Australian X-ray Analytical Association

Newsletter Issue 3 2020

President's Address

Dear AXAA members,

For those who don't yet know me, I had better introduce myself! I first became involved with AXAA as a student at Monash Uni, attending one of AXAA's student seminar days. This was the first time I ever presented my research, and it was a great introduction to the experience, with friendly people, free pizza, and prizes! I was lucky enough to win (along with Anita, our new AXAA Secretary) a bursary to attend the AXAA-2014 conference in Perth. A highlight for me at that conference was the talk on mineralogy results from the Martian rover Curiosity, particularly because we had a version of the same portable XRD instrument used within Curiosity in our lab at the time. I remained involved with AXAA through the student events, and was invited to join the AXAA-2017 conference organising committee, before being elected to the National Council in the role of Communications Editor which I have held for the past three years. I feel very grateful for the opportunities and friends I've gained through AXAA, and I'm humbled to be elected President of this wonderful community.

Our AGM was held on 2nd Oct in an online format for the first time. A big thanks to Mark for coordinating this and ensuring a successful meeting, and to all who were able to attend. I would like to sincerely thank every member of the outgoing National Council for their contributions to AXAA over the past three years. Over this time AXAA has delivered fantastic events for an increasingly multi-disciplinary membership, improved diversity in representation, and supported options for our members to access AXAA events. If we can only keep up the good work, AXAA will have a very bright future.

The incoming National Council are myself as President, Nathan Webster as Vice President, Anita D'Angelo as Secretary, Sally Birch as Treasurer, Valerie Mitchell as Communications Editor, and Brianna Ganly, Matthew Rowles, and Daniel Fanna as Council Members. Please echo my warm welcome to all!

You will all know that AXAA had a challenging 2020, as I'm sure many of our members have personally and professionally too. As Nathan wrote in the August issue, AXAA is very lucky to remain in a strong position after the cancellation of our national conference. Going forward, we will need to adapt to new ways of operating, and I feel sure that that the online connectedness we have grown accustomed to will remain a prominent feature of our lives as well as AXAA activities in the future. I feel very positive about this, as the accessibility of the online world will allow us to connect across the nation far more easily and often, to share science and stories. However, with state borders re-connecting very recently I do hope that in-person events will also be possible next year, as I'm sure many of us are very keen to connect again in real life!

Finally, I would like to share congratulations to AXAA members Mark Raven, Peter Self, Nathan Webster, Rong Fan, and Rodrigo Gomez-Comacho for their 2nd place in this year's Reynold's Cup – a truly exceptional achievement!

Jessica Hamilton

AXAA President

CSIRO Team Places Second in 2020 Reynolds Cup Competition

A CSIRO team led by Mark Raven and consisting of Peter Self, Nathan Webster, Rong Fan and Rodrigo Gomez-Comacho have achieved second place in the 2020 Reynolds Cup Competition. The biennial global competition is for accuracy in quantitative mineral analysis, with emphasis on clay mineralogy, with three samples containing mineral mixtures commonly found in clay bearing rocks or soils to be analysed by each participating laboratory in a blind round robin format. This affords participants an opportunity to test their methods in sample preparation, data collection and analysis and thereby identify both strengths and weaknesses and how they may improve. Mark and Peter are previous winners of the competition, in 2010, and also achieved second place in 2016.

The samples for the 2020 Reynolds Cup were: a hydrothermally altered shale, a muddy limestone, and a Martian volcaniclastic sediment/soil analogue. Samples were analysed by X-ray diffraction and X-ray fluorescence after careful and rigorous preparation, with the XRD data analysed using a combination of SIROQUANT and TOPAS software. Each sample presented its own distinct challenges, such as quantification of amorphous material in two of the three samples, the identification of multiple plagioclase and K-feldspar minerals in the Martian analogue sample, the detective work necessary to successfully identify halloysite rather than serpentine and the presence of an illite/smectite interstratified clay in the muddy limestone sample.

A CSIRO team took second place in the 2020 Reynolds Cup. From left to right, Rodrigo Gomez-Camacho, Peter Self, Mark Raven, and Rong Fan.

The team's achievement was announced at the recent 57th Annual (virtual) Meeting of the Clay Mineral Society, Richland, Washington State, held from October 18 to 23.

Teaching the basics of Rietveld refinement

Dr. Matthew Rowles, Curtin University

Powder diffraction can be used to examine many aspects of materials, including phase identification, phase quantification, cell parameter determination, and crystal structure analysis. A key protocol in the determination of these analyses is the Rietveld refinement method.

Over the past 16 years, Professors John SO Evans and Ivana Radosavljević Evans have co-run a residential Powder Diffraction and Rietveld Refinement School. This School teaches graduate students (and others) from a wide range of disciplines with very different backgrounds and prior training, the finer points of powder diffraction analysis. During this time, they identified that many students treated the refinement process as a tick-the-box exercise, where the options presented by the software were turned on and off in some fashion to make the refinement "better", with little regard to the physical or chemical reasonableness of the resultant model; this poor understanding of the process led to a poor understanding of the outcome.

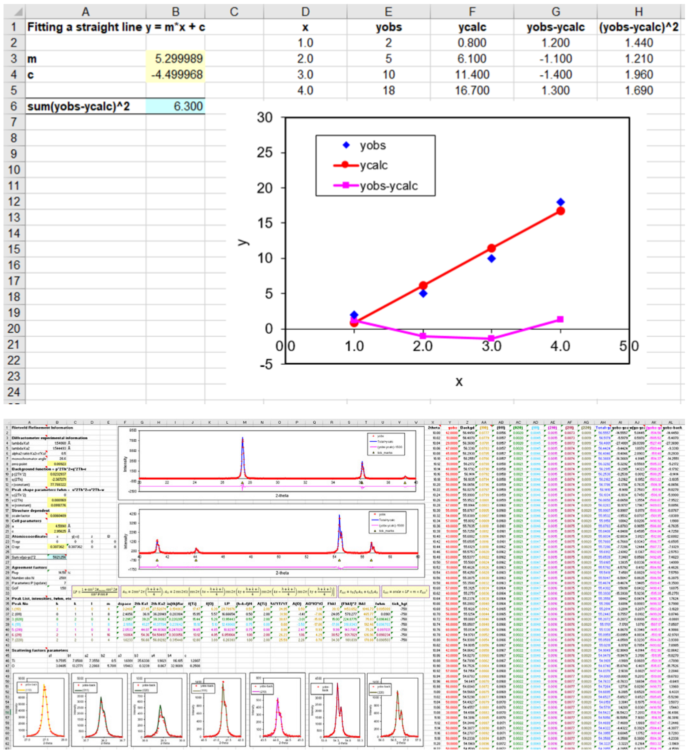

Figure 1. The teaching tools developed by Evans and Evans begins with a simple least-squares fit (top) and expands to a full Rietveld refinement (bottom).

To help remedy this situation, they developed an Excel spreadsheet to teach basic least-squares fitting (Figure 1, top), expanding up to a (simple) full Rietveld refinement (Figure 1, bottom). The spreadsheet starts from simple straight lines, progressing to single peaks, through to simulated and real diffraction data. The models increase in complexity from individual independent peaks, semi-constrained peaks (Pawley/Le Bail), to fully constrained peaks (Rietveld). Each step builds complexity and adds to the previous learning outcomes, allowing students to experiment with initial conditions, peak shapes, and parameters constraints in a highly flexible and transparent environment. Models are minimised through the use of Excel's in-built Solver, which allows for non-linear functions. The educational package including the excel spreadsheet is available for download in a recent publication in the Journal of Chemical Education [1].

The choice of an Excel spreadsheet was driven by pragmatism. Assuming a common knowledge in a single conventional programming language in a large number of students from chemistry, archaeology, earth sciences, engineering, materials, pharmacy, physics, and industry, would result in disappointment. Also, for better or worse, Excel is a ubiquitous piece of stand-alone software, with power and flexibility in visualisation, calculation, formulae, and non-linear function minimisation. Yes, if you were to write a piece of dedicated data analysis software, Excel shouldn't be your first choice, but the value of being able to see all the data, formulae, and calculations, shouldn't be underestimated.

Following this grounding in least-squares fitting and Rietveld basics, students then moved on to Rietveld-specific refinement software for further learning. Student feedback revealed that Excel-based exercises led to a better understanding of what Rietveld-specific software does and cultivated a more critical approach to their use. Furthermore, the data analysis philosophy is widely applicable in non-diffraction applications.

Undergraduate and postgraduate coordinators teaching crystallography, diffraction, or even just data analysis, would be well-served by investigating how this (or a similar) spreadsheet could be integrated into their courses.

References

1. J.S.O. Evans and I.R. Evans. Journal of Chemical Education, 2020, doi: 10.1021/acs.jchemed.0c01016

A New tool for studying minerals leaching – in-situ PXRD under flow-through condition

Associate Professor Fang Xia, Murdoch University

Introduction

Studying the mechanism and kinetics of minerals leaching is important for further improvement of leaching efficiency [1-3]. Leaching is commonly studied by ex-situ techniques, which involve the recovery of leached products by cooling and filtration and subsequent characterization [2-4]. Ex-situ techniques provide valuable information, but the results may not reflect the true mechanism of leaching because samples may have changed during recovery. On the other hand, in-situ techniques characterize reaction products under leaching conditions [5, 6], avoiding the problems experienced by ex-situ techniques. Hence, in-situ techniques are important complementary tools to ex-situ techniques for a complete understanding of leaching.

For in-situ powder X-ray diffraction (PXRD) studies of mineral leaching, flow-through reaction cells are particularly useful for achieving a wide range of solid/solution ratios to mimic various types of leaching conditions. For this purpose, we have designed a flow-through cell setup and demonstrated its in-situ PXRD capability for minerals leaching at the Australian Synchrotron.

The Flow-Through Cell Setup

The design and photograph of the flow-through cell setup are shown in Figure 1 and Figure 2, respectively. The leaching solution is pre-heated in a 500 mL solution reservoir by a hot plate under stirring and pumped by a peristaltic pump and flow through the mineral sample in the sample cell. The temperature of the sample cell is maintained by a hot air blower underneath the sample section. After reacting with the mineral sample and flowing out of the sample cell, the leaching solution returns to the solution reservoir, completing a closed loop. PXRD patterns

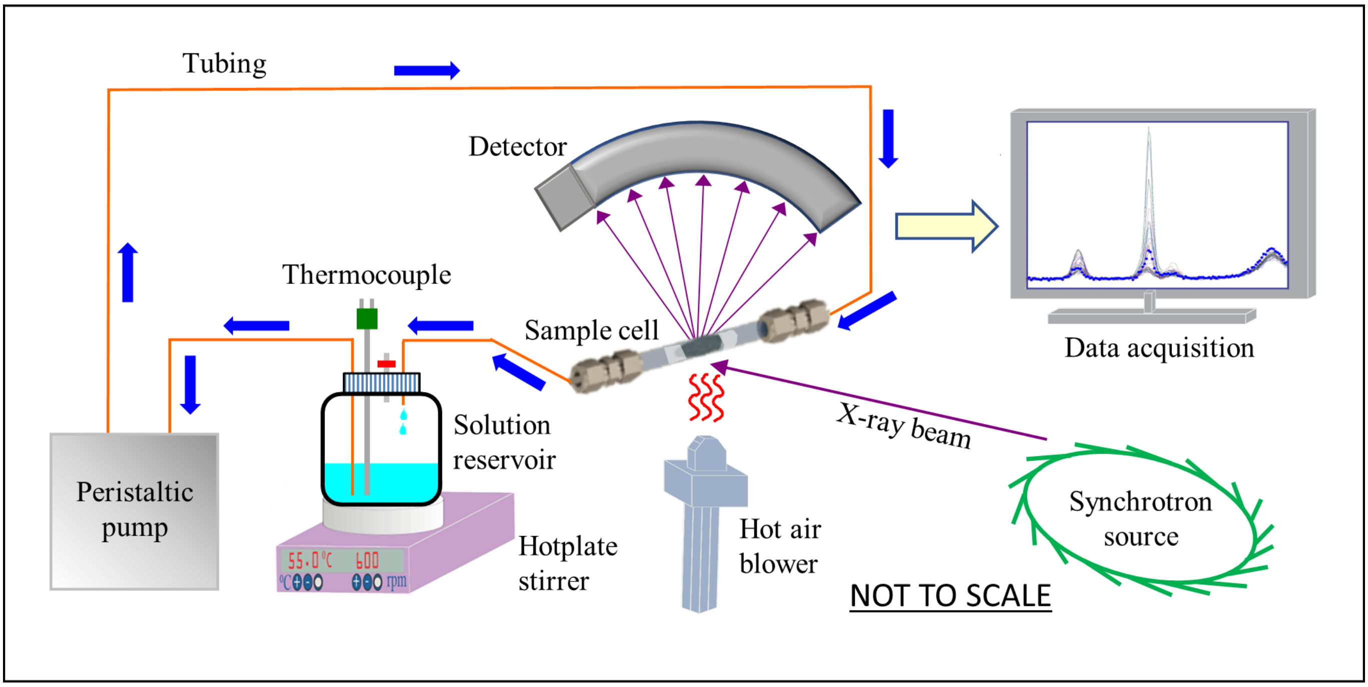

Figure 1. Schematic diagram of the flow-through cell experimental apparatus. Blue arrows show the flow direction.

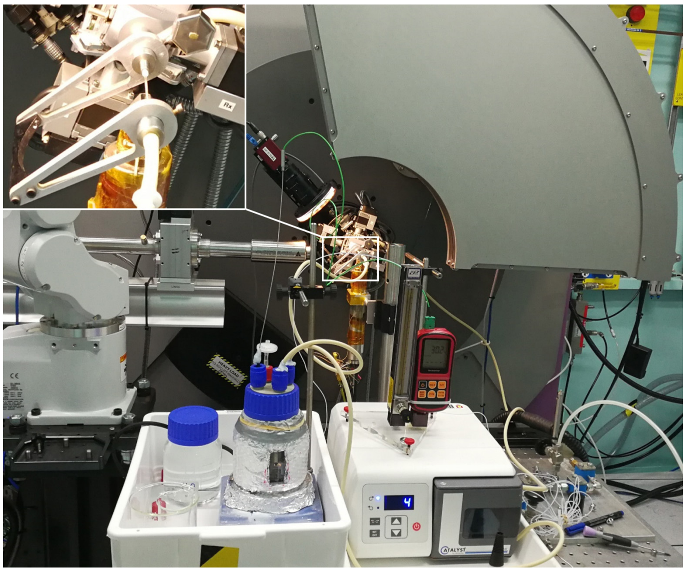

Figure 2. Photo of the flow-through cell setup at the Powder Diffraction beamline at the Australian Synchrotron.

are continuously collected during solution-mineral reactions in the sample cell, achieving in-situ monitoring of dissolution/crystallization processes. The fluid reservoir was insulated using a silver foil air bubble wrap, in which a small window (20 × 80 mm) was cut to allow flow observation during in-situ PXRD experiments.

The design of the sample cell is shown in Figure 2 inset. It consists of an open-end silica glass capillary tube (1.0–1.3 mm in internal diameter and 0.1 mm in wall thickness), two 316 stainless steel fittings for solution inlet and outlet, and an aluminum frame. The powder sample (~3 mm long) is loaded into the middle section of the tube, and the sample position is fixed by adding a silica glass wool plug on both sides of the sample. The capillary tube is sealed to the two stainless steel fittings which are fixed to the frame.

In-Situ PXRD of Galena Leaching

The flow-through cell was tested at the Powder Diffraction beamline at the Australian Synchrotron using an X-ray beam energy of 21 keV. An in-situ PXRD experiment on galena leaching was carried out at 35 °C, in a pH 6 lixiviant, consisting of 0.08 M citric acid, 0.92 M trisodium citrate, and 0.75 M H_2O_2, under a flow rate of 0.5 mL min^{-1}. The galena powder was diluted by silica glass powder to reduce the total X-ray microabsorption (mµR) to less than 1.

Figure 3 shows the PXRD patterns during the in-situ leaching of galena. A systematic reduction in galena peak intensity with time over 14 min suggests progressive dissolution of galena. This result agrees well with rapid leaching of galena reported in an ex-situ study under similar conditions [7].

The data quality was good, suggesting effective minimization of microabsorption by dilution. As an example, Rietveld analysis of the first dataset is shown in Figure 4. The broad peak centered at about 8° is from the amorphous silica tube, silica powder, and leaching solution. Preferred orientation of galena was observed as the intensity of the (200) peak at 11.4° is higher than expected. This is due to the

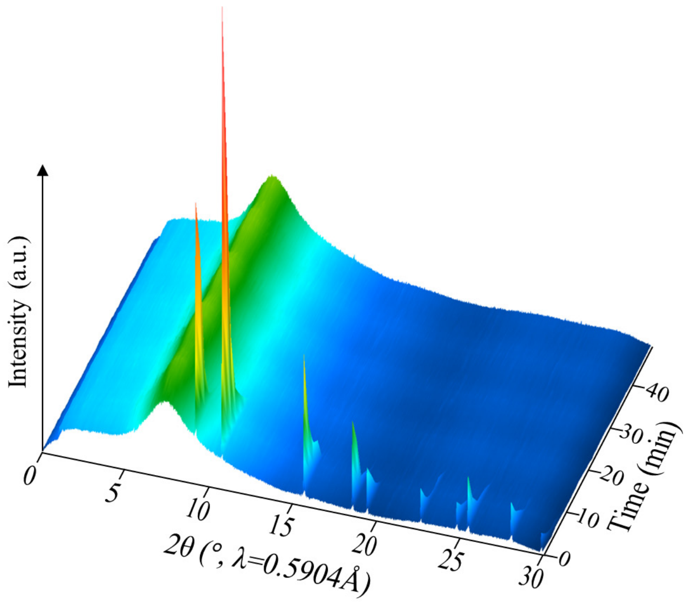

Figure 3. In-situ PXRD patterns showing rapid galena leaching at 35 ° C in a pH 6 solution.

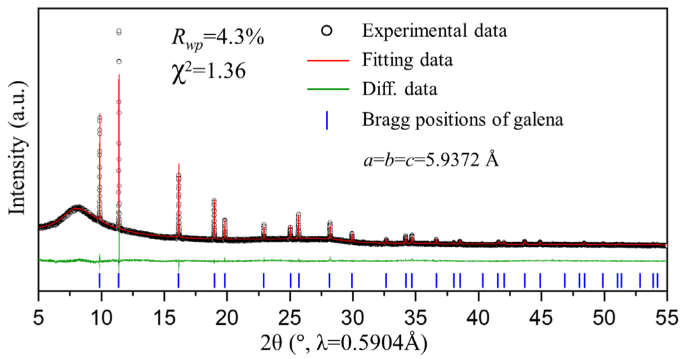

Figure 4. Rietveld refinement of the first dataset from the in-situ PXRD experiment.

perfect cleavage of galena on {100} lattice planes. The refined unit cell parameters of galena agree with the literature data.

Conclusions

A flow-through cell has been developed for in-situ PXRD studies of minerals leaching, in which the solid is kept in a sample cell as the stationary phase, and the desired amount of solution circulates through the sample. The flow-through cell can be used to study atmospheric pressure leaching of other ore minerals by in-situ PXRD at temperatures up to 95 °C.

This is a collaborative effort between Murdoch University (Fang Xia, Fatemeh Nikkhou, Xizhi Yao, Idowu A. Adegoke) and the Powder Diffraction beamline at the Australian Synchrotron (Qinfen Gu, Justin A. Kimpton). For more details, please see our published paper [8]. https://doi.org/10.3390/min10110990

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8. F. Nikkhou, F. Xia, X. Yao, I. A. Adegoke, Q. Gu, J. A. Kimpton. Minerals, 2020, 10, 990.

New Grant (ECR/MCR)

Regional Collaborations Programme COVID-19 Digital Grants

Grants of up to A$10,000 each are available for early-career and mid-career researchers to increase connectivity and engagement between Australian and Asia–Pacific economies in response to the COVID-19 pandemic.

The grants support projects that utilise digital methods of collaboration to address shared regional challenges that directly or indirectly relate to the COVID-19 pandemic response and recovery. This can include data sharing platform support, open science data formatting costs, online conference/webinar access support, or to research and develop digital tools to respond to regional COVID-19 needs.

Deadline: 14th December

More information: https://www.science.org.au/supporting-science/awards-and-opportunities/regional-collaborations-programme-covid-19-digital-grants

BRUKER

Live from the Lab

Live from the Lab is a series of YouTube videos where we explore Bruker AXS technologies and products. Each month we broadcast live from the Application Studio in Madison, Wisconsin, featuring insights from experts into a wide range of fields. Subscribe to our YouTube BrukerTV channel for the livestream or watch it later on-demand.

Watch now:

August episode: What is IPGID?

September episode: Resolution in XRM

October episode: Perfect Powder Diffraction Data, Automatically?

Featured Webinar

Elemental Analysis of Food and Feed Products with XRF

The quality of forage, pet food, and food products is subject to strict regulations. Screening of incoming material, monitoring of production processes and verification of final product quality therefore need to be fast, reliable, and cost-efficient. X-ray fluorescence (XRF) spectrometry is the optimal elemental analysis method for:

- Positive Material Identification (PMI)
- Foreign Body Identification (FBI)
- Quantification of mineral nutrients
- Quantification of toxic, heavy metals

Discover the benefits of XRF for food and feed applications in this 45-minute webinar. Click here to access the recording.

Featured Webinar

Advanced Topics in Practical Crystallography School

You are invited to a series of 5 tuition-free, hour and a half webinars on advanced topics in practical X-ray crystallography. We will cover Powder and PDF Data Collection and Processing, High Pressure Cell Data Collection and Processing, Using Ewald3D and new features in CrysAlisPro, and Non-spherical Atom Refinement with NoSpherA2.

The series runs Dec. 7–11, 2020 at 8 am CDT every weekday.

For more information and to register please visit:
https://www.rigaku.com/Webinars/SCX/advanced-topics-crystallography-school/

Applications now open

FameLab 2021

FameLab is an international competition to find and support the world's most talented new science communicators. Participants have three minutes to win over the judges and audience with a scientific talk that excels for its content, clarity and charisma.

FameLab is produced in Australia by the Foundation for the WA Museum. Find details about the competition and training workshops here.

Deadline for entry is 25 February 2021

Applications now open

FutureNow Scholarships

These scholarships are for graduate students or early career researchers working on industry-focused research projects aligned with ANSTO's strategic objectives. Scholarship recipients will have access to the expertise and technology needed to facilitate cutting edge discoveries in advanced manufacturing, health, environment and the nuclear fuel cycle. To be eligible you must:

- Be a graduate of a bachelor or equivalent degree or higher and be within five years of graduation or be undertaking a higher degree by research
- Be available to spend 6 months researching at an ANSTO facility
- If you are an Australian or New Zealand student, be enrolled at a university that is an AINSE member

More information: https://www.ansto.gov.au/work-us/innovation/futurenow-scholarships
Applications close 29 January, 2021

NEW RESPIRABLE SILICA DUST LAWS TO CURB SILICOSIS IN THE WORKPLACE

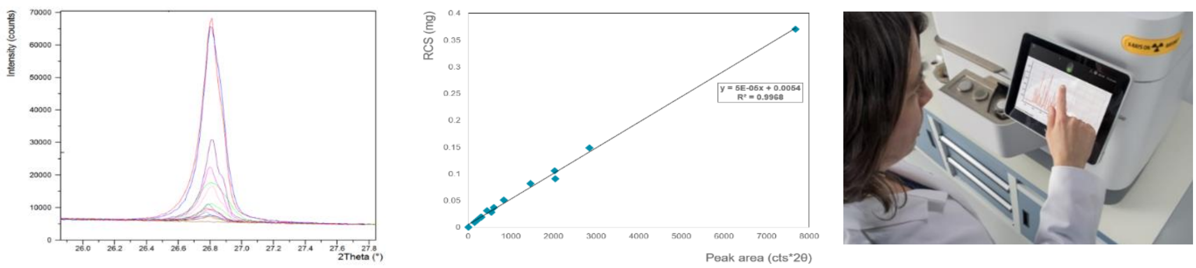
Is your lab equipped for this stringent testing?

As of October 2020, Australia's New South Wales parliament has amended the Work Health and Safety Bill. Silicosis, asbestosis and mesothelioma will now be notifiable diseases and companies are required to indicate in the new dust diseases' register if their employee acquired these through workplace exposure. In addition, if companies don't acquire compliant methods to measure dust levels in the air, they run the risk of under declaring and possible fines by the government. An increase in silicosis cases within Australia led to stricter regulations. As you may know on July 1st 2020, the daily limits of silica was halved from an eight-hour time-weighted average of 0.1 mg/m^3 to 0.05 mg/m^3 under the Work Health and Safety Regulations 2011. Any mechanical treatment of silica will cause airborne particles of silica dust; occupations most at risk are those in stonecutting, quarrying, tunnelling, brick and tile making amongst others.

X-ray diffraction (XRD) can meet the ISO norms with ease. Malvern Panalytical is proud to announce that Safe Environments is the first NATA accredited lab for highest level of testing as per ISO 16258-1:2015. This standard is superior as it uses XRD to analyse not only the α-quartz but also the cristobalite – another crystalline silica polymorph. This testing is conducted using XRD, which is capable of accurate and repeatable low limits of quantification.

"Ultimately, we want greater confidence in our results so that timely decisions can be made to safeguard workers from overexposure to respirable crystalline silica. Previous testing of only α-quartz may have led to underreporting, particularly in the engineered benchtop stone industry. XRD is preferred for more precise testing as it is less prone to mineral interferences. There was also an opportunity in XRD to semi-automate the process and optimise the measurement time to increase accuracy and reduce detection limits."

Carl Strautins, Occupational Hygienist at Safe Environments Testing Laboratory



Learn how to use X-ray diffraction (XRD) to collect data (left) and get practical tips on sample preparation to setting up your XRD calibration line (center). See how Safe Environment complies with stringent ISO test standards using the compact Aeris XRD (right) to collect superior quality data in under 5 minutes.

For a limited time, we are offering our EIGER2 R 500K detector as an upgrade for D8 DISCOVER and D8 ADVANCE DaVinci systems, at **20%** off list price. Get more information here.

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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Daniel Fanna (WestSyd, NSW)

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 2021. 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 Communications Editor Valerie Mitchell at ausxray@gmail.com.
www.axaa.org/a-day-in-the-life.html