AXAA e-newsletter

Issue 3, 2015 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Friends,

The National Council is excited to announce that the AXAA 2017 National Schools, Conference and Exhibition will be held from 5-9th February 2017 at the Pullman Albert Park in Melbourne! The conference website is now live; details about important dates and sponsorship opportunities will be added in the New Year. Details will also become available on the AXAA-2017 Facebook page (https://www.facebook.com/AXAA2017/) and on Twitter (@axaa_org#axaa2017). For now, further information can be obtained from the Conference Secretariat (please see link on the conference website). The Conference Committee will meet in February 2016 to begin the design of an exciting and stimulating scientific, educational and social programme.

For the second year in a row AXAA was a Bronze Sponsor for the Australian Synchrotron User Meeting, which this year was held in conjunction the Asia-Oceania Forum for Synchrotron Radiation Research (AOFSRR 2015). In continuing efforts to extend our outreach, we are poster session sponsor for the "Wagga 2016" Condensed Matter and Materials (CMM) meeting. The popular annual meeting is an excellent opportunity to further increase the visibility of AXAA, particularly to students.

September, October and December saw AXAA Student Seminars events held in NSW, VIC and WA, respectively. The response that we received for each event was fantastic, with the quality of the presentations very high, and cash prizes awarded to the three best presentations in each state. Thanks to PANalytical, Bruker and AXT for their sponsorship of the NSW, VIC and WA events, respectively. Next year, as we gear up for AXAA-2017, we also plan to hold events in SA and QLD. These events are an excellent opportunity for students to present their work in a friendly, collegial atmosphere and meet others in the X-ray and neutron scattering community.

Finally, I wish everyone an enjoyable festive season and a happy and healthy 2016.

Nathan Webster
AXAA President

Matters for Scatterers: Characterisation of the Tent Hill Sn-smelting site, NSW

Leveraging economic potential for remediation

Australia has more than 50,000 abandoned mine or processing sites, the majority of which require remediation as they contain highly elevated concentrations of heavy metals and metalloids. In particular, heavy metal(loid)s are commonly toxic and are associated with a wide range of health problems, including cancers, organ failure or reduced development in most organisms. As a result, understanding the mineralogy and geochemistry of these sites is vital for effective remediation and planning.

Profile through the mine waste at the Tent Hill site

This study characterised the geochemistry and mineralogy of an historic Sn-smelting site in Tent Hill, NSW that contains a significant amount of heavy metal contaminated tailings. Tailings samples were analysed using portable X-ray fluorescence and qualitative X-ray diffraction to determine the geochemistry and mineralogy of the waste material at the site. This analysis identified high concentrations of As (0.23-9.75 wt.%), Pb (0.01-1.38 wt.%), Sn (0.03-4.25 wt.%) and Cu (0.06-0.51 wt.%), with As the primary environmental concern at Tent Hill. This As is predominantly hosted by scorodite ($FeAsO_4 \cdot 2H_2O$), although pharmacosiderite [$KFe_4(AsO_4)_3(OH)_4 \cdot 6H_2O$] and jarosite [$KFe^{3+}{}_3(OH)_6(SO_4)_2$] are also present.

These new data indicate that the dominant metal(loid)-bearing phases are currently thermodynamically stable within the tailings, although As mobilisation may be induced in several ways, including (1) dissolution of the As-bearing minerals, (2) desorption of As under alkaline, oxidising conditions and (3) desorption of As from Fe-hydroxides either under acidic or reducing conditions. This indicates that As will be released from the tailings site should the current physical and geochemical conditions be altered.

Proximity of tailings to waterways has implications for AMD run off and heavy metals contamination of local rivers.

Future uses of the Tent Hill site will need to take changing water levels into consideration, as this could impact on the surrounding environment. Phytoremediation with native metal(loid) resistant or hyper-accumulative plants such as *Eucalyptus michaeliana* may be a potential mitigation option but greater understanding of the site-specific soil/plant interactions would determine whether the option is economically and environmentally efficient. Finally, the data acquired during this study illustrate the potential of generating wealth from waste from both current and former mine sites, as the economic value of the metals within the tailings material is ~AUD$919,000, even at currently low metal prices. The geochemical and mineralogical data obtained during this study should be considered the first step in the potential reprocessing of this tailings material, an environmentally and cost effective remediation strategy that may have applications for economic gain or for the funding remediation of mine sites both across Australia and elsewhere in the world.

Harriet Wilson
Monash University

Passion for the environment saw me completing my undergraduate and honours degrees at Monash University, focusing in Geoscience and Ecology. A strong interest in environmental geochemistry led me to undertake Honours in Earth, Atmosphere & Environment (EAE), where I worked with Sasha Wilson and Simon Jowitt to complete my degree in November, 2015. Through the course of my degrees I have gained experience in analytical techniques such as Raman and atomic mass spectroscopy, XRD, XRF and population modelling.

AXAA Student Seminar Days

NSW – "Scattering Matters"

The AXAA Student Seminars kicked off once more in September this year, with an afternoon of presentations titled "Scattering Matters" hosted by Western Sydney University. Presentations were given by 10 honours and PhD students from the University of Sydney, UNSW and Western Sydney, on topics ranging from chemistry to mineralogy to magnetism. The standard of all the talks was outstanding, stimulating enthusiastic discussions during the breaks. First and second prizes, generously sponsored by PANalytical, were awarded for the best honours and PhD student presentations. James Christian (UNSW, ANSTO) and Tim Murphy (Western Sydney) took out first and second prizes in the PhD student category, for their talks on next generation batteries and bismuth mineralogy, respectively. Cheryl Wong (USyd) and Sean Injac (USyd) were awarded first and second prizes in the honours student category for their talks on magnetism in P2-type layered oxides and ordered double perovskites, respectively. Special thanks to PANalytical for their support, and to Ric Wuhrer for helping to coordinate the event.

(Top left) James Christian, UNSW, (Top right) Tim Murphy, Western Sydney, (Bottom left) Cheryl Wong, USyd, and (Bottom right) Sean Injac, USyd, receiving their awards from AXAA Vice-President Vanessa Peterson.

VIC – "Something to Bragg About"

The AXAA Student Seminar series continued in Victoria in October, with an event titled "Something to Bragg About" held at the CSIRO laboratories in Clayton. The quality of the student presentations was amazing, attracting an audience from various institutions across Melbourne. 11 honours and PhD students from Monash, Deakin and RMIT presented their work on diverse topics ranging from metallurgy to the structure of spun yarn, using a wide range of X-ray techniques including XRD, XRF, SAXS and tomography. Monash University made a clean sweep of the event, with 1st, 2nd and 3rd prizes going to Emily Hebbard, Harriet Wilson and Anita D'Angelo, respectively, for their talks on arsenic-rich processing residues at the Ottery mine site, geochemical and mineralogical characterisation of Tent Hill Sn-smelting site, and vacancy generation and oxygen uptake in Cu doped Pr-CeO_2 mixed oxides. Many thanks go to Bruker for sponsoring the event, which concluded with pizza and refreshments.

(L-R) Chris Kelaart (Bruker), Nathan Webster (AXAA President), first prize recipient Emily Hebbard (Monash Uni), second prize Harriet Wilson (Monash Uni), third prize Anita D'Angelo (Monash Uni).

WA – "Bright Scatterers"

After a hiatus of several years, 2015 saw the return of the AXAA WA Student Seminars, held at the CSIRO Waterford laboratories on 10 December. The event, "Bright Scatterers", was sponsored by AXT and was well attended with an audience of 20 AXAA members and guests. Five students (all PhD) presentations were given by students from Curtin and Murdoch Universities. The student talks were preceded by an invited presentation, "Case studies of the mechanism of extractive metallurgy by powder X-ray diffraction" delivered by Dr Fang Xia, Senior Lecturer - Chemical and Metallurgical Engineering at Murdoch University. Ehsan Mohammadpour (Murdoch University), took out first for his presentation "High temperature structural properties of CrAlTiN coatings from in-situ synchrotron radiation X-ray diffraction", with Hani Albetran (Curtin University, "Effect of ion implantation and atmospheres on the crystallization kinetics, phase transformation, and band gap of electrospun titania nanofibers using in-situ high-temperature synchrotron radiation diffraction") and Pavam Jayadian (Curtin University, "Hydrogen storage properties of nanoconfined (complex) metal hydrides") taking and second and third prizes, respectively. The presentations were followed by pizza and drinks, which rounded out an enjoyable and stimulating afternoon. Thanks to AXT for helping to make the event a success, and also to Jian Li and Peter Austin from CSIRO for their organisational efforts behind the scenes.

(L-R) Nathan Webster (AXAA President), Second Prize recipient Hani Albetran, Pavam Jayadian (Third Prize), Ehsan Mohammadpour (First Prize), Arnaud Griffon (Curtin University), Enrico Ianni (Curtin University), and Fred Hoetmer (AXT).

AXAA Life Membership: Ian Madsen

Ian Madsen is a very well-known and respected member of AXAA, and has contributed a great deal to the both the Australian and international X-ray communities over many years. In recognition of his long association and outstanding contributions to the AXAA community, Ian was awarded Life Membership to AXAA by the National Council in April this year.

Ian began his career in X-ray analysis in March 1969 as technical assistant with the CSIRO Division of Soils in Adelaide, conducting XRF analyses of plant and soil samples under the supervision of Keith Norrish. After deciding that X-ray diffraction would be his future, Ian transferred to the Division of Mineral Chemistry in Port Melbourne in 1977. Since then, Ian has gone on to develop an international reputation as a leader in X-ray diffraction science, and is now well recognised as the preeminent expert in X-ray diffraction-based quantitative phase analysis (QPA). Ian has authored numerous research papers and book chapters on the subject, and has been the technical chair of the most recent Accuracy in Powder Diffraction conference, which is considered to be the pinnacle of powder diffraction conferences internationally. In addition to QPA, Ian has been a driving force behind the development of *in situ* diffraction capabilities in Australia. He has had a very large impact on Australia's two major radiation facilities, the Australian Synchrotron and the Bragg Institute at ANSTO, through his involvement in the design stages and his guidance during the early operation of the powder diffraction instruments at both facilities. Ian retired from CSIRO in July this year, after more than 46 years, but has returned as an honorary fellow where he will no-doubt continue to contribute to the development of X-ray diffraction techniques and methods.

Ian attended his first AXAA National Conference in 1971 (or was it 72?), and has been to every meeting held since. He became a paid-up member of the AXAA Victorian branch in 1978 (or 79?) and member of the National Council from the late 80's. Ian was the President of the National Council in the late 80's and early 90's, after which he stood aside to take a more active role in international activities such as organising and running the IUCr CPD Round Robin on QPA in the late 90's and early 2000's. However, Ian has continued to contribute to the AXAA community, as a committee member for the 2005, 2008 and 2011 AXAA conferences, and as a regular lecturer at the workshops and schools. Ian has always been very generous with his time when it comes to

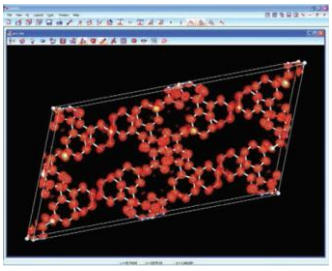
teaching students and postdocs about X-ray diffraction and Rietveld analysis, and is always happy to talk diffraction.

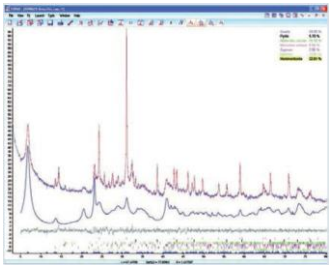
In 2008 Ian was the inaugural winner of the AXAA Bob Cheary Award for “Excellence in X-ray Analysis”, in recognition of Ian’s standing and reputation within the X-ray community. We are now delighted to award Ian Life Membership to AXAA, for his contribution AXAA over many years. Ian was presented with his award during the recent Victorian Student Seminar day. Congratulations Ian and thank you!

Left) Ian Madsen receiving his AXAA Life Membership award during the recent Victorian Student Seminars. Right) Ian’s trophy.

TOPAS Beginners School

The TOPAS Beginners Course was held in late November at Monash University, and it was attended by over 20 participants from both academia and industry over the course of 2 days. The course was open to all beginners to TOPAS who already had a good knowledge of XRD techniques and focused mainly on the use of TOPAS. Presented by former CSIRO scientist Ian Madsen the course covered such topics as peak profile fitting, whole pattern analysis and quantitative phase analysis.





Coating research engineer, Dr Andrew Ang from Swinburne University said, “Coached by Ian Madsen, we could not ask for a better introduction to TOPAS. A right balance of theory and examples given throughout the course. I personally look forward to using the methods and techniques that I learnt for quantitative analysis of thermal spray coatings.”

In 2016, Bruker will be running the TOPAS Intermediate Course for users who attended the Beginners course, or who already have a basic knowledge of TOPAS and would like to further their skills. The next Beginners’ course is planned for 2017.

We would like to thank Ian for his time and effort in preparing and delivering an outstanding course and look forward to future courses.

If you are interested in attending future TOPAS training courses please contact: martin.duriska@bruker.com

XRF Quick-Reference Posters

Professor James Willis started his career in XRF in 1962, and has thoroughly enjoyed running training courses in XRF since 1974. He has been fortunate to have had four great teachers: Ron Jenkins, Gerry Lachance, Bruno Vrebos, and the XRF Listserver, and would like to give back to the X-ray analysis community in the form of three quick-reference posters. These posters have been carefully crafted over the years and are valuable tools for XRF analysts. The details of the posters are:

Poster 1: The WDXRF spectrometer, illustrated with appropriate wavelength scans and pulse height distributions, including: (1) The components of a WDXRF spectrometer; (2) End-window Rh target X-ray tube spectra showing the effects on the tube spectrum, and thus excitation efficiency, of changing the kV from 60 to 40 to 25kV; (3) Primary beam filters: none, "thick" Al, "thick" brass; (4) Primary collimators: fine, medium, coarse; (5) Analysing crystals: LiF(200), LiF(220), LiF(420); (6) Detectors: Scintillation detector, Ar-CH4 gas flow detector, tandem Xe-SGD + Ar-CH4 gas flow detector; (7) Pulse height distributions for Fe Kalpha for a Scintillation detector, Ar-CH4 gas flow detector, and Xe-sealed gas detector; (8) Pulse height distributions using a Ar-CH4 gas flow detector for P Kalpha with a Ge analysing crystal, for Al Kalpha with a PE crystal, and for Mg Kalpha (some Ca present in the sample) with a 5nm LSM (Si+W) crystal.

Poster 2: Some factors affecting XRF Sensitivity (Kalpha lines Sn to Na): Excitation efficiency of a Rh tube; Mass attenuation coefficients (MACs); Fluorescence yields; Scintillation and Flowcounter detector efficiency; Measured sensitivity for the Kalpha lines of elements Sn to Na.

Poster 3: Theoretical intensities for Sn, Cu, Ti, K and S K lines excited by an end-window Rh tube operating at 60, 50, 40, 30 and 25 kV and 4kW. It gives the equations for calculating theoretical intensities and demonstrates with X-ray spectra how they are used to calculate theoretical intensities; illustrates the excitation efficiency of different parts of the tube spectrum and how to maximise that efficiency, the change in excited intensities from Sn to S and for different kV settings, and the increase in Ka/Kb intensity ratio as the atomic number decreases.

If anyone would like free copies of any of the posters you can preview them on the "Contact us" page at website xrfguidelines.co.za and then email Prof. Willis for a link to high resolution PDF files suitable for printing on A1 size (posters 1 and 3) or A2 size (poster 2). These posters look good when laminated.

Prof Emeritus James P Willis
James Willis Consultants

UNSW Poster Day

The UNSW School of Chemistry held its annual Research Poster Day in September. This year the event saw over 60 posters from students and post-docs within the rapidly growing School and representing research stretching from catalysis and energy, to nanoscience and medicinal chemistry. Over 250 people participated in the event, from undergraduates to academic staff, discussing the latest research from the School and building enthusiasm for science.

At least two-thirds of the posters presented contained research where X-ray diffraction measurement instrumentation from Bruker within the School or the nearby UNSW Mark Wainwright Analytical Centre (MWAC) was used. A similar proportion of posters showed results based on NMR studies using the suite of Bruker NMR spectrometers at the UNSW, and in half of the posters researchers used a combination of X-ray diffraction and NMR instrumentation in their work!

Bruker has had a long-standing relationship with the School of Chemistry at UNSW, including being the principal sponsor the Annual School of Chemistry Research Poster Day. This year Dr Martin Duriska presented the PhD student poster prizes including the Bruker Energy Cluster Prize to Bryan Suryanto, supervised by A/Prof. Chuan Zhao and the Bruker Nanoscience prize to Manish Srimam, supervised by ARC Laureate Fellow Prof. Justin Gooding.

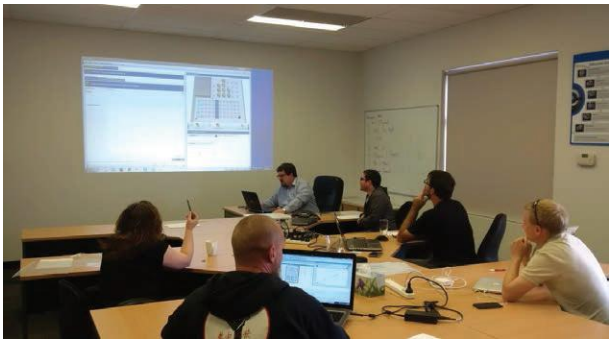
(L-R) Manish Srimam, Bryan Suryanto and Dr. Martin Duriska (Bruker)

Contributed by Palli Thordarson
University of New South Wales

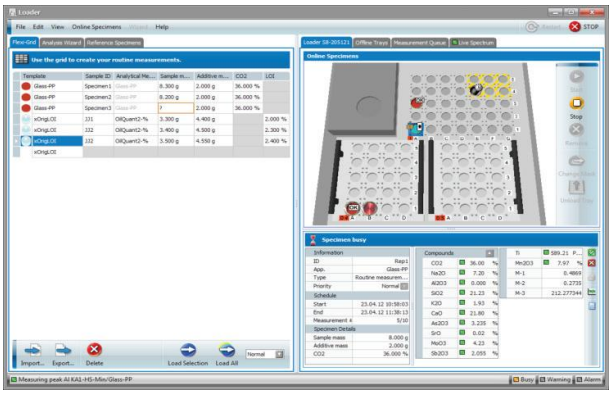
Bruker XRF Training School

Following on from our inaugural New Zealand XRF training course held earlier in the year, the Melbourne Bruker application laboratory ran another successful XRF training school for Bruker XRF users.

The attendees came from across Australia & New Zealand from diverse backgrounds in academia, mining and the cement industries. Our XRF application scientist, Elvy Grigolato, provided five days of in depth training on the SpectraPlus XRF software focused on utilising the new improved productivity tools of SpectraPlus version 3 and improving XRF calibrations for best analytical results.



Spectraplus Version 3 has replaced the loader and results reporting modules. With these come many features as Elvy Grigolato explained: “Sample entry allows touchscreen options for production environment or spreadsheet interface for labs manually entering or uploading large sample sets. With the support of offline trays, it is possible to pre-prepare samples or for sites which have regular test samples e.g. Well-01A.



Additionally, we support deferred entry, enter the sample name and method and enter other information (eg sample weight) later before evaluation.”

“We also have a New Scheduler – Run QC samples at specified frequency, if the QC fails the drift monitor is then run and the QC analysed. Runtime/QC charts are available with minimal effort within standard software.”

“Reporting has a fresh new look with improved speed and very flexible selection criteria. Do you want tabs with just results for one QC sample or all your cement samples, easily defined and used? If you need to analyse unknown materials, it is possible to select a sample from

the result monitor and view in the interactive evaluation program, to enter elements analysed by combustion systems (C, H, N, O) or define matrix e.g. Cellulose."

"Many users routinely analyse samples with calibrations created when their systems were installed. The course showed how you can mix standardless and calibrations together. Add a standardless element to their existing fusion program within 30 minutes or create a pressed powder standardless method which analyses volatile or low concentration trace elements but obtains the majors data from the sample measured against their fusion calibration." Additional training sessions covered service and diagnostics tools, and we had a guest presentation from Danny Verbeeten of XRF Scientific covering fusion sample preparation methodologies and the successful preparation of difficult sample types.

If you are interested in attending future Bruker training courses, please contact: neil.hughes@bruker.com

Upcoming Events

40th Annual Condensed Matter and Materials Meeting in Wagga Wagga

2-5 February 2016
Charles Sturt University, Wagga Wagga

The Australian Synchrotron is proud to host the 40th Annual Condensed Matter and Materials Meeting in Wagga Wagga. The meeting will be held across four days, on 2 - 5 February 2016, at the Wagga Wagga campus of Charles Sturt University.

Wagga 2016 brings together the condensed matter community across the country and New Zealand, and we are pleased to welcome speakers and delegates from each state and territory as well as New Zealand. Once again this meeting will showcase the best research from around the area, and update the community on the latest techniques and application developments.

For more information:
Email: wagga16@synchrotron.org.au
Website: https://events.synchrotron.org.au/event/19/

AXAA is a proud sponsor of this event

X-ray Materials Analysis Internet Courses – Wavelength Dispersive XRF and Powder XRD (Plus New Mentoring Program on Rietveld XRD analysis)

Mode of Instruction for XRF and XRD Courses

These internet-delivered courses provide XRF and XRD analysts, particularly those new to X-ray analysis, with on-site and/or at-home instruction on the underlying principles and principal analytical methods. Features of the courses -

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

The courses have a substantial cohort of international participants, as well as Australians, and are being used by companies as vehicles for in-house XRF and XRD training, and also for Rietveld phase composition analysis.

Courses Director: Dr Brian O'Connor

Internet XRF Course: Series 9, 2016

The *Internet XRF Course* comprises modules on - XRF Overview; X-ray Excitation of the Specimen; X-ray Dispersion and Detection; XRF Data Measurement; Data Analysis Basics; Methods of Quantitative Analysis; Absorption-Enhancement Corrections; Specimen Preparation; Major Component Analysis Using Fusion Buttons; Trace Element Analysis Using Powders; and Analysis of Sub-Milligram Environmental Samples.

Course fee: $2,900 including GST

Internet XRD Course: Series 4, 2016

The *Internet XRD Course* comprises modules on - XRD Overview; Essential XRD Fundamentals; XRD Measurement Strategies (I); XRD Measurement Strategies (II); Search/Match Identification Analysis (I); Search/Match Identification Analysis (II); Case Studies in Search/Match identification Analysis; Phase Composition Analysis Using Line Intensities; and Introduction to Advanced Methods (indexing, Rietveld phase analysis, structure solution, etc.)

Course fee: $2,900 including GST

Internet Rietveld XRD Analysis Personalised Mentoring Program (New Program)

The *Internet Rietveld XRD Analysis Personalised Mentoring Program* is designed to support people who need help in becoming proficient in Rietveld-analysing their materials for phase composition. The program is customised to meet the needs of the participant, and will include learning how to efficiently Rietveld-analyse their own XRD patterns and will also address requirements for analysing large suites of XRD patterns. The program is structured according to the background knowledge of the mentee, and also the Rietveld software used in the person's laboratory.

Course fee: On application, as the fee will depend on the participant's background knowledge.

Further Information and Enrolment Procedure:

brian_oconnor@iprimus.com.au (Tel 08 9291 7067)

AXAA Website and Contacts

Please visit our website, www.axaa.org, for further information, or follow us on Facebook or Twitter @axaa_org.

NATIONAL COUNCIL PRESIDENT:
Nathan Webster
CSIRO Minerals Resources Flagship, Box 10, Clayton South, VIC 3169
Telephone: (03) 9545 8635
e-mail: nathan.webster@csiro.au

NATIONAL COUNCIL VICE PRESIDENT:
Vanessa Peterson
Bragg Institute, ANSTO, Locked Bag 2001, Kirrawee DC NSW 2232
Telephone: (02) 9717 9401
e-mail: vanessa.peterson@ansto.gov.au

NATIONAL COUNCIL SECRETARY
Natasha Wright
CSIRO Manufacturing Flagship, Normanby Rd, Clayton, VIC 3168
Telephone: (03) 9545 2041
e-mail: natasha.wright@csiro.au

NATIONAL COUNCIL TREASURER
Gordon Thorogood
Institute of Materials and Engineering, ANSTO, Locked Bag 2001, Kirrawee DC NSW 2232
Telephone: (02) 9717 3183
e-mail: gordon.thorogood@ansto.gov.au

NATIONAL COUNCIL NEWSLETTER EDITOR
Mark Styles
CSIRO Manufacturing Flagship, Private Bag 10, Clayton South, VIC 3169
Telephone: (03) 9545 8179
e-mail: mark.styles@csiro.au

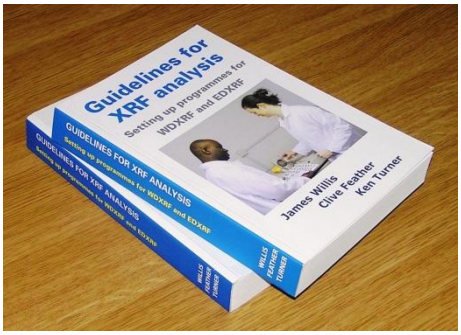
Please email contributions for Issue 1 of the 2016 AXAA Newsletter to Mark Styles by Friday the 25th of March. Any comments or feedback about the Newsletter are welcome.

AXAA Membership

All registered participants of the AXAA-2014 conference are automatically granted AXAA membership for 3 years. Alternatively, new memberships can be obtained free of charge, by making an application to the National Council. Candidates should provide their CV and a short statement about how they intend to contribute to the organisation. Please send these to the National Council Secretary Natasha Wright (see AXAA contacts) if you would like to apply.

Company News and Advertising:

New Text for XRF Analysis



Guidelines for XRF Analysis contains everything you've ever wanted to know about setting up successful XRF analytical programmes. This book is loaded with detailed analytical guidelines for geological materials, commodities and industrial materials that are suited to analysis by XRF.

It is a one stop XRF reference manual, and no XRF laboratory should be without it. Sample preparation describes well-tried and tested methods, and there is sufficient theory to enable the reader to gain maximum advantage from the book. Trace elements, grouped by spectral region, are carefully evaluated, and the authors, with a combined XRF experience of over 130 man years, share with you their accumulated knowledge, "tricks-of-the-trade", and information on:

- Optimum settings for WDXRF and EDXRF instrumentation
- The most suitable analyte spectral lines
- The best background positions to measure
- Identification and correction of line overlap, and
- Choice of procedures for matrix correction

Determine with confidence fluorine to uranium, atomic number 9 to 92, in a wide range of materials. If you are working on any of the following commodities or materials, you need this book. Commodities and materials chapters are self-contained and have all the information needed to analyse:

- Silicate rocks
- Exploration samples
- Alloys of precious metals
- Activated carbon and catalysts
- Ferrochrome & Ferromanganese
- Lateritic nickel ores
- Iron ores and slags
- Aluminium ores and alumina
- Mineral sands & heavy minerals
- Refractories and ceramics
- Plastics and polymers
- Fuels, oils and wear metals
- Metal alloys
- Coal and coke
- Environmental materials
- Sulphide base metal ores
- Uranium ores and "Yellow cake"
- Cements and carbonates

Published in 2014 by James Willis Consultants cc, Cape Town, South Africa
ISBN 978-0-620-62961-4

Guidelines for XRF Analysis

By James Willis, Clive Feather, Ken Turner

240mm (H) x 168mm (W) x 30mm (T)
544 Pages, 253 Figures in full colour,138 Tables
Website: www.xrfguidelines.co.za

Available directly from our
Australasian agents or the authors
XRF Scientific Ltd
98 Guthrie St
Osborne Park
WA 6017, Australia
steve.prossor@xrfscientific.com | www.xrfscientific.com |

Please e-mail your enquiries to
XRF Scientific directly, or James Willis: jwcc@iafrica.com

Bruker Australia / NZ News *December 2015*

In this update:

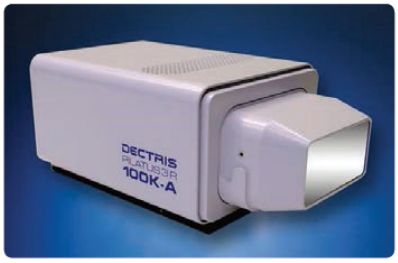
- D8 Discover XRD with PILATUS3 2D Detector
- New Generation of Single Crystal Instruments





D8 Discover XRD with Pilatus3 2D Detector

At the Japan Analytical & Scientific Instrument Show 2015 (JASIS), Bruker AXS announced the PILATUS3 R 100K-A 2D X-ray detector for its D8 DISCOVER diffraction solutions.



The PILATUS3 R is a new Hybrid Photon Counting (HPC) pixel detector developed by the Swiss company DECTRIS, the technology leader for HPC detectors. In close collaboration, Bruker AXS and DECTRIS have seamlessly integrated this powerful new detector into the D8 DISCOVER to enable full usability for various X-ray diffraction applications.

The PILATUS3 R 100K-A significantly extends the capabilities of the D8 DISCOVER for various types of diffraction experiments. The HPC pixel detector offers a unique combination of superb performance features: no dark current and read out noise, an excellent point spread function, and highest available frame rates, count rates and dynamical range. These unmatched capabilities allow the HPC detector to produce excellent diffraction data in a broad range of applications, especially in those experiments where 2theta and gamma coverage is essential, including micro diffraction, texture, residual stress, grain size, GISAXS, micro-HRXRD and kinetic studies.

The addition of the HPC pixel detector completes Bruker AXS' cutting edge X-Ray detector portfolio which features the 1D LYNXEYE™ silicon strip detectors with unrivalled energy resolution, the high speed, shutterless PHOTON 100 CMOS APS detector for single crystal diffraction, and the large active area, noise-free VÅNTEC MikroGap™ detector.

For further details, contact Martin Duriska on 0487-800-317 or martin.duriska@bruker.com

Bruker Pty Ltd ABN: 86 002 561 328 | Tel: 1300-BRUKER (1300-278-537) | Email: sales.anz@bruker.com | Web: www.bruker.com

Melbourne Office:
1/28A Albert St Preston VIC 3072
PO Box 8432, Northland Centre VIC 3072
Ph: (03) 9474-7000
Fax: (03) 9474-7070

Sydney Office:
Unit 7, 163-167 McEvoy St, Alexandria NSW 2015
PO Box 202, Alexandria NSW 1435
Ph: (02) 9550-6422
Fax: (02) 9550-3687

Wellington Office:
Unit 6, 5 Bouverie St Lower Hutt 5012, NZ
PO Box 39-202, Lower Hutt 5045, NZ
Ph: +64 (4) 589-3355
NZ Company #: 4042738

New Generation of Single Crystal Instruments

At the Asian Crystallographic Association (AsCA) meeting in Kolkata, India, Bruker AXS announced its new, next-generation instrumentation platform for crystallography, the D8 Gen 2 (www.bruker.com/d8gen2).

The D8 Gen 2 will become available in the D8 QUEST and D8 VENTURE and features the ground-breaking, new PHOTON II™ detector (www.bruker.com/photon2). For the first time, the PHOTON II™ harnesses advanced Charge Integrating Pixel Array Detector (CPAD) technology, originally developed for X-ray Free Electron Lasers (XFELs). The PHOTON II features the largest monolithic active area of 10 × 14 cm2, the highest detective quantum efficiency and the highest frame rate of any home laboratory detector. By design, the PHOTON II™ also completely eliminates the charge sharing noise and parallax issues that plague the current generation of Pixel Array Detectors (PADs).

The D8 Gen 2 also features the latest version of the highly successful Incoatec Microfocus Source, the IμS 3.0 (www.bruker.com/ims3).The IμS 3.0 is the first ever microfocus source specifically designed and optimized for X-ray diffraction. Previous microfocus sources feature electron optics and anode geometries that are designed for non-scientific applications such as non-destructive testing and are thus not fully optimized for X-ray diffraction. By completely redesigning the X-ray source, the IμS 3.0 increases X-ray intensity by 70% compared to conventional microfocus sources while retaining the legendary reliability of the IμS family, backed by a full three-year warranty.

The advanced technology is complemented by the completely new APEX3 (www.bruker.com/apex3) software package. It greatly enhances the crystallographers' productivity by integrating a new, highly intuitive user interface with advanced new analysis engines for the investigation of twinned and intergrown samples and powder data. It also features new, best-ever structure solution, and refinement routines.
Dr. Jérôme Basquin, from the Max-Planck Institute of Biochemistry in Martinsried, Germany, notes: "The first results from the D8 VENTURE Gen 2 have been indeed impressive. The speed of the PHOTON II™ is ideal for screening and the detector's single photon sensitivity helps us to collect the weakest reflections. We have obtained a number of high-quality data sets with resolutions comparable to synchrotron data, notably on very challenging problems."

Roger Durst, PhD, CTO and General Manager of Crystallography for Bruker AXS, notes that "the D8 Gen 2 is the culmination of over 6 years of intensive development and epitomizes Bruker's ongoing ambition to put the most advanced, powerful and reliable tools into the hands of our customers. We are especially excited and proud to announce our new instrument at the Asian Crystallographic Association meeting. This underscores the ever-growing global importance of science in Asia and also the commitment of Bruker to support fully the global scientific community globally."

For further details, contact Martin Duriska on 0487-800-317 or martin.duriska@bruker.com

Charting into strategic waters for PANalytical Asia Pacific

New leadership for Asia Pacific
After many years of being at the helm, Anant Bhide is retiring after close to 20 years of service with the PANalytical team, as Regional Manager for Asia Pacific. The company bids him farewell and wishes him all the best. Taking over from 1st January 2016 is Gjalt Kuiperes. He is no stranger to the region, having been instrumental in starting the Asia Pacific office in 1998.

Commitment to driving growth
In other news, PANalytical's regional HQ office will make a strategic move from Singapore to Shanghai from the start of 2016. "As such a strong part of the world's economy, with a diverse spread of industries and research, China continues to be a key focus for PANalytical. Moving the regional hub office to China, illustrates our commitment to driving growth in this market" says Gjalt.

Newest XRF spectrometer recognised by R & D 2015
PANalytical is proud to share that our newest XRF floor spectrometer, Zetium, has been shortlisted as the top 100 finalists for R & D 2015. This new innovation combines not just ED and WDXRF as well as Small Spot Mapping, offering a range of benefits not seen when operating each of them independently. Zetium also comes in various industry editions including minerals, petrols and many more. With the Zetium Minerals, enjoy other benefits like a reduction in analysis time of up to 50% plus analyse a wide elemental range from Beryllium (Be) to Americium (Am) at concentrations from ppm to 100 wt%. The system is delivered with a new, faster sample changer, the latest version of our renowned SuperQ software (including our new Virtual Analyst) and your choice of application solutions like the analysis of oxides in geological samples.

Interested to find out more about the Zetium? Read more at panalytical.com/The-Minerals-edition-of-Zetium

"Having a regional hub office in China illustrates our commitment to driving growth in this market"

Gjalt Kuiperes, PANalytical Asia Pacific's new Regional Manager on the move of the office to Shanghai

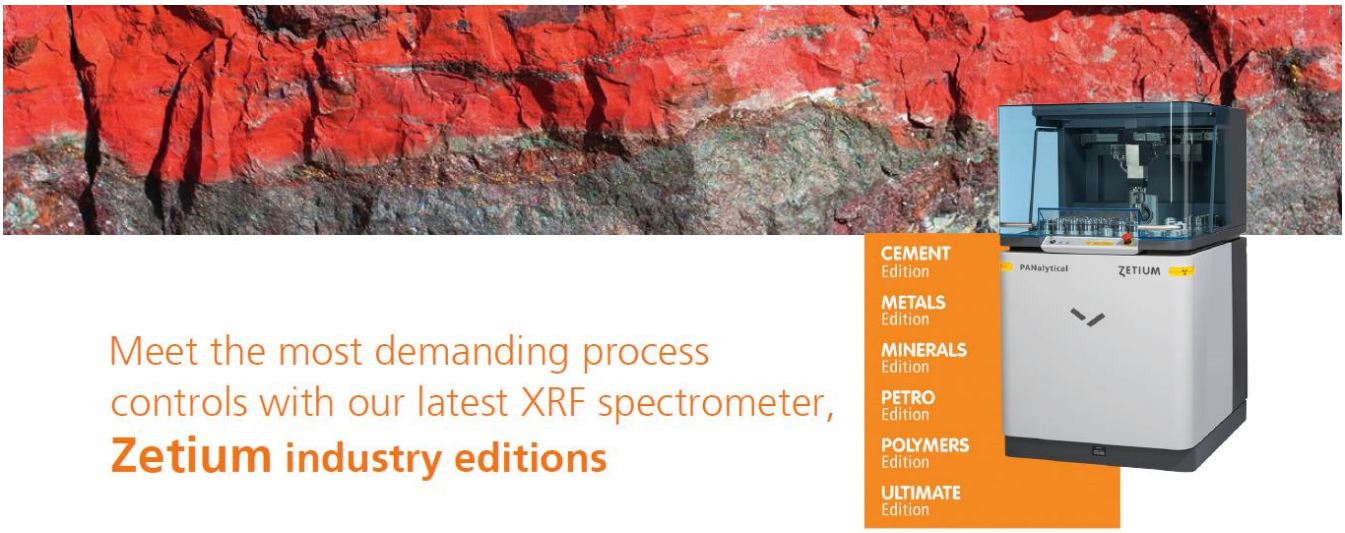
Hot details about Zetium XRF for Minerals

1. Combines ED, WDXRF & Small Spot Mapping!
2. Reduce analysis time by up to 50%
3. Analyse elements from Be to Am at concentrations from ppm to 100wt%
4. Enjoy a faster sample changer, latest software

Christos Tsouris (above), Product Manager from our HQ in The Netherlands, shared about the Zetium at Perth's Iron Ore Conference 2015. He was instrumental in designing the Zetium XRF.

PANalytical Australia
Unit 24/31 Governor Macquarie Drive
Chipping Norton, NSW 2170, Sydney
T: +61 2 8700 2700
E: info.australia@panalytical.com
W: www.panalytical.com

PANalytical
get insight



Meet the most demanding process controls with our latest XRF spectrometer, **Zetium industry editions**

Read about the Zetium XRF, customised for mining & minerals. We analyse iron ores, prepared as fused beads, using PANalytical's SumXcore technology

In large-volume environments, like those in the mining industry, any time savings that can be made, without a loss in accuracy and precision, results in higher sample throughput and faster turnaround times for fast-moving commodities.

Integration of the revolutionary ED core into the Zetium spectrometer sees two technologies, namely WD- and EDXRF, converging to make a unique and powerful analytical heart that we call **SumXcore**, which delivers unique benefits for mining applications.

In this example, 19 iron ore certified reference materials (CRMs) were used to set up applications using the WD core (conventional sequential WDXRF) and the SumXcore to demonstrate the achievable time savings with SumXcore technology.

WD core + ED core = SumXcore

- **Reduce analysis times by up to 50 %**
- **Identify and flag unexpected elements**
- **Fast sample screening**
- **Spectrum archiving**

Iron ores as fused beads	WD core	SumXcore
Oxides measured with WD core	18	11
Oxides measured with ED core	0	7
Measurement time (s)	216	136
Overhead time (s)	122.5	80
Total analysis time (s)	338.5	216
Time saving with SumXcore	**36%**	

Table 1. Achievable time savings with SumXcore technology

The applications contained calibration lines for 18 oxides. All 18 oxides were measured sequentially, using optimal settings, on the WD Core.

In the SumXcore application, 11 of the 18 oxides were measured sequentially using the WD core and the remaining 7 oxides were measured simultaneously using the ED core.

Table 1 provides a summary of the measurement times. It can be seen that the combination of the WD and ED cores within the SumXcore delivered a 36 % reduction in total analysis time, due to time savings in both measurement time and overhead time (the time required to change crystals, collimators filters, etc.). Table 2 demonstrates the accuracy of the SumXcore application when measuring Japanese Standard Sample JSS 830-3.

WD/ED	Element/ compound (as per certificate)	Certified concentration (wt%)	SumXcore measured (wt%)
ED	Total Fe	60.57	60.52
	Mn	0.61	0.62
	SiO_2	2.26	2.19
	TiO_2	6.33	6.34
	V	0.30	0.30
	Zn	0.075	0.075
WD	MgO	2.15	2.24
	Al_2O_3	2.75	2.75
	P	0.124	0.124
	S	0.005	0.006
	CaO	0.68	0.71
	Cr	0.018	0.016
	Cu	0.011	0.009
	Ni	0.006	0.006

Table 2. Accuracy of the SumXcore application

get insight

PANalytical Australia
Unit 24/31 Governor Macquarie Drive
Chipping Norton, NSW 2170, Sydney
T: +61 2 8700 2700
E: info.australia@panalytical.com

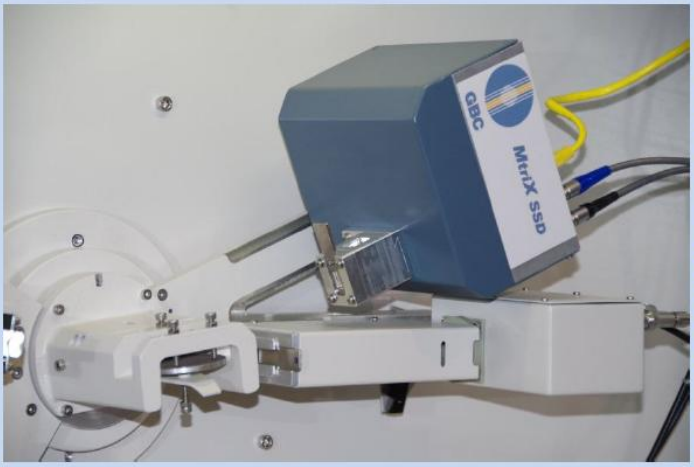
www.panalytical.com

Diffraction Technology

Instruments and accessories for X-ray analysis

The FCT Nova SSD /GBC MtriX.

Now with enhanced energy discrimination. Gives better signal/noise ratio by rejecting Fe fluorescence when analysing Fe rich samples using CuKα.



The Nova SSD can be added to any XRD where stepping motor pulses are available.
It is stand-alone and there is minimal intrusion into the normal functioning of the XRD.
Scanning is done with the XRD operating software, data collection is done in the detector and a file saved in the PC.

A GBC MtriX detector shown fitted to a GBC EMMA

The addition of a fast detector allows you to scan 30x faster with the same counting statistics. It can give you a real boost in throughput without the expense of a new instrument.
The SSD comes as a complete system with detector, power supply and software. The only installation required is to take out the stepping motor pulses from the XRD controller.
Documentation is included , but if you want a turn-key system, Diffraction Technology can offer installation.

Diffraction Technology e-mail diffraction@bigpond.com phone 03 9787 3801 Web www.diffraction.com.au

IMP Introduces Premium Free Flow Flux

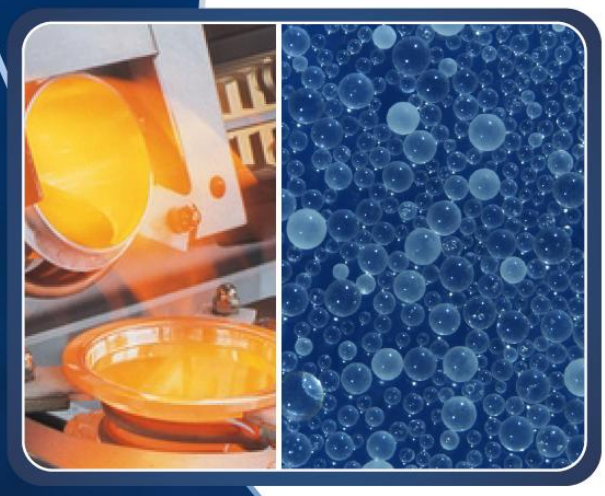
IMP now offers a premium free flow flux for use in XRF fusions.

This flux is spherical in nature and offers much better flow characteristics while producing far less dust.

Purity is exceptional and the spherical shape means that dosing is faster and the flux is less susceptible to moisture.

It typically consists of 35.3% Lithium Tetraborate and 64.7% Lithium Metaborate, but any other flux ratios are available upon request.

We also offer a granular version of this high quality flux for other applications where the spherical nature may not be preferable.



IMP Group Australia
Tel: +61 (8) 9204 4200
Fax: +61 (8) 9204 1128
E-mail: info@impgroup.com.au
www.impautomation.com

Challenging Conventional Sample Processing!

IMP
Innovative Solutions

New Automated HP-MP Combination Mill/Press for Pressed Pellet Production at BlueScope Steel

IMP is the proud supplier of a new automated HP-MP combination mill and press machine for BlueScope Steel in Wollongong.

This included a custom software interface to communicate with their existing XRF and XRD instruments as well as their LIMS. The HP-MP is a hermetically sealed and sound proofed automated mill and press machine for the production milled or pressed analytical samples.

Integrated Simatic PLC control system guarantees error-free, automatic preparation of the widest possible variety of samples. Up to 8 parameter-definable preparation programs can be stored and password-protected.

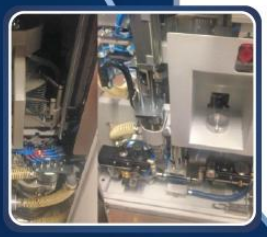
The milling and pressing processes can be executed separately if required. The machine automatically self-cleans between each sample to minimise cross-contamination.

Benefits of the system

- Space saving design with the integration of the components for milling and pelletizing in one machine
- Machine parameters are flexible and easily set
- The milling and pelletizing process can be executed separately if required
- Available as a stand-alone solution or ready for integration in all automation types

Specifications

Dimensions (L x W x H)	750 x 1120 x 1404 mm
Machine weight	~630 kg
Grinding vessel	Tungsten carbide or hardened tool steel
Steel press ring dimensions	Ø51.5 x 35 x 8.6 mm Ø40 x 35 x 14 mm
Processing parameters Milling time Press force	 0 – 999 s 0 - 160 kN
Processable samples Particle size Hardness	 < 5 mm up to 50 cc – dry < 9 Mohs



IMP Group Australia
Tel: +61 (8) 9204 4200
Fax: +61 (8) 9204 1128
E-mail: info@impgroup.com.au
www.impautomation.com

Challenging Conventional Sample Processing!

IMP
Innovative Solutions

INTERNATIONAL CENTRE FOR DIFFRACTION DATA

LET OUR TEAM OF EXPERTS HELP YOU TAKE YOUR SKILLS TO THE NEXT LEVEL!

Practical X-ray Fluorescence: 25 – 29 April 2016

From theory to hands-on exercises, this course offers techniques and skills to improve lab performance. Discover the latest in cutting-edge instruments such as TXRF, hand-held devices, energy dispersive and wavelength dispersive spectrometers through live demonstrations.

The XRF course covers the basics of X-ray spectra; instrumentation design; methods of qualitative and quantitative analysis; specimen preparation and applications for both wavelength and energy dispersive spectrometry. Emphasizing quantitative methods; use of automated X-ray spectrometers; review of mathematical matrix correction procedures and new developments in XRF.

Fundamentals of X-ray Powder Diffraction: 16 – 20 May 2016

For the novice with some XRD knowledge or for the experienced with an interest in the theory behind XRD, this clinic offers a strong base for increased lab performance.

The clinic covers instrumentation, specimen preparation, data acquisition and qualitative phase analysis. Hands-on use of personal computers for demonstration of the latest software; data mining with the PDF. The powder diffractometer: optical arrangement, factors affecting instrumental profile width, choice and function of divergence slit, calibration and alignment, detectors, X-ray optics.

*Advanced Methods in X-ray Powder Diffraction: 23 – 27 May 2016

For the experienced XRD scientist, this session offers enhanced analysis skills through intense problem solving, as well as an introduction to the Rietveld Method. Emphasizing computer-based methods of data collection and interpretation, both for qualitative and quantitative phase analysis.

The advanced clinic covers factors affecting d-spacings of crystals: unit cell, crystal structure, and solid solutions, as well as factors affecting diffraction-line intensities: relative and absolute intensities; structure-sensitive properties (atomic scattering and structure factors), polarization effects, and multiplicity; specimen-sensitive effects (orientation, particle size), measurement-sensitive effects (use of peak heights and peak areas), and choice of scanning conditions.

Rietveld Refinement & Indexing Workshop
Basic Workshop: 26 – 28 September 2016
*Advanced Workshop: 28 – 30 September 2016

Powder Pattern Indexing and Rietveld structural refinement techniques are complementary and are often used to completely describe the structure of a material. Successful indexing of a powder pattern is considered strong evidence for phase purity. Indexing is considered a prelude to determining the crystal structure, and permits phase identification by lattice matching techniques. This workshop introduces the theory and formalisms of various indexing methods and structural refinement techniques. One unique aspect of this workshop is the extensive use of computer laboratory problem solving and exercises that teach method development in a hands-on environment.

Take the three-day basic workshop, the three-day advanced workshop or both together for a full week of hands-on training.

Register Today at WWW.ICDD.COM/EDUCATION

* See the ICDD web site for prerequisites for advanced courses.

LOCATION

ICDD Headquarters, 12 Campus Boulevard
Newtown Square, Pennsylvania 19073-3273 U.S.A.

ICDD® International Centre for Diffraction Data

ISO 9001:2008 Quality Assured Company

FOR MORE INFORMATION CONTACT
Eileen Jennings, Education Coordinator
Tel: **610.325.9814** Fax: **610.325.9823**
Email: **clinics@icdd.com**

ICDD 12/15

PRESS RELEASE

Katanax X-600 Automated Fusion Machine Performs Faultlessly During Intense Trial

Sydney, Australia, November 10, 2015 -
Following the recent release of the Katanax X-600 automated fluxing machine for producing XRF fusion beads we had Magid Sawiris, Chief Chemist at Ravensthorpe Nickel Operations (RNO), owned by FQM Australia Nickel Pty Ltd, a subsidiary of First Quantum Minerals Limited evaluate the system. RNO operate an open cut nickel and cobalt mine producing mixed hydroxide precipitates through a highly technical hydrometallurgical process.

Magid's state-of-the-art laboratory provides technical support for their entire operation. As such they are required to analyse samples from all areas of the operation, from minesite ore through to final product. The results they produce can have a direct impact on plant efficiency, productivity and ultimately profitability.

One of the key technologies they employ is XRF which provides elemental compositions of samples. To produce results of sufficient accuracy and reliability, fusion beads are the only solution. To date, they have been using an upgraded Katanax K2 Prime which can process 6 samples simultaneously.

Following installation of the new Katanax X-600 and comparison with the K2 Prime, Magid said "Katanax have obviously listened to the feedback from users of previous models. The X-600 has a much more robust design and other features that are improvements that will endure the rigors of a modern high volume production laboratory and should produce better reliability and longevity."

Some of these features that Magid was referring to include a higher content of ceramic componentry that can better deal with the heat, heating elements located away from the samples providing better protection from evolved gases and element redundancy, which means the system can continue to operate if you lose an element.

Magid Sawiris putting the Katanax X-600 through its paces making XRF fusion beads.

Media Contact:

Dr. Cameron Chai
Marketing Communications Manager
T. +61 (0)2 9450 1359
E. cameron.chai@axt.com.au

RNO trialed the system for 6 weeks in which time the X-600 was run 24/7, performing a minimum of 20 cycles per day. This resulted in the production of well over 5000 fusion beads. At the conclusion of the trial, he remarked, "the X-600 did everything it was supposed to do. It performed faultlessly in the time we had it. It was easy to use and to the best of my knowledge, is the most advanced system on the market. When the time comes, I would certainly be interest in buying one!"

AXT distribute Katanax automated fusion machines in Australia. For more details please visit www.axt.com.au or contact info@axt.com.au.

PRESS RELEASE

Another High-Powered X-Ray Diffractometer Destined for Australia

Sydney, Australia, November 16, 2015 – AXT is proud to announce the sale of another 9kW Rigaku SmartLab X-ray Diffractometer (XRD). This is the second order for a high-powered XRD in as many years. This system will be installed at CSIRO's Manufacturing Flagship in the Melbourne suburb of Clayton.

The 9kW SmartLab is powered by Rigaku's patented rotating anode technology which generates an X-ray flux second in intensity only to a synchrotron. These systems produce a flux almost 6 times greater than conventional sealed tube systems that are rated to 3kW, but rarely operate above 1.6kW (40kV, 40mA). The higher X-ray flux generates greater intensity at the detector allowing you to generate data faster as well as providing you with a better chance of detecting trace phases.

The system that the CSIRO have ordered also benefits from a HyPix 3000 detector. These detectors are based on Hybrid Pixel Array Detector (HPAD) technology and are the perfect partner for a high flux x-ray source. They employ semiconductor technology enabling resulting zero noise generation and extremely fast data acquisition. Furthermore, using Rigaku's intelligent Guidance software, the detector can be switched from 0D, 1D or 2D detection modes by simplistic operator selection.

Rigaku's Guidance software also makes switching between analysis modes a trivial task. The intelligent system shows you what hardware components are required and guides you through their installation. Optically encoded components ensure you connect the correct items. Following an auto alignment (unique to Rigaku diffractometers), the system is ready to start collecting data.

"We were looking for a cutting edge analytical solution. After reviewing what the marketplace had to offer, the 9kW Rigaku SmartLab was the best fit for our requirements", said Dr. Aaron Seeber, Research Engineer from CSIRO Manufacturing. His colleague Natasha Wright, Group Leader Biophysics added, "with wide a range of users with varying needs, the combination of high power, system intelligence and flexibility was best placed to serve us now and into the future."

The 9kW Rigaku SmartLab X-ray diffractometer.

Media Contact:

Dr. Cameron Chai
Marketing Communications Manager
T. +61 (0)2 9450 1359
E. cameron.chai@axt.com.au

Richard Trett, AXT's Managing Director commented, "The University of Queensland recently installed a very similar system earlier in the year. While both of these system are tailored specifically for thin film analysis with an in-plane arm, it is also capable of analysing a range of other materials including solids and powders. This sale demonstrates Australia's willingness to purchase instruments to future proof their labs and the acceptance of rotating anode technology."

AXT represent several Rigaku product lines including XRD, WDXRF, Thermal Analysis and NDT in Australia and New Zealand. These form part of AXT's comprehensive materials science product portfolio. More information on these products can be found at www.axt.com.au.