AXAA

Australian X-ray Analytical Association

Newsletter Issue 3 2022

President's Address

Dear AXAA members and friends,

Thank you for your continued support and attention in 2022. We recently held our AGM (online) and it was wonderful to see so many familiar faces in the space. We farewelled Sally Birch from the role of Treasurer after nearly 6 years in which she has kept us impeccably organised through a particularly tumultuous time, and leaves us in excellent shape going forward. Thank you Sally!

I am delighted to welcome four members to the National Council, Tony Wang (Treasurer), Ruoming Tian (Council), Mark Paskevicius (Council) and Rodney Clapp (Council). Our new council increases our representation of the diverse AXAA membership across the country, across expertise, and across industry and academia.

Our final newsletter for 2022 is a special holiday edition featuring some updates from the X-ray Absorption Spectroscopy at the Australian Synchrotron which has an exciting new fast scan capability and a limited time opportunity for users to run High Energy Resolution X-ray Fluorescence Detection (HERFD), an event report of the recent XRD workshop hosted by AMCF, and a healthy serve of community news items.

As we approach the end of the year at full speed, I'm relieved at the prospect of some downtime,and in that spirit I'm going to keep this summary short and sweet, and simply wish you all a wonderful new year!

See you in 2023!

Jessica Hamilton
AXAA President

WSU TOPAS Workshop

Daniel J. Fanna
Scientist, Instrumentation Specialist & AXAA NSW Council Member
AMCF, WSU

The Advanced Materials Characterisation Facility (AMCF) at Western Sydney University (WSU) was delighted to host a 3-day TOPAS workshop run by AXAA life member and preeminent expert Ian Madsen. The 3-day workshop was conducted at the WSU Parramatta South campus from the 21st to 23rd of November and attracted attendees from WSU, several other universities, ANSTO and Australian industry giants in mining and construction.

Ian's workshop was a level 1 TOPAS beginner course aimed at intermediate XRD users looking to take the next step in their data processing. Day 1 of the workshop opened with Ian presenting an introduction to TOPAS and this then flowed into other topics such as the information content of a powder diffraction pattern, peak profile fitting, crystallite size/strain determination and specimen-preparation. Attendees were then guided through several tutorials relating to single peak and whole pattern convolutions, whole pattern profile fitting models as well as crystallite size and strain analysis.

The second day of the workshop was focused on quantitative phase analysis (QPA) of XRD patterns and guided users through several different QPA strategies including Rietveld, PONKCS as well as strategies for quantifying the amorphous content of samples and more. Ian also enlightened users to some tips, tricks and pitfalls in QPA including micro-absorption, errors in sample preparation and modelling, and what to look out for when extracting crystal structures from databases.

On the final day attendees were introduced to TOPAS's launch mode and jEdit, a free Java-based text editor. Here Ian guided attendees through

Fig 1. Workshop attendees working their way through TOPAS tutorials under the guidance of Ian Madsen.

setting up input files as well as showing the power of launch mode for processing batch data sets of in -situ non-ambient XRD data. The workshop finished up with an engaging Q&A session where Ian took the time to address queries and provide insightful solutions to specific challenges the audience had previously encountered during their own XRD analysis.

Thank you, Ian, for taking the time to run the course. We cannot express enough how invaluable this was for our local academic researchers, industry partners and also ourselves. A special mention also needs to be given to Neil Hughes from Bruker for supporting the event, which was co-organised by Bruker Pty. Ltd and the AMCF.

100% efficient: An update from the ANSTO XAS Beamline

Dr Bernt Johannessen
Senior Scientist @ ANSTO

Imagine a conveyor belt carrying Christmas presents with an elf by its side. As each present passes the elf's job is to pick up that present for inspection and then return it to the belt after (maybe discarding some faulty ones, but how that fits with our analogue requires a tedious explanation[1]). In this scenario, each time a present passes the elf the conveyor belt stops – entirely – for the inspection to take place. Once completed, the belt carries on until the next present comes along. Like at Santa's workshop, this is the way energy scanning at the XAS Beamline has operated for well over a decade; an energy *step-scan* of typically 600 points or so, proceed step-by-step. At each step the energy (monochromator Bragg angle) decelerates and comes to a halt, a sample is exposed to an X-ray beam, data is collected and written to file, after which the energy accelerates on to the next point.

Repeat. 600 times.

There is a natural overhead to this process, specifically all that time used *not collecting* the actual data that ends up in file otherwise known as

[1]The keen XAS practitioner might think of monochromator glitches.

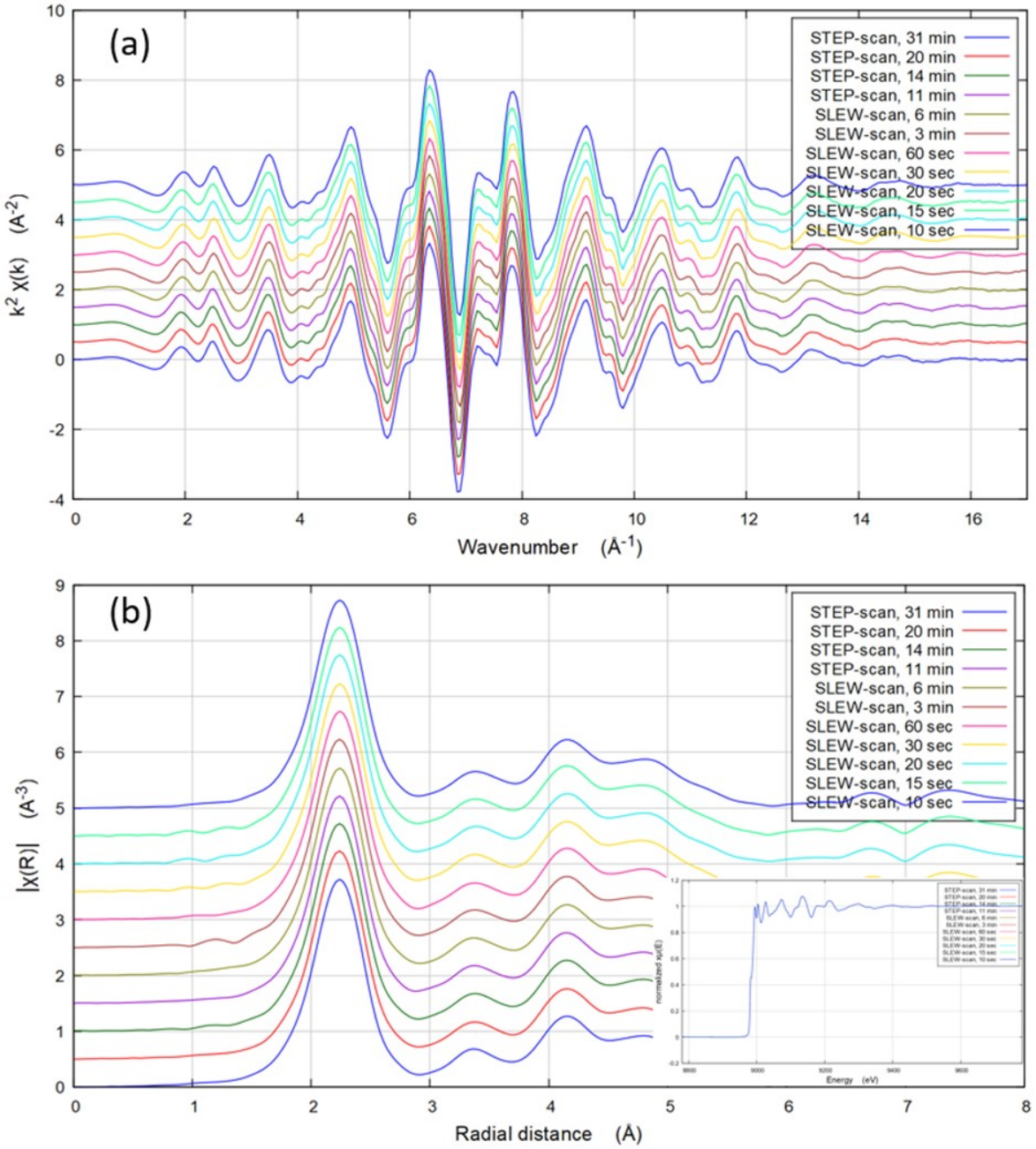

Fig. 1: XAS spectra for bulk metallic Cu measured both using step-scan (31, 20, 14, and 11 minutes) and using slew-scan (6, and 3 minutes, and 60, 30, 20, 15, and 10 seconds), with (a) the post-edge XAS region, and (b) the corresponding Fourier-transformed signal. The inset shows the 11 scans overlayed in energy space

the XAS spectrum. In our case that accounts for a second or so per data point, accumulating to about 10 minutes per scan. This is a fixed overhead that exists regardless of how short the collection time (beam exposure) is per data point. This works reliably for us and indeed for many XAS beamlines around the world. However, business as usual no more.

So, let's imagine another scenario in which the conveyor belt does not stop. No need to speed it up, just let it be slow enough for the elf to inspect each present as it travels along on its merry way, not stopping until the entire conga line has been inspected. This represents what we are doing at the XAS Beamline in 2023 and beyond[2]. Helped by a Zebra/Xspress on the hardware side, and Ophyd/Bluesky on the software side, we run energy *slew-*

[2]Be pleased to know that more beamlines will follow, including the MEX Beamlines.

scans. The scan starts, the energy changes, often with a variable speed, and only stops when the entire scan is completed. Along the way, data is collected dynamically on-the-fly. The result is a scan with no overheads – at all – in other words *100% efficient*.

Figure 1 shows a series of spectra, some of which are energy step-scans (31 down to 11 minutes), others energy slew-scans (6 minutes down to 10 seconds). Clearly, there is little that differentiates these measurements, certainly to the naked eye, and indeed the majority of our user community would be able to extract information with sufficient detail from even the fastest scan. Turning back to our analogue, incredibly, it appears

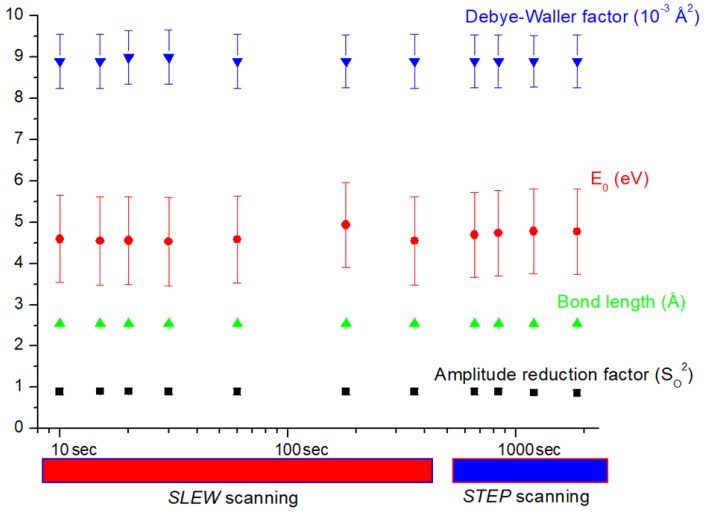

Fig. 2: XAS data parameters extracted from the scans shown in Figure 1 and plotted as a function of scan time. Special thanks to Simon James, MEX Beamline, for doing the data analysis

the conveyor belt can be very fast indeed, with the elf retaining the capacity to accurately inspect the presents. The footnote, as always with these types of proof-of-concept tests, is that this is an ideal sample (bulk metallic Cu foil), but it does show the strength and promise of the approach.

Figure 2 shows a set of common parameters, as a function of scan time, that a researcher will typically extract from their data. Clearly, this validates statements already made regarding Figure 1, and pertinently it is pleasing to realise that the step scan of about 11 minutes duration contains the same quantity and quality of data as the slew scan of about 60 seconds duration (recall how the overhead in a typical step scan is 10 minutes).

In conclusion, I will make two further reflections on slew-scanning:

Efficiency is no longer how fast a scan is. The entirety of time spent on a scan (to achieve a sufficient signal-to-noise for example) is time spent efficiently. There is no time wasted when there is no overhead. Instead, efficiency is what happens between scans, and for those in our user community it is for you to streamline your approach to your beamtime.

A new approach opens for new opportunities, be it higher sample throughput, new and exciting ways to measure samples *in situ*, or radiation sensitive samples that naturally benefit from short scan durations. The choice and scientific endeavour belongs to our community.

Thank you to Letizia Sammut, Senior Scientific Software Engineer, and her team of Scientific Computing champions, Ben Baldwinson, Senior Control Systems Engineer, and the wonderful team of scientists at the XAS Beamline.

Crystal Spectrometer available for a limited time (rounds 2, 3 in 2023) at the XAS beamline at the Australian Synchrotron

We are pleased to announce that at the next call for beamline proposals, the X-ray Absorption Spectroscopy (XAS) beamline at the Australian Synchrotron will also consider applications to utilise the crystal spectrometer which is currently located at the beamline in experimental Hutch C. This is a

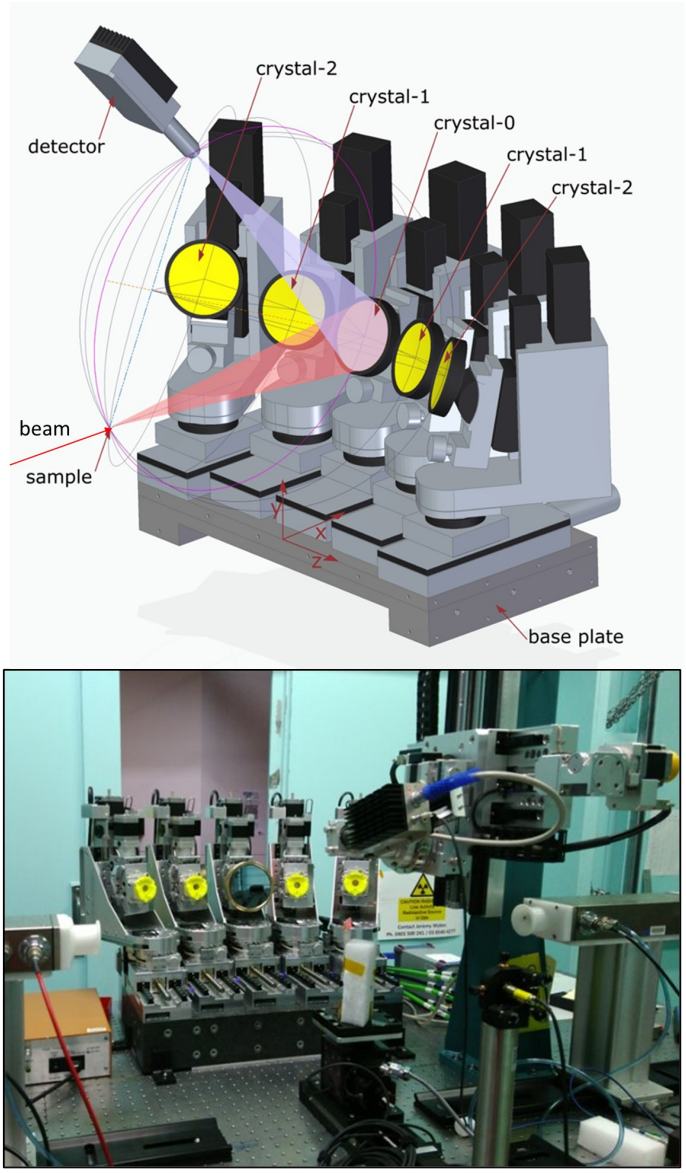

Fig. 1: (Top) Schematic of crystal spectrometer and (bottom) photo of the MEX crystal spectrometer, currently hosted at the XAS beamline, Hutch C.

limited time opportunity with the spectrometer slated for a return to its owner – the Medium Energy XAS (MEX-1) Beamline – towards the end of 2023.

The crystal spectrometer enables High Energy Resolution Fluorescence Detection (HERFD). This is advantageous in cases where 'regular' XAS spectra are difficult to distinguish because they are relatively featureless or don't show a large edge shift between different chemical species.

In Figure 2, we see an example of two polymorphs of mercury sulfide (HgS). Cinnabar (α-HgS) and metacinnabar (β-HgS) appear relatively similar

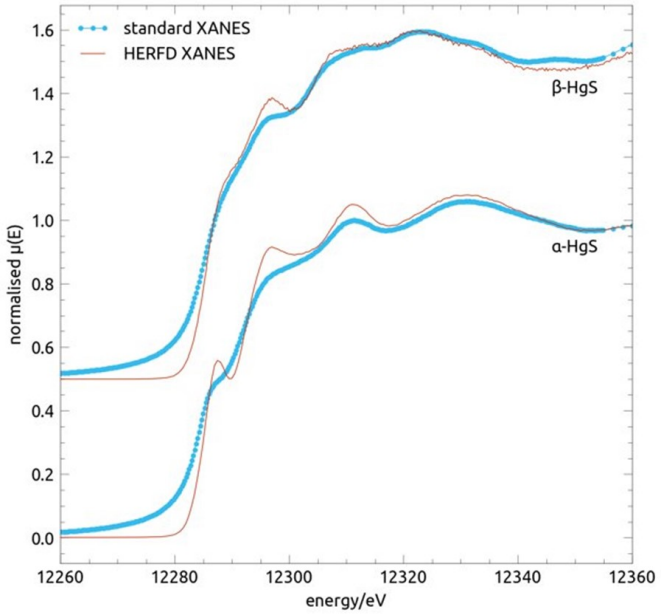

Fig. 2. Preliminary data from the spectrometer in comparison to standard XANES of the mercury edge, on samples containing approximately 2000 ppm Hg. The β-HgS scan was a single XANES scan collected over 30 min, while the α-HgS is an average of five 30 min scans

in regular XAS analyses shown in the blue trace, which introduces high uncertainties in speciation analyses, particularly when trying to tease apart and quantify mixtures using linear combination fitting.

In 'regular' fluorescence XAS measurements, the sample is irradiated with X-rays, and the emitted fluorescence signal is collected by a fluorescence detector. The detected signal includes fluorescence from all the absorbing elements within the sample, as well as a substantial amount of scatter.

When a crystal spectrometer is used, the emitted fluorescence from the sample is diffracted by a set of specially cut crystals, such that only the wavelength of the element you're interested in (e.g. mercury) will pass through the crystal and to the detector. As such, the detector does not receive background fluorescence from any other element within the sample, or scatter from the beam itself. The resulting fluorescence spectra has essentially zero background, and due to the high energy resolution of the crystals, spectral features can be greatly enhanced, such that formerly indistinguishable spectra become, well.. distinguishable!

If you're convinced that this instrument could benefit you, a few things to consider are firstly, by

design this configuration sacrifices most of the X-ray flux in return for high resolution. As such, it can take longer to collect spectra. Figure 2 shows in the thin red traces one 30 minute scan for β-HgS, and the average of five 30 minute scans for α-HgS. There is a clear benefit to long data collection time.

Secondly, this is a new instrument designed for the MEX beamline, and as such we do not have a full set of crystals to enable analysis of all elements usually available at the XAS beamline. Prior *to submission of any spectrometer proposal, y*ou must contact us on as-xas@ansto.gov.au to discuss your specific requirements.

We are also excited about the ramping up of operations at the MEX beamline(s) in 2023! Proposals are also open for this beamline, so please check the website if you'd like to submit. As always, please see our websites and in particular the beamline updates page before submission.

We thank you for your continued support and collaborations throughout 2022 and we look forward to a productive 2023!

All the best for the holidays,
Your XAS Beamline Team

26TH CONGRESS AND GENERAL ASSEMBLY OF THE INTERNATIONAL UNION OF CRYSTALLOGRAPHY

22-29 August 2023

Melbourne Convention and Exhibition Centre

The IUCr Congress and General Assembly is held every 3 years around the world and it's coming to Melbourne in 2023! Don't miss this excellent chance to participate in THE major scientific meeting for crystallographers world wide.

The abstract submission deadline has recently been extended until February 21, 2023
Submit your abstract here

We would like to encourage interested AXAA members to submit an abstract to one of the 2023 IUCr Microsymposia "**What Every Crystallographer Should Know About Powder Diffraction**", or "**Industrial and Engineering Powder Diffraction**", which are sponsored by the Commission on Powder Diffraction.

The first microsymposium is chaired by David Bish, Cora Lind-Kovacs, and Helen Brand. Two excellent invited speakers, Helen Maynard-Casely and Matthew Rowles, will highlight the session. The second microsymposium is chaired by Anna Paradowska and Matthew Rowles. Jim Kaduk will be an invited speaker.

Hope to see some of you in Melbourne!

The abstract submission deadline has recently been extended until February 21, 2023
Submit your abstract here

ACMM 27

29 January - 2 February 2023
Perth, WA

The 27th Australian Conference on Microscopy and Microanalysis is committed to being a face-to-face event that will bring together colleagues across the life and physical sciences, to work towards new visions and goals in microscopy. We take pride in putting together a strong program for the conference based on advances in electron and light microscopy and microscopy-enabled research in the life sciences and physical sciences. The conference will include oral and poster presentation sessions, pre-conference workshops, Special Interest Group-focussed sessions, trade displays and social events.

High Purity Alumina for QXRD

Independent Mineral Standards (IMS) is a specialist producer of Certified Reference Materials, (including mineral reference materials). Driven by data and world leading expertise, we service the global mining industry.

Based in Perth, Western Australia, we are internationally recognised for providing 100% impartial, high-quality and affordable standards.

IMS was the first dedicated CRM producer in Australia to achieve ISO17034 accreditation. We are also SAI Global Assurance ISO 9001:2015 certified.

Introducing IMS135, a high purity alpha-alumina powder used in Quantitative X-ray Diffraction (QXRD) analysis.

Manufactured and certified to replace NIST SRM676a stocks.

- High-purity alpha-alumina powder.
- Equally high corundum concentration level and near-identical particle size & trace level impurities as NIST SRM676a.
- Certified using ISO Guide 35 methodology with NIST SRM676a as a reference.
- The certificate includes the percentage alpha-alumina content and an estimate of the uncertainty, alongside a detailed trace element analysis by XRF and laser ablation ICP-MS.

IMS Independent Mineral Standards

www.imstandards.com.au
+61 8 61557616
enquiries@imstandards.com.au

FIND OUT MORE

Scan QR code for direct link to **IMS135 Certificate**

For sales and enquiries please email enquiries@imstandards.com.au or see website.

Malvern Panalytical's "Ask an Expert" webinar series

Malvern Panalytical a spectris company

X-ray diffraction is one of the most powerful techniques that reveals information about the crystal structure of a material. Our webinars are tailored to anyone in academia who needs help on experimental set up to sample preparation and sample handling and finally to data interpretation. Got a question on how to achieve better XRD data quality? Access our free webinar on demand series over here

Improve your XRD analysis: Guidelines for getting good quality PXRD pattern for pharmaceutical samples
Watch webinar: https://www.malvernpanalytical.com/en/learn/events-and-training/webinars/W290422-PXRD-pharmaceutical-samples

Ask an Expert! Getting the best out of non-ambient measurements
Watch on demand: https://www.malvernpanalytical.com/en/learn/events-and-training/webinars/W220421XRDAskanExpert

Check for microstructural defects in additive manufacturing using XRD
Watch on demand: https://www.malvernpanalytical.com/en/learn/events-and-training/webinars/W20220526AMXRD

ROWE SCIENTIFIC PTY LTD www.rowe.com.au

For accuracy and professionalism

Providing laboratory supplies to the scientific community across Australia since 1987.

We are proudly a 100% Australian owned company.

Quality System
Quality Endorsed Company
ISO 9001:2015
LIC 10372
SAI Global

XRF - XRD Sample Preparation

Rowe Scientific are now exclusively supplying the SOMAR brand of Australian made XRF pellet cups.

We have purchased the assets of SOMAR Australia and incorporated their pellet cup manufacturing into our Perth Facility.

XRF Liquid Cups

FREE SAMPLES
CONTACT US TODAY

These cups allow the analysis of solutions by XRF, and fit all common makes of XRF instruments, including X-Unique II, PW2400, PW2404, Axios, and many PANalytical instruments.

- Free trial samples available
- Very cost competitive.
- Avoids cross contamination between samples - cups are disposable.
- Made from polypropylene - chemically inert.
- Packaged under clean room conditions - free from silica and other airborne particulates.

For ordering information, download the XRF - XRD brochure by visiting our website

www.rowe.com.au

To find out more or to acquire your FREE samples, call your local Rowe Scientific Pty Ltd office

SCAN TO DOWNLOAD XRF-XRD BROCHURE

https://goo.gl/1kCVUw

ROWE SCIENTIFIC PTY. LTD. www.rowe.com.au

South Australia & NT
Ph: (08) 8186 0523
rowesa@rowe.com.au

Queensland
Ph: (07) 3376 9411
roweqld@rowe.com.au

Victoria & Tasmania
Ph: (03) 9701 7077
rowevic@rowe.com.au

Western Australia
Ph: (08) 9302 1911
rowewa@rowe.com.au

New South Wales
Ph: (02) 9603 1205
rowensw@rowe.com.au

Quality System
Quality Endorsed Company
ISO 9001:2015
LIC 10372
SAI Global

AXAA Website and Contacts

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

NATIONAL COUNCIL PRESIDENT:

Jessica Hamilton
Australian Synchrotron (ANSTO),
800 Blackburn Road,
Clayton, VIC 3168
Telephone: (03) 8540 4297
e-mail: hamiltoj@ansto.gov.au

NATIONAL COUNCIL VICE PRESIDENT:

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

NATIONAL COUNCIL SECRETARY:

Anita D'Angelo
Australian Synchrotron (ANSTO),
800 Blackburn Road,
Clayton, VIC 3168
Telephone: (03) 8540 5397
e-mail: anitad@ansto.gov.au

NATIONAL COUNCIL TREASURER:

Tony Wang
Level 6, P Block
Gardens Point Campus
Queensland University of Technology
Brisbane, QLD, 4000
Telephone: (07) 3138 1904
e-mail: tony.wang@qut.edu.au

NATIONAL COUNCIL COMMUNICATIONS EDITOR:

Valerie Mitchell
Australian Synchrotron (ANSTO),
800 Blackburn Road
Clayton, VIC 3168
Telephone: (03) 8540 4297
e-mail: mitchelv@ansto.gov.au

NATIONAL COUNCIL MEMBERS:

Matthew Rowles (Intertek, WA)
Brianna Ganly (CSIRO, NSW)
Daniel Fanna (WestSyd, NSW)
Ruoming Tian (UNSW, NSW)
Mark Paskevicius (Curtin, WA)
Rodney Clapp (Diffraction Technology, Vic)

AXAA Membership

All registered participants of the AXAA-2017 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 send the membership form from the AXAA website, and a short statement about how they intend to contribute to the organisation, to the National Council Secretary Anita D'Angelo.

AXAA Resource Centre

There are a range of resources available on the AXAA website, including video recordings of the two Public Lectures at AXAA-2017, tips for Rietveld Analysis, Clay Analysis, XRF tips, and more. We welcome further contributions to our Resource Centre.

Next AXAA Newsletter

The next issue of the AXAA Newsletter will be distributed in April 2023. Please feel free to send contributions for the newsletter to Valerie Mitchell at *ausxray@gmail.com*. Any comments or feedback about the Newsletter are welcome.

A Day in the Life of an X-ray / Neutron Scientist

We are seeking posts for our 'Day in the Life' series. If you'd like to contribute, or know someone who might be interested, please contact National Council