet XRF Course: Series 5, 2012

The course provides XRF analysts, particularly those new to the field, with on-site instruction in the practical principles of wavelength dispersive XRF. Features of the course include -

- Start at any time, subject to the availability of places in the course.
- Self-paced instruction to accommodate the needs of busy people.
- Study materials transmitted as e-mail attachments in the form of 11 modules; with an assignment being set for each module.
- Feedback on the assignments provides excellent mentoring.

The course now has a substantial number of international participants, as well as Australians.

Course availability: Starting date by arrangement.

Course director: Dr Brian O'Connor

Course fee: \$2,530 including GST

Further information and enrolment:

brian_oconnor@iprimus.com.au

Tel: (08) 9291 7067

2. National XRD Course X-ray Powder Diffraction Analytical Methods

Curtin University, Perth

Sat 25 August – Tue 28 August, 2012

Venue: Department of Imaging and Applied Physics, Curtin University, Bentley (Perth), Western Australia

[Client-specific version of the course can be presented at the customer's site]

Duration of Curtin course 4 days

Dates: 25 – 28 August, 2012

Course Presenters: Professor Brian O'Connor and Dr Robert Hart

Enquiries and further information:

B.O'Connor@curtin.edu.au

Cost: \$2,539 including GST

Availability of places strictly limited.

Overview: The course has been designed to give participants a theoretical and practical grounding in the principal characterisation methods which make use of X-ray powder diffractometry data. Approximately 60% of the course involves hands-on instruction. Participants personally collect diffractometry data sets and then process these, both manually and with PC computers, in exercises on various analytical methods, including Rietveld analysis. Public domain software will be used, including *WINPLOTR* and *Rietica*. The course also includes overviews and demonstrations of the commercial software packages *X'Pert HighScore Plus* and *DiffraPlus Topas*. While the course is relevant to the analysis of all classes of crystalline materials, attention is devoted mainly to materials relevant to the mining and mineral processing sector.

Upcoming Events, Deadlines (cont'd)

3. Siroquant Training Course

Location: Sydney

Date: 27th to 29th March, 2012

Venue: TBA

Course Fee:

New and Intermediate Users (3-day Course): \$2,800

Advanced Users (2-day Course): \$2,200

Early bird and multiple Company candidates discounts apply.



Overview: Following sold out Courses in Darmstadt, Germany and Kuala Lumpur, Malaysia, SIETRONICS is pleased to announce the upcoming Siroquant Training Program in Sydney. This program is designed and conducted to meet the requirements of users at every level. Participants will be provided with a temporary license for Siroquant and SiPhase software for the duration of the training course. Participants are also entitled to ongoing email support for six months following the course completion.

For further information and enrolment, please contact:

siroquant@sietronics.com.au

www.sietronics.com.au

Sietronics – (02)-6251 6611

4. 13th European Powder Diffraction Conference (EPDIC-12)
Grenoble, France
28-31 October 2012

<http://epdic13.grenoble.cnrs.fr/>

5. Accuracy in Powder Diffraction Meeting (APD-IV)
NIST - Gaithersburg, MD, USA
April 22, 2013 - April 25, 2013

http://www.nist.gov/mml/apdiv_conference_2013.cfm

6. Australian Synchrotron proposal round
For beamtime May-August 2012
Closes 15 February 2012

<http://www.synchrotron.org.au/index.php/features/applyin-g-for-beamtime/proposal-deadlines>

7. OPAL, ANSTO, proposal round
For beamtime July-December 2012
Closes 15 March 2012

http://www.ansto.gov.au/research/bragg_institute/users/c_all_for_proposals

AXAA Website and Contacts

<http://www.axaa.org>

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**Please email contributions to the first 2012 e-newsletter edition to Nathan Webster by 16th March 2012.
Any comments and feedback about the newsletter are welcome.**

Sietronics Pty Ltd

ANALYTICAL SPECIALISTS

Portable XRD/XRF aboard Mars Science Lab

After receiving the Editors' Choice GRAND GOLD Prize as the most outstanding new product at the 2009 Pittcon Conference and a prestigious R&D 100 Award for their field portable XRD/XRF analyser, world leading designers of field portable analytical instruments, INXITU was chosen to be part of NASA's Mars Science Laboratory (MSL), which left Cape Canaveral on November 26, 2011. Samples drilled from the rock on the surface of Mars by the Exploration Rover will be identified and quantified by the *Terra* technology.



The success of the *Terra*, is due largely to INXITU's patented powder handling methods which dramatically improve the quality of XRD data while reducing the requirements for sample preparation. Requiring only 15mg of sample with grain sizes up to 400 μm , *Terra* convects the sample with its integrated sample vibration chamber and presents all different orientations of the crystal structure to the instrument optics. This results in superb X-ray diffraction pattern, virtually free of problematic preferred orientation effects found using more classic preparation methods.

With no mechanical goniometers or moving parts a specifically developed direct excitation charge coupled device (CCD) "camera", *Terra* collects X-ray photon data for both XRD and XRF simultaneously. This is the result of the integrated camera's ability to detect both photon position and photon energy at the same time.

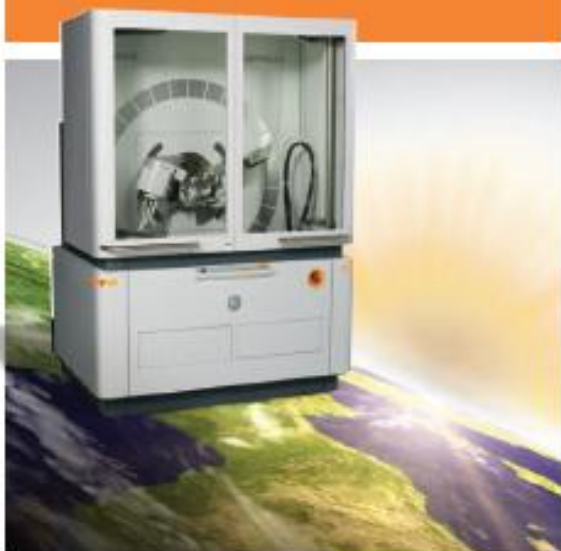
Distributed globally by Olympus, the completely contained, battery operated *Terra* and Laboratory bench top model *BTX-II*, has been embraced by exploration geologists, mineral and materials analysts, pharmaceutical manufacturers, forensic scientists and teaching institutions.

Sietronics is proud to be part of this next generation technology.

Greg Brighton
Sietronics Pty Ltd

EMPYREAN

The world of X-ray diffraction is no longer flat



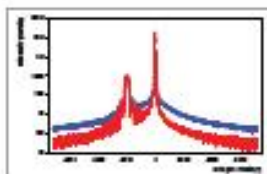
The only XRD platform that does it all

- Powders
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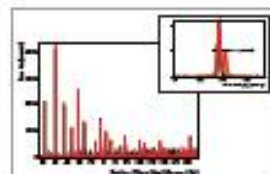
Imagine your sample in 3D: a reality with CT Imaging

Computed Tomography gives an inside view in solid objects by acquiring multiple 2D images while rotating the object. Thanks to the unrivalled dynamic range and the small pixel size of the PIXcel^{2D} detector, we can offer CT as a complementary technique on the Empyrean diffractometer.

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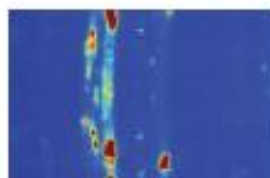


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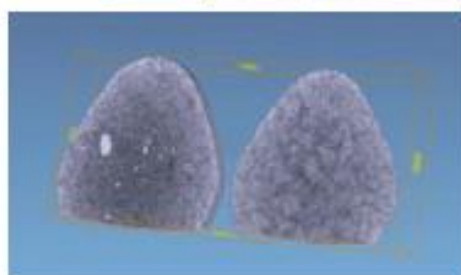


1D

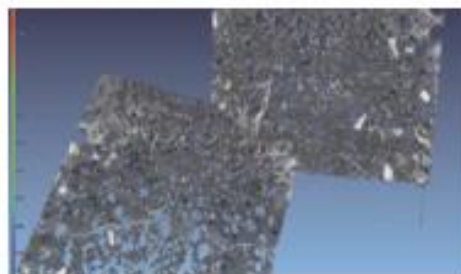
2D



3D



Inside view in counterfeit and original tablet



Porosity differences in normal and reinforced concrete

For more information, please contact:

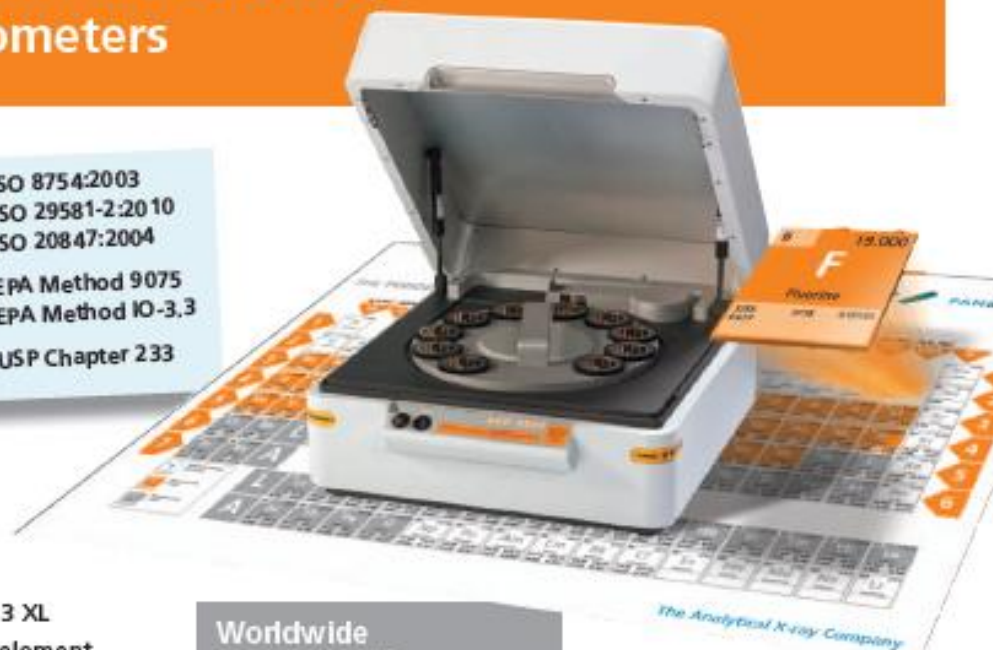
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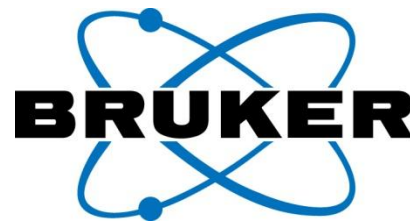
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The Analytical X-ray Company



D2 PHASER Desktop XRD: SAXS-Analysis of the Mesoscopic Catalyst SBA-15



The D2 PHASER is a portable desktop XRD instrument for research and quality control. It is easy to operate and independent of external media such as cooling circuits. Thanks to the LYNXEYE detector it is the fastest desktop XRD system on the market. This report demonstrates its use for fast and reliable SAXS measurements of material exhibiting large d-spacings up to about 10 nm.

Catalysts are indispensable to modern-day society because of their prominent role in petroleum refining, bulk and fine chemical processing and reduction of environmental pollution. High surface-to-volume ratios are often important for these particles since catalytic processes take place at the surface. Therefore, supports such as silica and gamma-alumina are generally used to obtain small and thermally stable particles.

Fundamental studies are often hampered by the heterogeneity of conventional supports that make it difficult to disentangle the effects of the individual preparation steps on the final dispersion. To overcome these problems meso-porous silica SBA-15 (Santa Barbara no. 15) can be used as a support system. The structure essentially consists of an amorphous silica framework forming a two-dimensional hexagonal primitive assembly of straight channels or pores. The structure itself is flexible and may adopt different pore diameters. The pore size can be measured from TEM pictures as shown below.

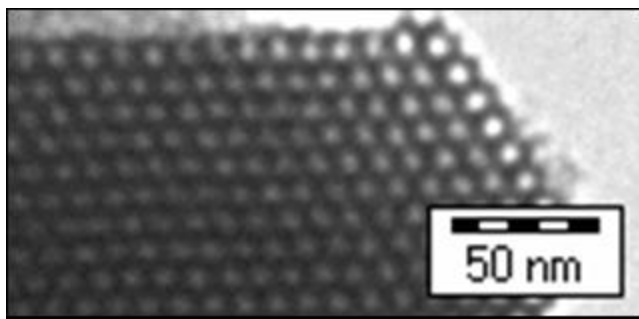


Figure 1: TEM Picture of zeolite SBA-15. This material exhibits pores up to 10 nm diameter which is larger than for other meso-porous materials such as MCM-41.

X-rays play an important role in the characterization of these materials. The x-ray diffraction pattern below shows small-angle powder data of CuO loaded SBA-15. The three peaks labeled in shown below are caused by the regular array of the pores. They are a measure of the average pore distance. A major advantage of powder X-ray scattering over other methods is the negligible effort needed to prepare the sample. The measurement is very fast and takes a few minutes only. Moreover, XRD has a superior sensitivity to dimensional changes of material on the below 10 nm length scale.

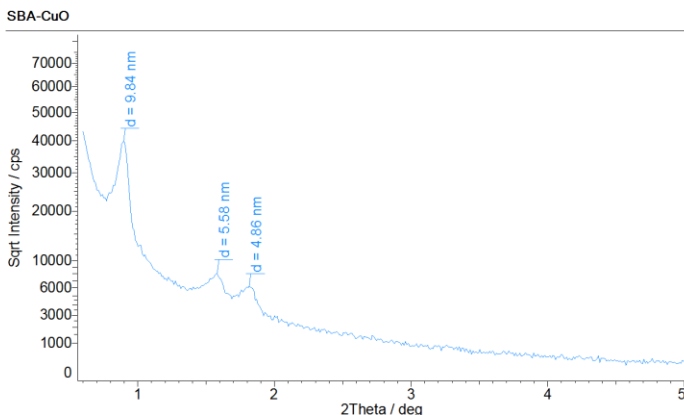


Figure 2: Small-angle X-ray powder pattern of CuO loaded SBA-15. The three signals correspond to the 100, 110 and 200 reflections on a hexagonal primitive lattice. They are used to estimate the average pore distance of this material, here about 11.25 nm.

The example presented in the pattern above indicates that impregnation of SBA-15 with CuO during the preparation of the catalyst does not affect the long-range order of the pores in the support material. Sample and graphics are courtesy of the Inorganic Chemistry and Catalysis group, Department of Chemistry, Utrecht University, The Netherlands.

For more information on the D2 Phaser, please contact Bruker Biosciences on sales@bruker.net.au or Ph 1300-BRUKER