AXAA e-Newsletter

Issue 1, 2016 **Australian X-ray Analytical Association**

President's Address

Dear AXAA Members and Friends,

Things at AXAA central are running hot, with our AXAA-2017 Conference Committee meeting twice so far to plan what is sure to be a great event. In 2017 we look forward to the event being hosted in Melbourne, which is home to the Australian Synchrotron, Australia's premier X-ray characterisation and research facility.

Our conference theme is "***Innovation from Characterisation***", with focus topics to include mining, mineral processing and metallurgy, geology and geochemistry, functional materials, energy, environment, cultural heritage, biomaterials, additive manufacturing and thin films. Analytical methodologies, in addition to XRF and X-ray and neutron diffraction, will include XFM, tomography, total scattering/PDF, small-angle scattering and XAS.

Our plenary speakers will be announced in the coming weeks, before abstracts open on 11th May 2016, with abstract submission deadline the 7th October.

If you have interest in holding a pre or post-conference workshop as a satellite to the main event, now is the time to contact us. Similarly, if members want to see something specific in the workshop program, we greatly appreciate this information.

There is a range of accommodation options around the Pullman Albert Park, including the conference venue itself, to suit a range of budgets. We also have an exciting social programme planned, as well as a student careers session which will make AXAA-2017 a must attend event for students and newcomers to the field.

Finally, now is also the time to consider nominations for the Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis, and the Bob Cheary AXAA Award for Excellence in X-ray Diffraction Analysis. These prestigious awards recognise significant long term contributions to X-ray analysis. Please read on for further details.

Nathan Webster
AXAA President

Key Dates for AXAA-2017:

- Abstracts open 11 May 2016
- Registration open 6 July 2016
- Abstract deadline 7 October 2016
- Earlybird registration closes 18 November 2016

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Achieving Truly Quantitative XRD Mineral Phase Analysis in a High-Throughput Industrial Setting

Context:

The following guidance on self-auditing of XRD phase composition analysis (PCA) capability is written principally for industrial XRD laboratories with high sample throughput requirements which must be performed cost-effectively and in compliance with ISO QA/QC specifications. Much of the advice is also relevant to the needs of laboratories with more modest requirements, notably university and government research XRD laboratories with a large number of non-specialist XRD users.

The advice is based on the premise that the laboratory has a high-quality Bragg-Brentano (BB) diffractometer, and also makes use of state-of-the-art XRD software such as that provided by Bruker, PANalytical and Rigaku.

It is a given that the laboratory uses Rietveld (RTV) PCA analysis of the XRD data, noting that the older line-ratio methods must not be used.

The likely staffing scenario for the laboratory will be that technical oversight is provided by a high-quality analyst who is not necessarily a RTV expert, but is closely mentored by an external RTV expert(s). Staffing-wise it is also assumed that the laboratory must make use of operator staff who have little knowledge of XRD and RTV, perhaps operating on a 24-7 basis to complete up to 200 samples/day.

Key Audit Questions and Comments:

1. What are the typical particle size ranges of your XRD samples for quantitative XRD?

- Acceptable answer is 'Mainly between 1 – 10 microns' as this is usually necessary for the XRD intensities to have acceptable reproducibility
- How does the XRD lab monitor particle size and is this incorporated in the QA program?
- Does the lab QA program include testing of intensity reproducibility?
- Coarse samples, say 30 microns as produced with a ring mill, make truly quantitative work utterly impossible
- The popular way of milling powders with acceptable granularity is to use a bank of micronising mills

2. How does the XRD lab mount the samples in the BB specimen holder?

- Acceptable answer is 'Using an XRD press which applies ca. 10 bar (0.01 tonnes/cm^2) to the sample'
- Manual packing of the sample is generally unacceptable unless the operator has remarkable skills. Also, manual packing typically leads to unacceptable variations in specimen displacement
- Use of an XRF press, delivering a pressure of (say) 10 tonnes/cm^2, produces unduly compacted samples which is entirely unacceptable
- How are mounting-dependent preferred orientation effects minimised?

3. Is the instrument correctly aligned?

- Acceptable answer is 'The laboratory QA program includes an assessment of RTV fitting of XRD data for a suitable standard'

4. Does the laboratory regularly determine the instrument two-theta-zero value of the instrument?

- Acceptable answer is 'The two-theta-zero correction is checked frequently using line position standards, and also whenever the instrument optics are altered', e.g. (i) by changing the incident beam divergence slit, and (ii) after instrument re-alignment
- Again, the two-theta-zero must be monitored as part of the QA program
- Once determined, the correction must be fixed for data analysis – for phase identification and RTV analysis

5. How do you ensure that the sample identification has been performed correctly prior to RTV analysis and then validated as part of the RTV computations?

- Do you use the ICDD Powder Diffraction File (PDF) and the complementary ICSD Structural Data Base, along with XRD pattern simulations, to validate the set of phase Crystallographic Information Files (CIF) to be used in the RTV PCA analyses?
- Does the laboratory use statistical cluster analysis to flag samples containing unexpected phases?
- How do you select an acceptable set of CIF files for RTV PCA analysis?

6. Are the RTV PCA procedures calibrated to ensure operator-proof XRD PCA analyses?

- Fast, reliable analyses requires optimisation of 'troublesome' RTV parameters for use in batch RTV PCA computations - notably two-theta-zero and line profile parameters
- Having minimised XRD pattern bias due to PO effects from sample preparation and mounting, how do you miminise PO PCA bias caused by your RTV calculations?
- Do you make use of batch RTV analysis to provide rapid sample throughput?

7. Do you use PCA calibration standards to place the PC levels on an absolute scale?

- You are not doing truly qualitative analysis if PCA standards are not employed – so-called 'standardless' analysis has no place in an ISO-compliant XRD phase analysis program
- Generally, use of external standards is preferable to using internal standards

- What collection of XRD PCA standards does the laboratory own? How have these been characterised? How are the PCA standards stored?

8. How does the XRD lab pre-determine the non-diffracting-content (NDC) corrections for your PCA target phases as part of the process of determining the 'amorphous' content of samples?

9. How do you use elemental analysis data and NDC values to check the validity of the RTV phase analysis numbers?

- The laboratory QC program must specify how the RTV PCA results are tested for ISO compliance
- Does the QC program include regular PCA analysis of in-house reference samples?

10. How have you protected the quality of your XRD PCA analyses against (i) the loss of key staff, (ii) compromising the XRD PCA methods by 'innovative' operators, and (iii) operator mistakes?

- Protective measures must be detailed in the QA/QC documentation
- Training is obviously of paramount importance, and must be documented.

Prof Emeritus Brian O'Connor
Curtin University
Brian O'Connor Consulting
brian_oconnor@iprimus.com.au

Rapid XRD Screening of Mineral Samples for Crystallinity, Phase Identification and Quantification

The fast pace of modern scientific investigations has increased the demand for rapid materials characterisation. Compounding this challenge has been the introduction of robotic sample preparation, where vast numbers of unique specimens are rapidly prepared. In this study, collection and analysis of XRD data in a high-throughput environment, is tested.

The instrument used for the data collection was a Bruker D8 Discover, equipped with a Vantec 500 high-speed 2D detector, motorised 3-axis stage, and a Cu targeted X-ray tube with poly-capillary optics for high beam intensity. The 2D detector was selected to help minimise errors caused by preferred orientation and grain size effects. The samples were packed into a 30 well steel plate, which was mounted vertically on the translation stage so that the samples were analysed in transmission geometry. Starting materials for this investigation were quartz (SiO_2), zincite (ZnO) and corundum (Al_2O_3) reagents, weighed and combined to produce 6 samples, mixed v/v in PVA for analysis.

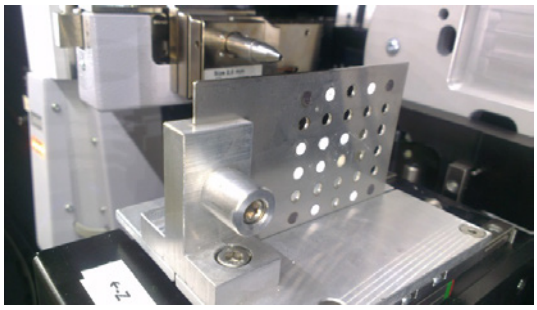


Figure 1. Well plate sample holder mounted on 3-axis translation stage

Data collection consisted of a two part strategy. Firstly, for the purposes of centring the well plate axially with the goniometer, a silicon reference material was loaded into the four corner wells. By undertaking a rapid (60 s) snapshot with a range of 33.6° 2θ, multiple Si peaks could be observed, and variation in sample displacement could be accurately corrected for. This correction was then applied to the remaining wells in the plate assuming a linear relationship both vertically and horizontally.

Analysis of the samples required data collection over the angular range 20°-80° 2θ, which exceeds the maximum collection angle of the Vantec 500 in one shot. However, the Diffrac.Suite collection software allows multiple overlapping ranges to be combined simply. Thus, collections proceeded with 3 detector positions and 480 seconds per position, to cover a 60° 2θ range in ~25 minutes.

The combined 2D images, containing multiple diffraction cone segments, were reduced to conventional 1D diffractograms using the analysis software EVA. To accelerate the search-match procedure, chemical information was obtained directly from the samples mounted in the 30 well plate using a Bruker M4 Tornado μ-XRF (Figure 2). Using this approach, the composition of each sample could be qualitatively determined in less than a minute, with no special sample preparation required. This information was then used as a filter in EVA, allowing the phases to be readily identified (Figure 3).

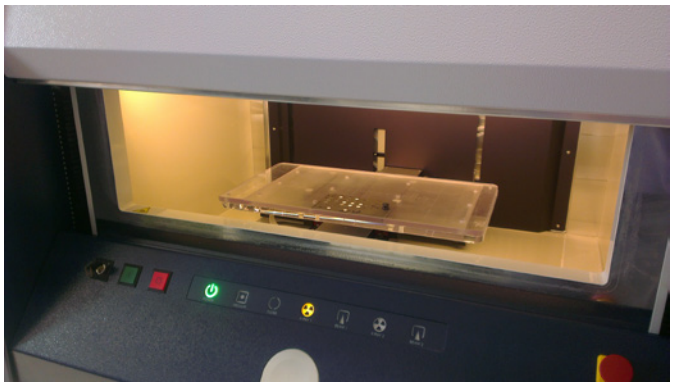


Figure 2. Samples mounted in the 30 well plate being loaded into the Tornado μ-XRF for rapid chemical analysis.

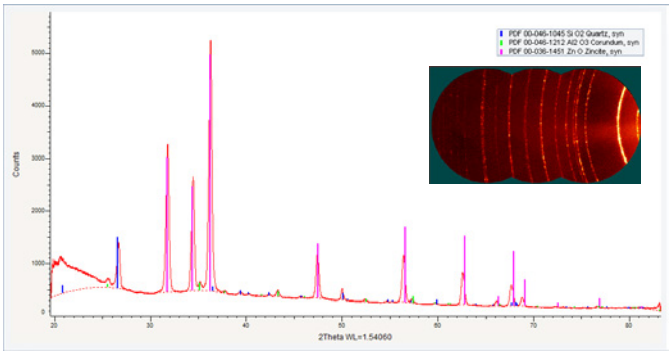


Figure 3. Phase identification was undertaken on sample C1 using the refined search criteria based on the Tornado µ-XRF findings. Inset shows the combined 2D detector images for this sample.

Rietveld-based quantitative phase analysis (QPA) was undertaken using Topas. The instrument profile was refined based on the reference samples in the four corners. The models for the sample phases contained only a few parameters that were allowed to refine from their accepted values, aiding the speed and stability of the refinement. Despite these constraints, the analysed weight fractions obtained were in good agreement with the known compositions (Table 1).

Table 1. Known phase fractions of the blended samples compared with the relative abundances calculated using Topas, and the elemental composition determined by µ-XRF (* denotes trace elements detected, ^denotes impurity phase).

Sample	Phase	Weighed wt.%	Measured wt.%	Elements µ-XRF
C1	Al_2O_3	10	8.3	Al, Zn Si, O
	ZnO	70	73.8	
	SiO_2	20	17.9	
C2	Al_2O_3	20	19.6	Al, Zn, Si
	ZnO	10	8.3	
	SiO_2	70	72.1	
C3	Al_2O_3	70	68.7	Al, Zn, Si
	ZnO	20	18.3	
	SiO_2	10	13.0	
S1	Al_2O_3	100	100	Al, O, *Br, *Zn, *Si
S2	ZnO	100	100	Zn, O, *Al
S3	Al_2O_3	0	0.6^	Si, O, *Fe, *Br, *Cr, *Al, *Zn
	ZnO	0	0.2^	
	SiO_2	100	99.2	

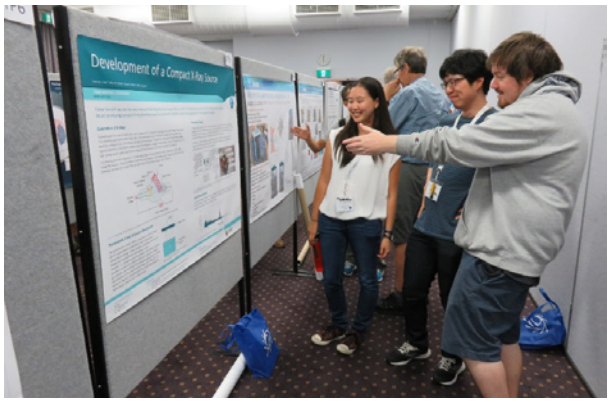
In summary, multiple samples have been analysed by XRD and µ-XRF in a high-throughput manner. Displacement of a multi-well plate in transmission geometry can be corrected for using reference samples located in the four corners. The 33.6° 2θ window of the Vantec 500 detector allowed for single snapshots with excellent resolution and repeatability, and multiple images could be easily combined to extend the 2θ range. The 2D detector is advantageous when examining samples with preferred orientation and larger grain sizes, particularly with stationary samples. In addition, the ability to collect chemical information directly from the samples mounted in a multi-well plate by micro-XRF can greatly accelerate data analysis.

Barry Halstead[1] and Chris Kelaart[2]
1) CSIRO Minerals Resources
2) Bruker Australia

Wagga 2016 Wrap-Up

Over 4 days from February 2 – 5 of this year, the 40th annual meeting of the Condensed Matter and Materials (CMM) group of the Australian Institute of Physics was held once again at its traditional venue, the Wagga Wagga campus of Charles Sturt University in New South Wales. This year's "Wagga" conference was organised by a committee from the Australian Synchrotron, bringing together the Australian condensed matter and materials community, as well as researchers from further abroad, to discuss the latest research and directions in the field. A total of 116 delegates attended the conference, including 10 international attendees from China, Scotland, Taiwan and New Zealand. The scientific program consisted of 11 invited and 29 contributed talks, as well as 70 posters covering topics ranging from surface science, semiconductors, thermoelectrics, magnetism, ferroelectrics, multiferroics through to theoretical physics and advanced materials. The use of hard and soft X-rays for materials characterisation was a common theme, with many presentations featuring synchrotron and/or laboratory based X-ray scattering and diffraction techniques. A strong student participation in to the conference was seen this year, with Masters/PhD students contributing approximately half of the submitted abstracts. Over the four days of the conference, delegates networked with each other in the informal atmosphere for which the Wagga conference is well known, whilst more than a few PhD students delivered the first oral conference presentations of their career, a rite of passage many within the Australian condensed matter community remember very well.

Conference chair Anton Tadich (Australian Synchrotron) discusses soft X-ray spectroscopy of graphene on SiC, with invited speaker Assoc. Prof. Francesca Lacopi, Griffith University.



PhD student Ms Emily Yap (UNSW) discusses her poster on the development of a compact X-ray source with fellow PhD students at the Thursday poster session

Anton Tadich
Australian Synchrotron

WA SAXS Facility Now Equipped With the Latest in X-Ray Technology

The Western Australian Small Angle X-Ray Scattering Facility (WA-SAXS) was established in 2002 with a sealed copper tube providing the X-rays for one of the most versatile techniques in the characterisation tool-kit. Since that time rotating anode technologies greatly improved the capability of laboratory based SAXS instruments. Using a liquid metal anode represents the next leap forward in laboratory X-ray generation and the improved brightness of the primary beam will open up applications previously out of reach at the facility. This new X-ray generation technology is provided as the source for the Bruker NANOSTAR with a VÅNTEC-2000 detector and is one of four such facilities in the world. The new Bruker NANOSTAR was purchased via funding from the ARC.

The MetalJet-D2 X-ray source provided by Excillum uses a pressurised jet of liquid gallium which can take the heat load from a more powerful electron gun than previously possible. In addition to the latest X-ray source the new instrument is equipped with scatterless pinholes that are currently configured to the high flux setting allowing the system to provide the maximum number of X-rays to the user's samples. As the only SAXS facility in Western Australia the facility services a wide range of users within the local research community so the system will be set up to be as flexible as possible.

In addition to the standard sample environment (powder and capillary samples mounted at ambient temperature under vacuum) there is also the possibility to measure samples on a heated stage up to approximately 300°C for time resolved experiments. The diverse local research community will drive the direction of the facility as we look to add a range of sample stages and features in the coming years ranging from a Grazing Incidence-SAXS (GI-SAXS) attachment, to a flow cell, to an auto-sampler for large numbers of liquid/solution samples. In addition to varying sample environments there is the potential to include high resolution pinholes which would provide greatly improved spatial resolution for the X-ray beam.

For a case study we present the last published data set from the original Bruker NANOSTAR equipped with a sealed copper tube X-ray source. This experiment aimed to investigate the structure of mesoporous silica with embedded palladium nanoparticles [1]. In the experiment powder samples were originally measured for 3 hours per sample at 22.5 cm sample detector distance. During the installation of the new MetalJet-D2 equipped NANOSTAR in March 2016, these same samples were measured with a collection time of 300 seconds. This represents a collection time of less than 3% of what was achievable at the facility prior to the installation of the MetalJet-D2 X-ray source.

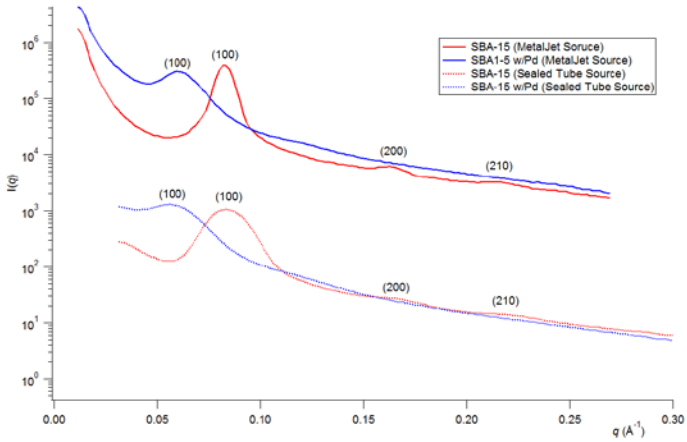


Figure 1. Scattering curves from the new MetalJet-D2 X-ray source (solid line) and the previous Cu sealed tube (dotted lines). The curves were corrected for collection time and the use of an Al foil to attenuate the beam from the MetalJet-D2 source (to prevent saturation of the detector). The curves are of samples of SBA-15 (red) and SBA-15 that has been doped with palladium nanoparticles (blue).

The scattering curves shown in Figure 1 have been indexed for the observed reflections from the hexagonal ordering of the pore structure. The peaks observed from the MetalJet-D2 source show a much improved signal to noise ratio even with the reduced collection time. The (100) reflections also show sharper profiles again related to the improved counting statistics on the new instrument. The sample-detector distance has been increased to 107 cm and along with the shorter wavelength of the gallium anode measurement is possible down to q = 0.0114 Å^{-1}.

The WA-SAXS Facility is available to external users, and we invite users investigating materials with a nanostructure to contact Dr Kevin Jarrett (kevin.jarrett@curtin.edu.au) to discuss your needs and the capabilities of the instrument.

Kevin Jarrett[1,2] and Craig E. Buckley[1,2]
1) John de Laeter Centre http://jdlc.edu.au/
2) Department of Physics, Astronomy, and Medical Radiation Sciences Curtin University, Western Australia.
[1] Tong *et al.*, *RSC Advances*, **5** (2015) 20557 – 20561

XRF In The Work Place Training Courses

The established and most sort after XRF In The WorkPlace (XITW) courses have been running for more than 10 years now in Australia. The next XITW course in Sydney will be held between the 11th – 15th July 2016 at the Analytical Centre in the University of New South Wales (UNSW). This course is run twice every year in Perth and in Sydney for the convenience of participants on both the East and West coasts.

The XITW course is aimed to provide quality training on XRay Spectrometry (XRF) technique and its use in work environment whether it is in industry or research. It starts as a basic building block for personnel who are new to XRF or for operators who run the daily routine on the XRF but would like to learn the principles and practical use. It also gives insights to Supervisors and Managers who would like to have better understanding and control on day to day analytical issues and decision making.

It is a full five days course which covers class room sessions on theory of spectrometry and basic principles of XRF instrumentation. Practical sessions in the lab on use of different types of XRF (WD and ED), calibrations and hands on sample preparation methods (fusion, pressed powders etc) for XRF analysis are comprehensively covered in this course. Participants are provided with lots of reading material including the "XRF in the Work Place" text book (Red book).

Ken Turner conducting the course exam on the last day.

The course is tutored by highly experienced industry professionals Ken Turner (XRF Consultant), Hari Bhaskar (PANalytical Specialist) and Dr Gary Pritchard (XRF Consultant). Invited lectures from industry and academia professionals are also part of the course giving a good opportunity for participants to interact with peers and experts from various fields.

XRF users of all types and brands have participated in the XITW courses in the past years and have found the course very educative and useful for their work environment.

For more info or to register for the course please contact hari.bhaskar@panalytical.com or your nearest PANalytical office to get a course flyer.

Hari Bhaskar
PANalytical Australia

AXAA Awards for Excellence in X-ray Analysis: Call for Nominations

Closing Date for Submission of Nominations:
28 October 2016

The Awards

Two awards have been established, one for XRF and one for XRD. These will be for "significant long term contributions" to X-ray analysis rather than say a single paper, and will perpetuate the contribution of the person after whom the award is named.

XRF – Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis

XRD – Bob Cheary AXAA Award for Excellence in X-ray Diffraction Analysis

Form of Awards
Each award will comprise an engraved medal.

Selection Criteria

1. The principal criterion will be the excellence of the applicant's development of high-impact, innovative X-ray analysis methods and their take-up by the X-ray analysis community. Work in which XRF or XRD has been a peripheral tool will not be considered.
2. The period over which the contribution is to be considered will be at least 5 years.
3. All or most of the cited work will have been conducted in Australia.
4. The recipient will have been a member of AXAA for at least 5 years prior to the application being submitted.
5. It is desirable, but not essential, that the applicant has contributed to AXAA in a substantial way, for example through quality presentations at AXAA national conferences and/or administrative service for AXAA.
6. Past recipients of an AXAA XRD or XRD award will not be considered for a second award in the same category.

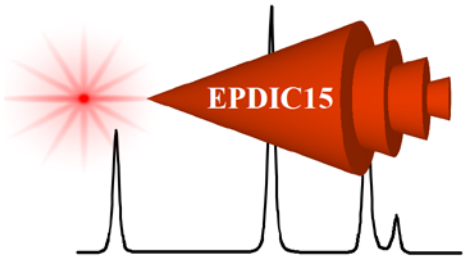
Applications

Applications will be submitted by a nominator on behalf of the applicant. The documentation will comprise:

- CV.
- Publication list. This may include items protected by confidentiality if the applicant can pre-arrange an appropriate confidentiality agreement.
- Advocacy statement highlighting the application's alignment with the selection criteria.
- Names and contacts for three technical referees, one being the nominator.

Applications are to be submitted in Nathan Webster (Nathan.Webster@csiro.au). Please send to Nathan as an attachment.

Upcoming Events



15th European Powder Diffraction Conference

12-15 June 2016
Bari, Itally

2016 is a landmark year for powder diffraction history: almost a century has passed since the pioneering works and experiments of Debye, Scherrer, and Hull; fifty years have passed since the first publication of the Rietveld method. In the last century, the developments theoretical, experimental and computational have enabled spectacular and stimulating progress in the field of powder diffraction.

Started in 1991, EPDIC is now the Biennial Conference that brings together scientists and instrumentation companies to brainstorm on the latest trends and challenges in powder diffraction.

Abstracts are now closed, however earlybird registration is open until 21 April.

For more information:
Email: epdic15@ic.cnr.it
Website: http://www.ba.ic.cnr.it/epdic15/

65th Annual Conference on Applications of X-ray Analysis – Denver X-ray Conference

1-5 August 2016
Rosemont, Illinois, USA

Attendees to the World's largest X-ray conference will have access to sessions on the latest advancements in XRD and XRF. Workshops are run by experts who provide training and education on many practical applications of X-ray fluorescence and X-ray diffraction techniques for the study of materials.

Abstracts are now closed, however earlybird registration is open until 1 July.

For more information:
Email: dxc@icdd.com
Website: http://www.dxcicdd.com/index.htm

X-ray Materials Analysis Internet Courses – Wavelength Dispersive XRF and Powder XRD (Plus 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 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

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 Twitter @axaa_org.

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Please email contributions for Issue 2 of the 2016 AXAA Newsletter to Mark Styles by Friday the 29th of July. 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.

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INTERNATIONAL CENTRE FOR DIFFRACTION DATA

Powder Diffraction

An International Journal of Materials Characterization

Published quarterly (four issues and one topical supplement) by Cambridge University Press

Powder Diffraction (PDJ) focuses on materials characterization employing X-ray powder diffraction and related techniques. With feature articles covering a wide range of applications, from mineral analysis to epitactic growth of thin films to advances in application software and hardware, this journal offers a wide range of practical applications.

Proceedings of the Australian X-ray Analytical Association (AXAA) Conference held February 9-13, 2014

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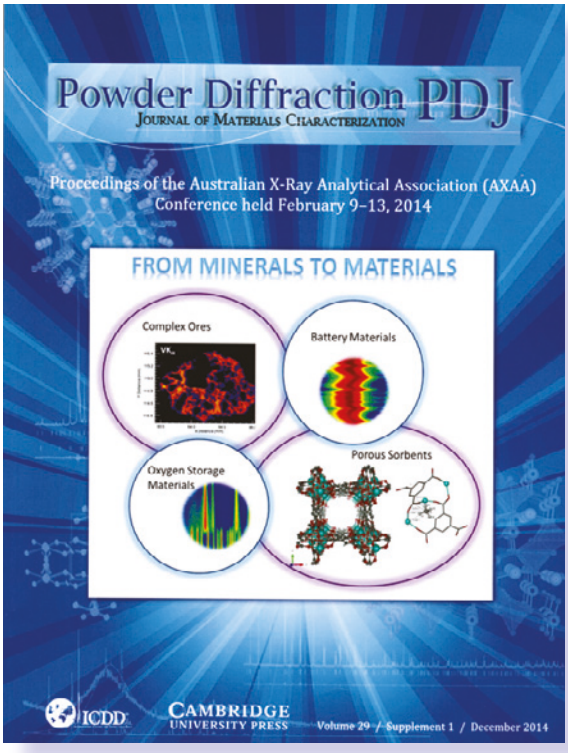

- Techniques and procedures in X-ray powder diffractometry
- Advances in instrumentation
- Study of materials including organic materials, minerals, metals and thin film superconductors
- Publication of powder data on new materials

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Figure 1-2. *Advances in X-ray Analysis*, Vol52; A PORTABLE NON-INVASIVE XRD-XRF INSTRUMENT FOR THE STUDY OF ART OBJECTS. P. Sarrazin, G. Chiari, M. Gailhanou. Copyright ©JCPDS-International Centre for Diffraction Data 2009 ISSN 1097-0002.

PANalytical
X'press Updates

Scott Gilroy
is named PANalytical Australia's new General Manager

as of 18 December 2015. Scott is a familiar face within the local market, having joined the team since 2004 as the Regional XRF Product Manager as well as Regional Marketing Manager for Asia Pacific. He was instrumental in sharing the latest innovations in XRF. For example, PANalytical's newest XRF spectrometer, the Zetium, finalist of R&D 2015 innovations which combines both energy and wavelength dispersive technologies.

"Scott has displayed great dynamism and technical knowledge. He is passionate about helping industries find the best solution for their analysis. Backed by a strong team, we are confident that Scott will be able spur greater awareness of X-ray technology for elemental analysis within Australia," says Gjalt Kuiperes, the Regional Director for PANalytical Asia Pacific.

Upcoming Webinars

Which is better - Wavelength (WD) or Energy dispersive (ED) X-ray Fluorescence? Several years ago the answer was WD. However due to the evolution of technology especially with ED XRF, the answer is not so simple.

A clear example of the change is the move from proportional counters with resolution at > 600eV down through to impressive Silicon Drift detectors at ~ 135eV. So tune into our live webinars to find out more!

Which technology is better – WD or ED XRF?
14th April

In situ / in operando XRD characterization of lithium-ion batteries
19 May

www.panalytical.com/webinars

3x the submissions
received for 2015 PANalytical Award!

from the Asia Pacific region compared to 2014. Australia accounts for 26% of the region's submissions. PANalytical thanks you for your active participation! The PANalytical Award is awarded yearly to promote the use of X-ray techniques, regardless of the brand of instrument or software. Our previous winner was *Matteo Bianchini*, from Europe, who researched on a promising new Li-ion system, using in situ and ex situ X-ray and neutron diffraction. The award has yet to be won by an Asia Pacific researcher and we hope for one from Australia! An international independent panel is currently reviewing the 2015 global submissions.

Participate in the 2016 PANalytical award and stand to win our €5,000 cash prize!

www.panalytical.com/award

Using X-ray techniques for your research? Stand to win the 2016 PANalytical Award

Are you a first author to a paper to be or already published between 1 Jan 2015 and 1 Dec 2016? Is you paper published in a journal recognized by the Institute for Scientific Information. Does the journal have an impact factor greater than 1.5? PANalytical wants to encourage the use of X-ray technology for research. No restrictions are made regarding the brand of X-ray instruments used.

Stand to win
Trophy & cash prize of €5,000
Submit your paper by 1 Dec 2016

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PANalytical is the world's leading supplier of instrumentation and software for X-ray diffraction (XRD) and X-ray fluorescence spectrometry (XRF). Our products are widely used for the analysis and characterization of materials ranging from cement, metals, industrial minerals, glass, semiconductors, pharmaceutical solids and many more. Headquartered in the Netherlands, PANalytical has close to 70 years of experience in innovating and delivering precision X-ray instrumentation and software.

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PANalytical
get insight

Achieve synchrotron quality PDF data

in your lab with PANalytical's latest XRD GaliPIX^{3D} detector

PANalytical
get insight

Recent years have shown an increased interest in the study of nanocrystalline materials due to their specific properties for application in e.g. polymers, semiconductors and pharmaceuticals. Structural information about these materials is presented as broad, not well-defined features in a diffractogram. Analysis of nanomaterials and amorphous phases therefore requires a total scattering approach, including both Bragg peaks and diffuse scattering.

One of the most promising analytical methods used is atomic pair distribution function (PDF) analysis. Since the method requires short wavelengths to obtain high resolution in real space (well defined interatomic distances), often the measurements are performed at synchrotron facilities, making use of both the high photon energies and the high photon flux that these facilities offer.

PANalytical has been pioneering the implementation of PDF measurements on laboratory systems [1]. Now, by adding the high efficiency of the new GaliPIX^{3D} detector we have managed to obtain data approaching synchrotron quality.

Experimental

X-ray diffraction experiments were performed on a PANalytical Empyrean diffractometer equipped with an X-ray source with a molybdenum anode, delivering Kα radiation with a wavelength of 0.0709 nm, a focusing mirror, a capillary spinner, and the new GaliPIX^{3D} detector. This detector has an efficiency of ~100% for Mo radiation, thanks to the CdTe sensor used to detect the X-ray photons. The sample used for the investigation is nano-spinel ZnAl 2-3 nm size. Data on the same sample were also collected at the ID31 and at ID11 beam lines at the ESRF in Grenoble, France.

Results/explanation

The data obtained at ID31, ID11 and using the Empyrean diffractometer are shown in Figure 2. The limitation in Q range to ~17 Å^{-1} with the Empyrean is due to the chosen radiation. It is possible to extend the range to ~22 Å^{-1} by using a silver anode instead and the efficiency of the detector would still be ~100%. Data collection time for the dedicated PDF beamline ID11 is only 45 seconds. Data collection times for ID31 and the Empyrean, however, are quite comparable with 5 hours against 7.5 hours respectively. In Figure 3 the comparison between the PDF data is made. Most of the features are the same; the main difference is indicated with arrows, where a slightly better atomic resolution from the data measured at the synchrotron is visible. In Table 1 the refined structural values obtained from the PDF data are compared. The comparison shows that the results are in good agreement within the standard deviations.

The great advantages of being able to collect excellent data using a laboratory diffractometer are multiple, for example, slow processes can be studied (i.e. over days), there is higher stability of the X-ray source over time and, in practice, there is no need for proposal approvals or lengthy trips to large facilities.

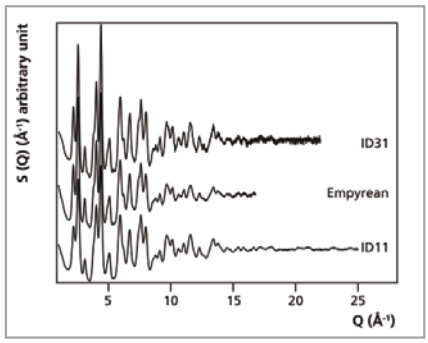

Figure 2. Structure function S(Q) comparison between two synchrotron beam lines and the Empyrean with GaliPIX^{3D} [2].

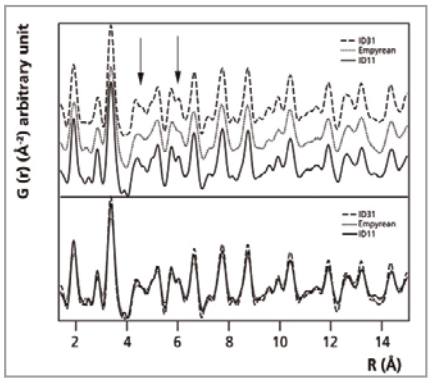

Figure 3. Comparison of the PDF obtained with data from the beam lines and the Empyrean diffractometer [2].

Table 1. Parameters obtained from the fit of PDF data obtained using the synchrotron beam lines and the Empyrean diffractometer [2]

	ID31	Empyrean	ID11
Cell (Å)	8.080(2)	8.088(4)	8.085(6)
U_{iso} Zn (Å^2)	0.0072(6)	0.012(1)	0.008(1)
U_{iso} Al (Å^2)	0.0049(8)	0.008(1)	0.005(2)
U_{iso} O (Å^2)	0.013(2)	0.017(3)	0.012(4)
Coordinate O	0.2617(7)	0.261(1)	0.262(2)
Delta 2	2.0(3)	2.1(3)	2.2(5)
Rw	0.215	0.207	0.207

Conclusion

PANalytical's latest XRD accessory, the GaliPIX^{3D} is able to collect data of quality approaching that obtained at a synchrotron and with data collection times comparable to those from non-dedicated PDF beam lines. The GaliPIX^{3D} may be used with PANalytical's multi-purpose diffratometer, the Empyrean XRD. Read more at **http://goo.gl/MZCPua**

Interested to find out more about the GaliPIX^{3D}?

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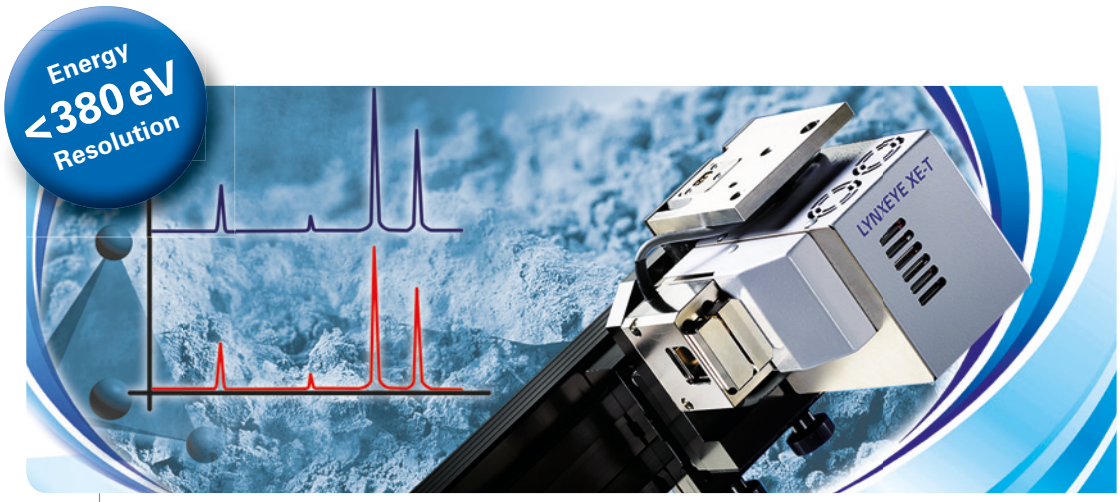
Bruker Australia / NZ News *March 2016*

In this update: • LYNXEYE XE-T



LYNXEYE XE-T

- High-Resolution Position Sensitive Detector with Superb Energy Resolution

The LYNXEYE XE-T is the next generation "Compound Silicon Strip" detector with superb energy resolution for ultrafast 0D, 1D, and 2D X-ray diffraction.

The LYNXEYE XE-T is particularly optimized to meet the increasing demands in X-ray diffraction of highest count rate capabilities, best angular resolution (peak widths), and best energy resolution.

The unique combination of sensor chip and front-end electronics as realized in the LYNXEYE XE-T makes it the highest performing detector on the market in terms of both data and manufacturing quality.

- High-speed data acquisition up to 450 times faster than a conventional point detector system
- Superior energy resolution better than 380 eV Cu radiation making Kß filters and secondary monochromators redundant
- Operation with all common characteristic X-ray emission lines (Cr, Co, Cu, Mo, and Ag radiation)
- Outstanding angular resolution (peak widths) and perfect line profile shapes
- Outstanding peak-to-background ratio for highest sensitivity and data quality
- No defective channels at delivery time – guaranteed

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LYNXEYE XE-T

Specimen fluorescence? You don't need any mirrors or monochromators!

Fundamentally, sample fluorescence is generated by the incident beam and can only be effectively reduced by choosing an appropriate wavelength. When fluorescence is present, it can only be filtered using suitable diffracted beam path components such as mirrors, monochromators, and energy discriminating / dispersive detectors.

Mirrors and monochromators are critical to alignment, and are usually tailored for a single wavelength. While mirrors always compromise the instrument resolution achievable in the traditional Bragg-Brentano geometry, diffracted beam monochromators are intensity killers.

Typical intensity losses range from more than 70% for point detectors up to more than 90% for one-dimensional detectors, compared to unfiltered radiation. At such losses, a one-dimensional detector loses all its advantages and operates at intensity levels close to traditional point detectors. Counting statistics are poor, resulting in noisy patterns and thus very poor lower limits of detection. Furthermore, present diffracted beam mirrors or monochromators cannot be used for two-dimensional diffraction, leaving an important gap for a huge range of applications where two-dimensional diffraction is of advantage.

This is demonstrated in Figure 1 for a natural hematite specimen (Fe-fluorescence with Cu-radiation) by comparing data acquired with the LYNXEYE XE-T and a scintillation counter with secondary monochromator. Figure 1 demonstrates the superb filtering of Fe fluorescence without any loss of peak intensity versus unfiltered radiation. Remarkable is the huge intensity gain by a factor of 450 by comparison to the secondary monochromator data.

No more Kß filter artefacts in your data!

There is almost no greater nuisance in diffraction data than artefacts introduced by the Kß filter, specifically absorption edges at the high energy tails of Kα diffraction peaks. Nevertheless, Kß filters are the most frequently used devices for monochromatization, as secondary monochromators do not represent a true alternative due to the very high intensity losses discussed earlier. As a consequence, absorption edges frequently prevent accurate profile fitting of peak tail regions and the background, and thus often represent a major part of the remaining misfit to the data, specifically for high intense peaks at low angles 2θ. With the LYNXEYE XE-T this is no longer the case.

LYNXEYE XE-T Lowest Background

Data acquisition at low angles smaller than ~20° 2θ requires some sophisticated beam conditioning to minimize instrument background (mostly air scatter), which otherwise is the most prominent contribution to the data. This is of particular concern at very low angles smaller than ~5° 2θ, the small angle X-ray scattering (SAXS) regime, where background suppression is of highest importance.

The unique Variable Active Detector Window™ feature of the LYNXEYE XE-T virtually eliminates low angle background scattering. This is achieved by the fully automatic, software-controlled change of the active detector window size as a function of 2θ: At 0° 2θ, the active detector window is closed, and gradually opens as the detector moves to higher angles 2θ, without any user-intervention. As a consequence, the use of beamstops becomes obsolete, and high quality data with virtually no instrument background can be collected starting at angles as low as 0.15° 2θ as shown in Figure 2.

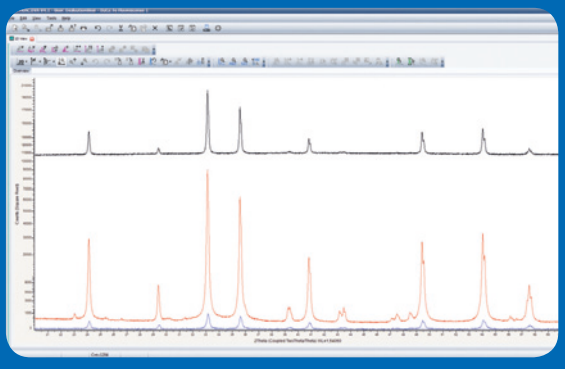

Figure 1: Unfiltered (black line) and filtered (red line) data demonstrating the superb filtering of fluorescence radiation by the LYNXEYE XE-T. The intensity gain over the secondary monochromator data (blue line) amounts to a factor of about 450.

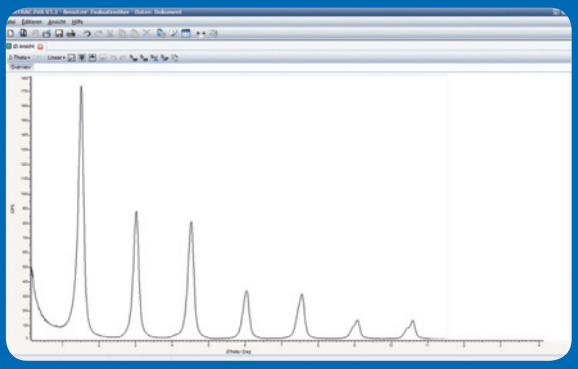

Figure 2: Low angle data collected on silver behenate. Thanks to the Motorized Anti-Scatter Screen and the Variable Active Detector Window™ of the LYNXEYE XE-T the instrument background is extremely low.

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

New Tube-Above WDXRF Spectrometer from Rigaku features Advanced Guidance System and Automatic Application Setup

March 1, 2016 – Tokyo, Japan. Rigaku Corporation is pleased to announce the introduction of the new Rigaku ZSX Primus IV tube-above sequential wavelength dispersive X- ray fluorescence spectrometer. The new instrument offers significantly improved functionality and performance, including the new "ZSX Guidance" software and error prevention functionality, enabling novice users to obtain precise analysis results.

WDXRF analysers are known for high detection sensitivity and spectral resolution offering non-destructive trace element analysis and are often employed by research institutions and for quality control. The ZSX Primus IV spectrometer maintains a tube-above configuration. Due to the tube-above optics, powder sample spills do not adversely affect the optical system. Since no protective film is required, intensity reduction due to film is avoided.

The system also includes a number of enhancements. A key new advantage is the ZSX Guidance software, now standard for the equipment. The software includes a quantitative application auto-configuration feature which automatically sets measurement conditions and various correction conditions once the user simply enters the standard value and sample information. An error prevention function, which can set an access level for each operator was also added.

The new Rigaku Primus IV WDXRF spectrometer.

The ZSX Primus IV system includes a dedicated analyzing crystal, resulting in a 30% improvement over conventional configurations – Significant, as measurement of boron is an important consideration in the manufacture of glass and ceramic materials, Light element sensitivity is also boosted by the system's APC (Auto Pressure Control), which regulates and stabilized the vacuum conditions.

During analysis, regions of interest can be located in enlarged images of the sample taken with a high-resolution camera. The r-θ sample stage can accurately be positioned to facilitate measurements with uniform sensitivity on these small areas.

Key Features

- Tube above optics - Contamination risk is minimized as sample spills to not affect the optical system. Intensity reduction is avoided since no protective film is required.
- ZSX Guidance software - Built-in XRF expertise handles sophisticated settings. Available application packages enable quick startup.
- High-speed analysis - Throughput has greatly been improved by high-speed sample transportation, goniometer drive, data processing and effective driving control. Measurement time has been reduced by 40 % for qualitative analysis and 20 % for quantitative analysis.
- Digital Multi-channel Analyser (D-MCA) - D-MCA X-ray counting system delivers improved precision with higher counting linearity.
- Point and mapping analysis - Regions of interest can be located in enlarged images of the sample taken with a high-resolution Camera.. The r-θ sample stage can then be positioned accurately to make measurements with uniform sensitivity on these small areas.
- Gas-sealed proportional counter (S-PC LE) for light elements – alternative to gas flow proportional counter (F-PC) is optional for sites where P-10 gas is not available.

Rigaku WDXRF products are distributed in Australia and New Zealand exclusively by AXT. For more information about other Rigaku product lines and XRF related products please visit www.axt.com.au.

AXT to Supply Two Rigaku SmartLab Diffractometers to Queensland University of Technology

March 21, 2016 – Sydney, Australia. AXT is proud to announce that they will be supplying Queensland University of Technology (QUT) with two Rigaku SmartLab X-ray diffractometers. These systems are destined for the Central Analytical Research Facility (CARF) where they will cater for research, routine and commercial activities, serving both internal and external clients.

The two SmartLab diffractometers are based on the 3kW sealed tube design. By choosing two systems sharing the same architecture that will be located side-by-side, their versatility can be fully appreciated with both systems able to share attachments, thus enabling operators to run a vast range of experiments.

The systems have been purchased with slightly different applications in mind i.e. research and routine measurements. The research focussed system has been ordered with a host of attachments allowing them to perform a range of in situ analyses such as high temperature (1200°C) XRD under controlled environments, cryogenic measurements and thin film and fibre/capillary analysis. The routine measurement SmartLab includes a 48 position sample changer, enabling large numbers of experiments to be performed unattended.

Both SmartLabs have been specified with HyPix 3000 Detectors that utilise Rigaku's latest generation Hybrid Pixel Array Detector (HPAD) technology. These high-speed detectors that produce no noise will reduce measurement time and increase sample throughput rates and productivity. Furthermore, their integrated capability which can switch between 0D, 1D and 2D analytical modes at the click of a button was a key factor in the selection of the SmartLab diffractometers with competitive systems needing multiple detectors to do the same job.

When asked about the selection of the Rigaku SmartLabs, CARF's Project Manager Tony Raftery replied, "QUT has a large research community who will benefit from this acquisition. The community has varying backgrounds and research expertise, ranging from Research Project Leaders through to higher degree research students and project-directed undergraduates. One key factor that made the decision easier was ease-of-use and Rigaku's Guidance software. This Guidance software helps users design experiments and steps them through configuration changes as well as performing an automated alignment and measurement sequence, minimising the input required from CARF technical staff."

Tony went on to say, "QUT has numerous research groups who will be lining up to use these instruments when they arrive. Some of their projects and interests include:

- Characterisation of energy materials for solar cells
- Characterisation of energy materials for advanced batteries and supercapacitors
- Characterisation of high performance materials with extreme hardness and refractory properties.
- Thermal properties investigations, particularly high temperature behaviour and corrosion."

Richard Trett, AXT's Managing Director said, "Tony and the team at QUT obviously identified the technical advantages of the Rigaku SmartLab in particular, speed, flexibility, simplicity and student-proof design and we look forward to supporting them both in the short term and into the future."

Rigaku are a Japanese-based manufacturer of X-ray based analytical instruments suited to a range of applications such as materials science, life science and non-destructive testing. They are world leaders in X-ray spectrometry, diffraction, and optics, as well as small molecule and protein crystallography and semiconductor metrology. AXT represent Rigaku in Australia and New Zealand. For more information about other Rigaku product lines and XRF related products please visit www.axt.com.au.

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BEAD ONE[R]

Bench-top Fusion System

IMP is proud to announce the release of the brand new BEAD ONE[R]. The BEAD ONE[R] is a bench-top fusion system that utilizes a resistance furnace capable of achieving temperatures of up to 1250 °C.

The furnace chamber is protected by ceramics to guarantee a long service life, even if corrosive samples are being prepared.

Specialized software features allow the operator to conduct complex fusion procedures, such as ensuring the complete oxidation of samples prior to fusion.

The BEAD ONE[R] is especially designed to meet the requirement of laboratories with small to medium sample loads.

Features

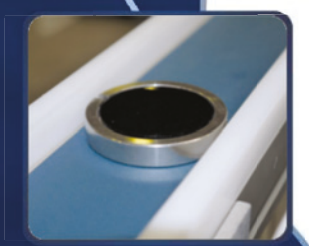
- Heating element protection
- Preheating & Switch-off function
- Adjustable dish & bead cooling
- Magazine extension available

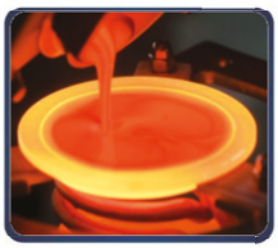
Software

- Predefined method library
- Up to 16 fusion methods
- Quick-start front panel operation
- Ethernet connection (PC/tablet)
- Password protected menu

Technical data

- Dimensions: 750 x 610 x 590 mm
- Weight: 75 kg
- Temperature range: 300° - 1250°C
- Voltage: 230 V, 50 Hz
- Power consumption: 1,3 KW
- No compressed air required
- External switch cabinet





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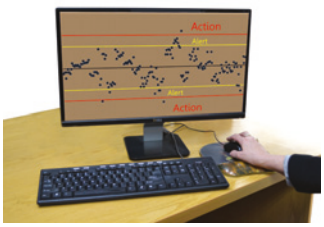

Why Use RRM's XRF Standards?

- RRM provides a comprehensive service to end users of XRF instruments; from handheld, desktop and more advanced laboratory spetrometers.
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- We ensure that all individual CRM's samples supplied to our clients are as representative as possible to the analysed batch.
- Our batches are prepared and sized to the specified PSD and then divided into sub-samples through a series of specially designed Rotary Sample Dividers (RSD's).
- RRM carries out the preparation and homogeneity testing of pressed pellets and fused beads, from materials provided by you or sourced by us.
- We provide assistance with method validation and training in safe and effective use of hand held analysers.

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