AXAA

# Australian X-ray Analytical Association

*Newsletter Issue 3 2017*

## President's Address

Dear AXAA Members and Friends,

You may be aware that Dr Keith Norrish, regarded as the pre-eminent pioneer of wavelength dispersive X-ray spectrometry for the analysis of minerals, passed away peacefully on the morning of Wednesday 13th September aged 93. Keith made a major contribution to the development of world-class analytical methods for the mining sector in Australia, and his methods are now widely used internationally. Keith worked closely with the Standards Association of Australia and the International Standards Organisation to standardise X-ray analytical procedures. Keith's significant contribution to X-ray analysis, and AXAA, is recognised through the Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis, awarded every three years for significant long term contributions to X-ray analysis. Keith was also a Life Member of AXAA.

Please read on for a fitting tribute to Keith given by a number of his long-term colleagues including our most recent Keith Norrish AXAA Award winner, Greg Moore.

The National Council will be ramping up efforts early in 2018 for the organisation of AXAA-2020. As mentioned in our last Newsletter one of the major considerations for future events is the cost, and we are currently looking to scale down the exhibition portion of the event a little in conjunction with a more modest venue to reduce registration fees. We welcome your thoughts on this, including possible conference venues, and invite you to contact the National Council with these (see contact details at the end of the Newsletter). We also welcome nominations from the AXAA community for AXAA-2020 Conference Committee members, so if you or someone you know is interested in helping to shape our next conference, please get in touch.

For now, as we approach the end of 2017 I wish all AXAA members, friends and their families a happy and safe festive season.

*Nathan Webster*
*AXAA President*

## Dr. Keith Norrish BSc, Hons, MSc, PhD, AO, FAA
## 09/07/1924 - 13/09/2017

*1977 Keith Norrish FAA Award*

Keith Norrish, an only child, was born on the 9th of July 1924 at Kojonup, Western Australia, a farming area south of Perth and it was there that Keith spent his early years. On reaching schooling age, he and his mother moved to Perth where for the early part of his education he attended school at Subiaco.

From an early age Keith had an enquiring mind and as he recalled in an interview about his early education "I had two books I can remember that I saved up and bought: one was called How it Works and the other was called How it is Made, and I would say that's still almost the basics of physics," (1)

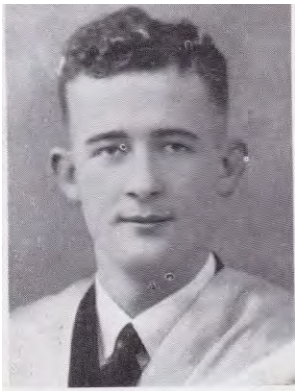
*Keith Norrish, Dux, CBC (Perth), 1941*

Secondary schooling was in the Catholic education system and he completed his secondary education at Christian Brothers College (CBC), as Dux of school, in 1941. Favourite subjects, which he no doubt excelled in, were Physics, Maths, Applied Maths and Chemistry.

On completion of his secondary education Keith was awarded a Hackett scholarship and this, coupled with the university being a free university, enabled him to go to the University of Western Australia where he studied for the "standard" science degree with Chemistry, Physics, Maths and also Geology. He graduated with first class honours in physics and followed this up with his MSc toward the end of 1946. He had the good fortune to do his Masters research work on the mineralogy of WA soils using X-ray powder diffraction under the mentorship of Mr John Shearer of UWA Physics, so career wise the die was cast. Looking back, his scientific lineage from those early days was remarkable. Shearer had worked with the renowned Swedish physicist Manne Siegbahn who had

been awarded the Nobel prize for Physics in 1924 for his work on X-ray spectroscopy. Down the track Keith was to become a world expert in the use of Siegbahn's X-ray spectroscopy for the analysis of minerals.

Keith's long association with CSIR (Council for Scientific and Industrial Research), latterly CSIRO, began in 1947 when he joined the CSIR Division of Soils in Adelaide. The Soil Physics section there was headed up by Dr Tim Marshall and it was there that Keith went to work on clays using a newly bought X-ray diffraction unit. This fitted in well with the work, X-ray crystallography, he had undertaken in gaining his MSc. (It should be noted that this was still early days in the study of clays using diffraction which had opened up a whole new way of looking at clays as a crystalline mineral group). Soil Physics was a relatively new section with some five or six research scientists plus technicians. Keith saw Dr Marshall as an excellent leader and role model.



*Dr Keith Norrish PhD, London University, 1953*

In 1950 Keith was awarded a CSIR studentship to work at Rothamsted Experimental Research Station in the UK. Awarded his PhD from London University in 1952 he returned to Adelaide. During Keith's time in the UK, Ted Radoslovich has been tasked by Keith with building an X-ray spectrometer. He succeeded in building a very basic curved crystal spectrometer which Keith used to good effect over the next nine years in improving his understanding of the instrument and development of methodology. It should be noted that Keith's experience with spectrometry went back to 1946 when, whilst working on his MSc, he built his own Bragg spectrometer so that he could examine the Co target of his diffraction tube for impurities – he thought there was only one but instead found twenty!

Soil Physics was initially housed at Waite Institute, and in 1952 a Mineralogy section headed up by Keith and including research staff from Soil Physics, was formed. Outgrowing the space at Waite they moved to the basement of the Geology faculty at Adelaide University in 1954 and eventually, in 1961, to the new Soils Division building at Urrbrae. Now Keith had quite a team of research scientists working with him: Reg Taylor, Rex Sweatman, and Ted Radoslovich, from his days in the basement at the Geology faculty, and John Hutton, Charles Oertel, Reg McKenzie, John Giles and technicians who all joined the group at the new Urrbrae facility.

In 1961 an AMIRA grant enabled the purchase of their first commercial X-ray spectrometer, a Philips PW1540. This was installed in a purpose built room (sheathed in copper) next door to the X-ray diffraction room. A very basic instrument, even by 70's standards, with valve electronics, manual operation of the goniometer and sample changer, limited analyser crystal options, and not particularly stable. Nonetheless this was the start of many years of research and development in X-ray spectrometry equipment, methodology for the preparation of soils and geological samples using powder briquetting and glass fusion techniques (CSIRO Divisional Report 3/64) and the analytical technique itself. The period from 1961 to 1967 saw the in-house development of 1 micron windows for the flow detector, a vacuum airlock, the growing and testing of analyser crystals, reduction of system dead time, installation of an automated PHS corrector unit, and inbuilt dead time correction in the counting electronics, amongst others. Very much a hands on person Keith was always in the thick of it and did much of his own work. With his home just down the road, Keith would often come back to work out of hours. Unfortunately this enthusiasm did result in Keith badly burning his fingers with X-rays when "playing around" with an X-ray tube mask. A constant reminder of this for the rest of his life were the pieces of sticky tape covering the damaged nerves. Without these improvements it is doubtful whether the Keith Norrish and John Hutton paper published in Geochemica et Cosmochimica in 1969 "An Accurate X-ray Spectrographic method for the analysis of a wide range of geological samples" covering the theory and practice of a robust XRF fusion method, using a Li borate flux with heavy X-ray absorber ($La_2O_3$) and matrix correction factors, would have been published when it was. Importantly this paper formed the basis of XRF analysis at many laboratories.

This was just one of many of Keith's publications – in his words "I think it's a hundred and something papers or possibly two hundred in good journals. Then I have lots of what they call technical reports, almost all of the work I did for industry is in this category." (1)

In 1967 he also co-authored with Prof Bruce Chappell

chapter 4. "X-ray Fluorescence Spectrography" in the publication "Physical Methods in Determinative Mineralogy" (4)

During Keith's time at CSIRO, the PW1540 was eventually replaced with a Philips PW1400, then after the flood in early 1990 a Philips PW1480, then finally a PANalytical Axios Advanced. Appointed a Chief Scientist in the 70's he retired in 1989 after 42 years with CSIRO but remained actively involved in XRF analysis for many years, presenting at X-ray schools, leading the development of Australian standards, precision testing of X-ray spectrometers, and advising laboratories. This was a continuation of what he started in the early 60's by helping the Port Pirie Lead smelter, followed by BHP Broken Hill, British Phosphate Commission, Brown Coal Victoria, MIM Mount Isa and from there bauxite, iron ore and mineral sands industries amongst many others.

X-ray spectrography was Keith's passion though it should not be forgotten that Keith's work for his MSc and PhD involved clay minerals (Montmorillonite in particular) and thus X-ray diffraction. His work on clays and mica continued during his whole time at CSIRO as evidenced by a number of his awards.

**A distinguished career recognised by numerous scientific bodies**

*1968 – 1969: President of the Australian Clay Minerals Society*
*1977: Fellow of the Australian Academy of Science - contributions to soil and clay mineralogy*
*1977: Soil Science Society of Australia Prescott Medal*
*1988: Denver X-ray Conference Birks Award for Excellence in X-ray spectrometry*
*1989: Australian Honours Commander Officer of the Order of Australia (AO)*
*1989: Mica Mineral named in his honour - norrishite [$K(Mn_2^{3+}Li)Si_4O_{12}$]*
*1995: Australasian Institute of Mining and Metallurgy award for Operating Technique*
*2001: Clay Minerals Society of USA Bailey Distinguished Member award*
*2002: University of Western Australia Honorary Doctor of Science (Hon DSc)*
*Australian X-ray Analytical Association Life Member*

Beyond the science, Keith was much loved for his warmth to others, for his wry sense of humour, for his modesty and his healthy disregard for bureaucracy. Most of all, he was devoted to his family. He and wife Betty raised a wonderful family in Adelaide – four daughters and a son, their much loved seventeen grandchildren and, to date, six great grandchildren.

Dr Keith Norrish worked with, mentored, and helped innumerable people during his career. A few of those who were privileged to work with Keith have put together some thoughts on their time working with him.

*Greg Moore and Brian O'Connor*

**Greg Moore**

I was privileged enough to work as assistant for both Keith and John Hutton from 1963 to the end of 1970. These were important years as many of the foundations for quantitative XRF analysis were established by Keith during this period.

Keith's easy going nature meant that the mineralogy section was a productive, exciting and happy place to work. Morning tea was always a highlight where the whole section, maybe fifteen research scientists and technicians, came together in the meeting room for tea and bikkies. A tremendous atmosphere where research work (successes failures and an exchanging of ideas) politics and bureaucracy, cars, holidays, in fact almost anything, was shared and aired with Keith always in the thick of it. The break often went past the official 15 minutes. Something that Keith had carried over from his days with Dr Marshall in Soil physics.

Who can forget Keith at AXAA conferences, working at calculations on the White board, looking down, fumbling with his tie, (surprising as it was a rarity for him to wear one!) looking up and writing numbers on the board - Turned out the tie pin was a miniature slide rule.

Neil Armstrong took " …. one giant leap for mankind" on July 20th 1969. Keith marched us off to his home for the day, where Betty plied us with food and drink all day, so we could watch the moon landing on his TV. As an addenda to this I heard years later that Keith got to touch, maybe even analyse, some of the moon rock and even got to secrete some moon dust into his hanky…….

Keith's technical officer John Pickering, was mentored by Keith from the early days and became an accomplished diffractionist.

Working on clays, often minute inclusions dug out of rocks, meant a Debye-Scherrer camera was often used. Good friends, they would eagerly wait for the film to be developed and often a spirited (but never heated) discussion would follow in identifying, by eyeballing the negative, what clay mineral it was. The card index was rarely needed.

Keith was forever wandering around seeing how things were progressing. One of my tasks was to prepare fusion beads using the practically challenging press quench technique. Pt/Au crucibles lose their colour whilst still at a high temperature (800C ?). Keith was want to pick up crucibles after the pour to see what the glass prill left behind looked like but lack of feeling in his fingers (from the X-ray tube accident) meant it was not uncommon to catch a whiff of charring protective tape. He did eventually learn not to pick them up…

Keith's desk, in fact his whole office, was best described as organised chaos. Anecdotally it is said that his briefcase (which never seemed to leave the office) was part of his filing system with incoming mail selectively filed there. Months end often saw it emptied into the wastepaper bin.

A distinguished scientist who imparted so much to those who worked with him (you always worked with him, not for him) I am indebted to Keith for providing me with the basis of my career in mineral laboratories.

**Ken Turner**

I first corresponded with Keith soon after joining BHP Research in September 1977. If memory serves me correctly, I had inherited a test program that was part of the Aluminium Ores Standard Method development and was reporting our results. It was my first experience with Keith's press quench fusion bead method. We corresponded at bit about the results (which were not too bad) and methods, etc.

Later, I served on the AS/ISO Iron Ores standards committee and it was on this committee that I had my main contact with Keith. He had a brilliant mind and his contribution to AS and ISO standard XRF methods of analysis is immense. Keith liked to mull over issues and ideas before commenting. It was not uncommon for Keith to put forward ideas and comments long after the rest of us had thought that a particular issue or comment was put to bed. I well remember one particular meeting where we had all packed up and were heading for the door when Keith said something like "we should really consider doing .....".

We finished up an hour later having achieved more in that hour than what we had all day! It is fair to say that without Keith's input, drive and above all vision most of the AS/ISO XRF standard methods and AS2563 for testing XRF Precision would never have seen the light of day.

During my 25 years with BHP I worked with and/or sought Keith's advice on a number of specific projects. Sometimes I just needed someone to bounce ideas off and at other times he was able to help me with curly problems. I recall one project involving measuring and quantifying F at trace levels in base metal concentrates. His initial response was "don't do it, sample preparation will beat you at the levels you are looking at" and then we sat down and went through all the technical XRF aspects. His XRF technical advice was spot on and the technique worked well except at low trace levels where, issues of reproducibility of sample preparation became insurmountable. The lesson to be learnt - I should have listened to his initial response and saved myself considerable time and effort!!

Keith's depth of XRF knowledge and experience was immense and he was always willing to share. Keith provided me with great support over the years. We didn't always agree and we had a few "robust" technical discussions over the years. Always good science!!

The XRF community has truly lost one of the pioneers and he will be sorely missed as a scientist and gentleman.

*Keith in Feb 2017 with AXAA award named after him*

**Sally Birch**

I joined CSIRO in 1989 to assist Keith with development of Standard XRF methods for analysis of iron ores and other mineral products. That was the year that Keith officially retired from CSIRO, but retirement did not slow him down at all and he continued to advance the development of ISO and Australian Standards for another couple of decades. As a direct result of Keith's work, Australia has led the way internationally in the use of XRF as a technique for analysis of iron ore and other materials.

As well as being a remarkable scientist with a great intellect, Keith was a great boss. His enthusiasm and tenacity to get to the heart of a problem was infectious and he inspired many people along the way. He enjoyed all aspects of experimental work, was always prepared to get his hands dirty and was very appreciative and supportive of the contributions of others. He was a kind and generous man and will be greatly missed by those that had the pleasure of working with him.

**Mark Raven**

I had the great pleasure of working with Keith as his technician in XRF analysis from 1986 before his official retirement 3 years later. Back then there were several larger than life characters in the Mineralogy Section, all pushing the frontiers of science, but none larger or livelier than Keith. Morning and afternoon teas were always a delight listening to Keith and his colleagues reminisce about the 'good old days' when funding was freely available for researchers to just get on and do the work. I can recall one of Keith's stories about a visit to the London Museum, where armed with a geology pick, was invited to just hack off a piece of any of the display specimens he would like. I'm not sure about the validity of Keith's recollection but over the years his passion for characterising geological materials has contributed to an impressive collection of type clay specimens from all points of the globe. Many of the specimens have been used subsequently for advancing the understanding of the structure and properties of clays and the crucial role that clay minerals play in a wide range of industries. One of Keith's favourite expressions was "bugga", mostly when something didn't quite work out, but his persistence and focus ultimately solved the problems he faced.

Keith was also well ahead of the game when it came to engaging with industry and attracting external funding, which has been the funding model for CSIRO since the late 80's. Many of Keith's achievements have had a profound effect on the scientific community and none more so than on the many colleagues he worked, mentored and inspired to go on to bigger and better things, myself included. He will be sorely missed.

**References**


(1) *State Library of South Australia, J.D. Somerville, Oral History Collection, Doc: OH794*

(2) *A History of the CSIRO Division of Soils 1927 – 1997, K.E. Lee, Technical Report 43/98*

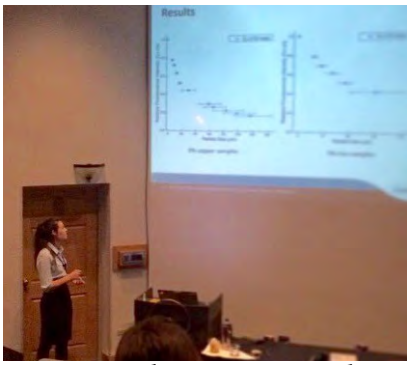
(3) *History of the School of Physics at UWA. The Beginning of Research. Author: Dr J L Robins (2009)*

(4) *Physical Methods in Determinative Mineralogy, Academic Press New York, Ed: J Zussman, 1st ed: 1967, revised 2nd ed 1977*


## AXAA students present at the Denver X-ray Conference

DXC DENVER X-RAY CONFERENCE
66th Annual Conference on Applications of X-ray Analysis
31 July - 4 August 2017 ▪ Big Sky Resort, Big Sky, Montana, USA

This past July, Jessica Hamilton and I travelled to the Denver X-ray Conference after receiving generous student travel grants at the AXAA 2017 conference. Our grants were awarded for the best student presentation and poster, and were funded by CSIRO and ICDD. Despite being named the 'Denver X-ray conference', the conference was actually held at the Big Sky resort in Montana, surrounded by the Rocky Mountains and breath-taking scenery.



*Brianna Ganly presenting at the Denver 2017 Conference.*

The conference began with two days of tutorials, focusing on all aspects of XRF and XRD, followed by two nights of poster sessions and networking opportunities. As every young scientist knows, networking is a crucial step to establishing a scientific career and this was an excellent opportunity to meet other scientists from both the XRF and XRD fields. The next three days were filled with insightful presentations, and both Jessica and I had

the invaluable experience of presenting our work to an international audience.

Overall the conference was a brilliant experience, and one of the highlights of my PhD. I made some great connections and broadened my knowledge of the XRF analysis techniques used by different companies and research teams. At the conclusion of the conference the attending Australian PhD students all teamed up and spent two days exploring Yellowstone National Park, a wonderful end to a great conference experience.

*AXAA student members Brianna Ganly, Jessica Hamilton and Connor Turvey at Yellowstone National Park.*

*Brianna Ganly*

## Upcoming XRF Workshops

- **XRF in the Workplace** – Perth, 19th – 23rd March 2018
- **XRF in the Workplace** – Sydney, 16th – 20th July 2018
- **SuperQ Software Hands On Training** – Perth, 1st – 3rd May 2018

The XRF in the Workplace course is a practically oriented training course that is suitable for all users of XRF (not just Malvern Panalytical users). Contact Grace (grace.perrone@panalytical.com) for further details and/or to register for any of the above courses.

## X-ray Materials Analysis Internet Courses – Wavelength Dispersive XRF and Powder XRD (Plus Mentoring Program on Rietveld XRD Phase 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 comprise 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 10, 2018

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.

### Internet XRD Course: Series 5, 2018

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.)

### 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 fast-analysis of 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.

## Reynolds Cup 2018


The 9th biennial Reynolds Cup competition for quantitative mineral analysis is now open.

You can register your interest in participating in the contest by sending an email to Rieko Adriaens at radriaens@qmineral.com.

The competition is open to anyone interested in quantitative mineral analysis, with particular emphasis on clay mineralogy. Information about the competition including guidelines and previous winners can be found at http://clays.org/Reynolds.html . The competition is free for all to enter, however, those that are not members of the CMS are encouraged (but not obliged) to become members (see http://clays.org/join_benefits_of_membership.html).

## Upcoming Conferences

XXII MEETING OF THE INTERNATIONAL MINERALOGICAL ASSOCIATION
13-17 AUGUST 2018 | MELBOURNE

### IMA2018

13 - 17 August 2018
Melbourne, Australia

The International Mineralogical Association is an umbrella organisation uniting professional associations from 38 countries, and its quadrennial meeting is the largest global forum for making contacts and exchanging information with mineral scientists from around the globe. The 22nd meeting in Melbourne, Australia, will be the first such meeting held in Australia and only the second in the southern hemisphere. The meeting will be hosted by the Geological Society of Australia, and held in the Melbourne Convention and Exhibition Centre, between 13–17 of August, 2018.

Abstract submission closing date: **28th February 2018**

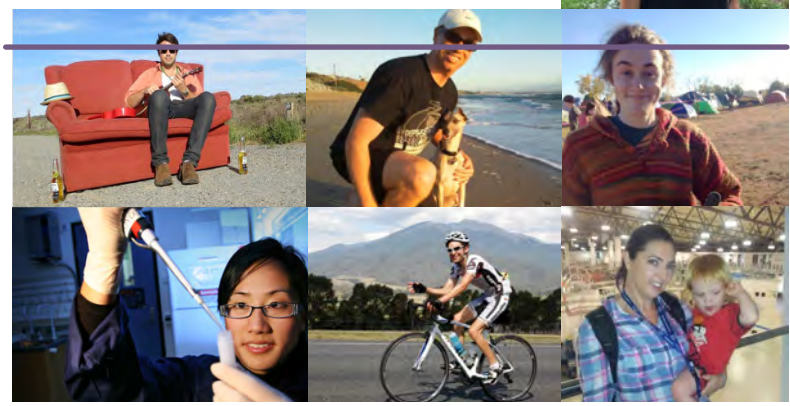
Website: https://www.ima2018.com/

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

In 2016, AXAA started a blog series as a new way to get to know our members. Our 'Day in the Life' posts take a peek behind the scenes of different workplaces to find out the fun bits, the challenging bits, and why you do what you do.

We are currently seeking posts so if you'd like to contribute, or know someone who might be interested, please contact National Council Communications Editor Jessica Hamilton.

W: www.axaa.org/a-day-in-the-life.html



## 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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### 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 provide their CV and a short statement about how they intend to contribute to the organisation. Please send these to the National Council Secretary Mark Styles.

### 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 December 2017. Please feel free to send contributions for the newsletter to Jessica Hamilton at any time. Any comments or feedback about the Newsletter are welcome.

# Bruker Australia / New Zealand News Dec 2017





## S8 TIGER Series 2 first Installation!

Bruker are proud to announce the first installation of our new WDXRF S8 TIGER Series 2 instrument at the Institute of Agriculture and Environment at Massey University in Palmerston North, NZ.

"Associate Professor Georg Zellmer from Volcanic Risk Solutions at Massey University has led the acquisition of the new Bruker TIGER S8 Series 2 XRF spectrometer, installed at the Institute of Agriculture and Environment (IAE).

This instrument will be used for major and trace element analysis in volcanology, river sediment provenance, and environmental and soil contaminant research – all major areas of the College of Science research strategy, and supporting research student projects across the board. The new instrument also has the potential to contribute to several related areas of research within the College of Sciences, including environmental sustainability studies.

Beyond IAE, staff members in chemistry, environmental health, and environmental engineering have interests in using the new instrument for their research."

For more information about the S8 TIGER Series 2, please visit www.bruker.com/s8tiger

## Melbourne & New Zealand XRF schools

October & November saw another series of SPECTRAplus training courses, held at our Melbourne application laboratory & Massey University in New Zealand.

Attendees to the course came from a diverse background including the cement, mineral sands, aluminium and academia.

The course was again conducted by Elvy Grigolato the Bruker XRF application scientist who has over 30 years' experience in XRF analytical techniques working in Australia and the Asia Pacific region. Attendees were trained in the advanced use of the powerful SPECTRAplus software for instrument calibration, diagnostics and standard less analysis using the powerful QUANT-EXPRESS.

Danny Verbeeten from XRF Scientific conducted an informative session on sample preparation with Lithium Borate Fusion including Fusion theory, care of platinum ware and troubleshooting the fusion of difficult sample materials.

The session concluded with a demonstration of the xrFuse2 and xrWeigh systems from XRF Scientific.


## Public XRF Seminar – New Zealand

As part of the S8 TIGER 2 installation at Massey University we took the opportunity to run a public seminar on X-ray Fluorescence analysis. The seminar was well attended with over twenty people from the University and as far away as Wellington & Auckland.

The seminar covered a number of topics including XRF theory, XRF instrumentation, Sample preparation, Common applications. XRF Scientific contributed to the seminar with a presentation of fusion preparation.

If you are interested in finding out about upcoming Bruker events please contact neil.hughes@bruker.com for more details.


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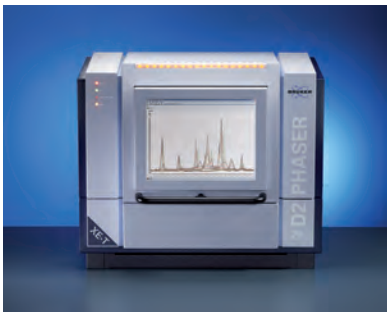
## Bruker Announces Updated D2 PHASER™ Benchtop X-ray Diffraction (XRD) System with LYNXEYE XE-T™ One-dimensional Detector

Bruker announces the availability of the LYNXEYE XE-T next generation one-dimensional compound silicon strip detector, further enhancing the performance of its popular D2 PHASER™ benchtop X-ray diffraction (XRD) system.

The versatile hardware of the updated D2 PHASER combined with the Bruker DIFFRAC.SUITE™ software allows users the flexibility to create automated push-button methods for ease of use and to customize measurement conditions and configurations for optimized data quality. This benchtop system needs no water cooling or special power requirements, making the D2 PHASER unmatched in its ability to bring powder XRD to any laboratory.

The LYNXEYE XE-T detector features four times better energy resolution than traditional silicon strip detectors in order to efficiently remove unwanted radiation,such as fluorescence, K-beta radiation, and

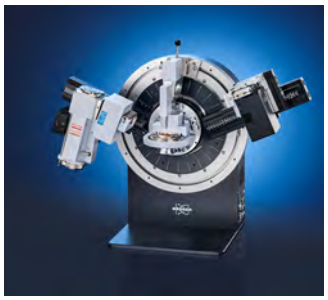



bremsstrahlung background, without meaningful losses in one-dimensional detection speed.

According to Arnt Kern, the Bruker AXS Product Line Manager for XRD: "The D2 PHASER with LYNXEYE XE-T sets a new standard for benchtop-class high-speed data collection, outstanding peak to background ratio, and data quality."

For more information about the D2 PHASER LYNXEYE XE-T, please visit www.bruker.com/d2phaser

## Bruker and DECTRIS Announce Advances in D8™ X-ray Diffraction Systems with the New EIGER2 R 500K Detector



Bruker and DECTRIS announce the EIGER2 R 500K, the latest generation of Hybrid Photon Counting (HPC) pixel detectors developed by DECTRIS, the technology leader for HPC detectors for laboratory instrumentation and synchrotron beam lines. In close collaboration, DECTRIS and BRUKER have seamlessly integrated this outstanding 2D detector into Bruker's D8 ADVANCE™ and D8 DISCOVER™ instrument platforms to further improve performance for numerous X-ray diffraction (XRD) applications.

The most recent generation of the proprietary EIGER HPC detector offers a distinctive set of enhanced and new benefits for XRD: high frame rate enabling 2D data collection in continuous scanning mode, single photon counting without spatial distortion, highest count rates and dynamic range. In combination with the Bruker D8™ X-ray diffraction systems, switching between 0D, 1D and 2D mode is possible.

These new features are fully integrated into the Bruker D8™ X-ray diffraction systems, and enable the EIGER2 R 500K detector to produce excellent diffraction data for applications from powder diffraction (XRPD), micro diffraction (µXRD), texture or residual stress analysis, crystallite size determination, thin film analysis by X-ray reflectometry (XRR) and high-resolution diffraction (HRXRD) to small angle X-ray scattering (SAXS and GISAXS). This powerful combination of the D8 ADVANCE or the D8 DISCOVER with the EIGER2 R 500K significantly extends and speeds up the analysis capabilities for XRD users.

"Collaborating from the early stage in the product development, we were able to provide our new EIGER2 R 500K detector with seamless integration into Bruker AXS D8 XRD systems. This is enabling customers to take advantage of cutting-edge detector technology for their laboratory right from the launch", stated Christian Broennimann, the CEO of DECTRIS.

Dr. Lutz Bruegemann, the Bruker AXS Vice President and General Manager XRD , added, "The coordinated development teams at DECTRIS and Bruker have done a tremendous job. The result is outstanding synergy between the latest HPC detector technology and the most powerful and user-friendly XRD platform on the market, enabling customers to use the new EIGER2 R 500K seamlessly incorporated into our D8 diffraction solutions for many important applications."

For more information, please visit
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### Accuracy and repeatability

The Epsilon 3$^{XLE}$ was used to measure a sediment sample 20 times consecutively. As you will note from Figure 1's calibration graphs, there is strong correlation between the certified concentrations and measured intensities. From Table 1, there is excellence accuarcy and precision when comparing the average concentrations against the certifitied concentrations as well as RMS and relative RMS values.

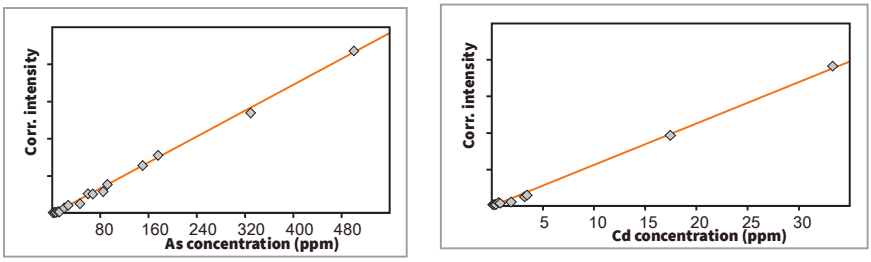


*Figure 1. Good correlation between the certified concentrations and measured intensities for As (left) and Cd (right) in soils and sediments*

| Compound | Certified conc. | Average conc. | RMS | Rel. RMS (%) |
|---|---|---|---|---|
| $Al_2O_3$ (wt%) | 13.44 | 13.44 | 0.0200 | 0.1 |
| As (ppm) | 84.00 | 81.00 | 1.0000 | 0.9 |
| Ba (ppm) | 720.00 | 742.00 | 5.0000 | 0.7 |
| CaO (wt%) | 1.66 | 1.63 | 0.0040 | 0.2 |
| Cd (ppm) | 1.05 | 0.90 | 0.3000 | 35.2 |
| Cr (ppm) | 122.00 | 124.00 | 2.0000 | 1.9 |
| Cu (ppm) | 38.00 | 35.00 | 1.0000 | 2.9 |
| $Fe_2O_3$ (wt%) | 6.49 | 6.71 | 0.0100 | 0.1 |
| $K_2O$ (wt%) | 3.55 | 3.62 | 0.0100 | 0.4 |
| MgO (wt%) | 3.06 | 3.12 | 0.0200 | 0.6 |
| MnO (wt%) | 0.09 | 0.09 | 0.0004 | 0.4 |
| Mo (ppm) | 1.40 | 1.40 | 0.1000 | 9.6 |
| $Na_2O$ (wt%) | 1.21 | 1.24 | 0.0500 | 3.6 |
| Nb (ppm) | 17.20 | 16.80 | 0.3000 | 1.7 |
| Ni (ppm) | 53.00 | 57.00 | 2.0000 | 2.9 |
| $P_2O_5$ (wt%) | 0.19 | 0.18 | 0.0040 | 2.2 |
| Pb (ppm) | 350.00 | 380.00 | 1.0000 | 0.4 |
| $SO_3$ (wt%) | 0.05 | 0.04 | 0.0010 | 2.6 |
| $SiO_2$ (wt%) | 64.63 | 67.74 | 0.0900 | 0.1 |
| $TiO_2$ (wt%) | 0.75 | 0.75 | 0.0020 | 0.3 |
| V (ppm) | 96.00 | 94.00 | 3.0000 | 3.2 |
| Zn (ppm) | 238.00 | 258.00 | 2.0000 | 0.7 |

*Table 1. Excellent accurate and repeatable results*

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# ICDD 2018 Product Summary

| Data Entry Source | PDF-2 2018 | PDF-4+ 2018 WebPDF-4+ 2018 | PDF-4/ Minerals 2018 | PDF-4/ Organics 2018 |
|---|---|---|---|---|
| 00- ICDD | 116,613 | 116,613 | 12,140 | 40,154 |
| 01- FIZ | 170,778 | 69,655 | 12,907 | 12,658 |
| 02- CCDC | 0 | 0 | 0 | 431,359 |
| 03- NIST | 10,067 | 2,917 | 213 | 282 |
| 04- MPDS | 0 | 208,741 | 20,175 | 0 |
| 05- ICDD Crystal Data | 800 | 800 | 62 | 41,673 |
| **Total No. of Data Sets** | **298,258** | **398,726** | **45,497** | **526,126** |
| Subfile Distribution: | | | | |
| Inorganic | 264,876 | 365,291 | 45,441 | 39,355 |
| Organic | 42,478 | 43,515 | 711 | 515,458 |
| New Entries | 7,139 | 14,113 | 1,156 | 10,072 |
| Rietveld—No. with atomic coordinates | 0 | 295,309 | 37,210 | 106,369 |
| Reference Intensity Ratio (RIR)—$I/I_c$ | 199,610 | 300,078 | 34,434 | 493,521 |
| Experimental Digital Patterns | 0 | 12,396 | 131 | 6,639 |
| Pattern Fitting—Calculated Digital Patterns | 0 | 398,726 | 45,497 | 526,126 |

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ICDD 11/17

# Bead One R for accurate and precise analysis (ED-XRF) in cement applications


## Abstract

For quantitative analysis of cement samples, IMP offers the HERZOG Bead One R, a new benchtop fusion model with electrical resistance heating. In this application note, we assess the fusion performance of the Bead One R for cement reference material (JCA-601B). Assessment of precision and accuracy according to ISO 29581-2 showed results on an expert level.


## Introduction

The borate fusion is a sample preparation method widely used in many quality control laboratories of various industries. Borate fusion allows the precise analysis of the elemental composition of a raw material, semi-finished or finished products. Dissolution of the material in a flux e.g., lithium borate eliminates particle size and mineralogical effects. With its features, the new Bead One R is a precise and reliable fusion system for process control meeting the latest standards of modern cement plant operation.

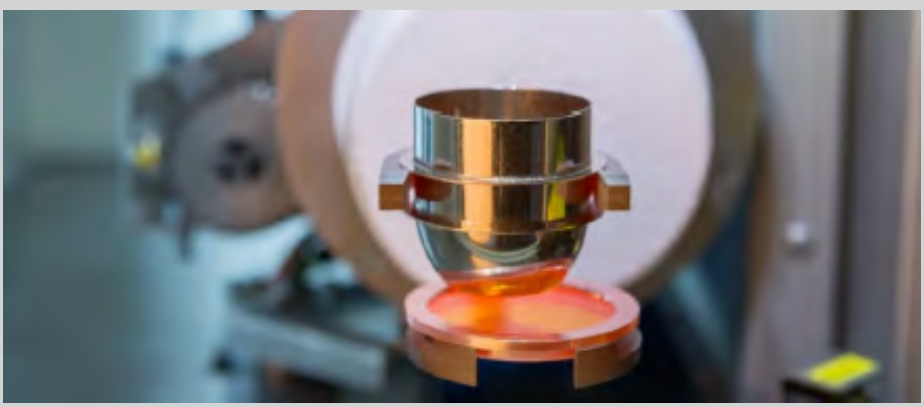
Figure 1: Perfectly homogeneous cement fusion bead

## Method

Weighing precision of sample and flux was at least ± 0.0003 g to minimize preparation error. The sample: flux ratio was 1: 10 resulting in perfectly shaped and homogeneous beads. For a 40 mm diameter bead, 6 g Lithium tetraborate and 0.6 g of sample were used.

The HERZOG Bead One R was used for fusion. For this specific application, a fusion temperature of 1075 °C with a total fusion time of 15 minutes was applied. Loss On Ignition (LOI) was determined according to ISO 26845. Figure 2 shows the platinum crucible and the dish with the melt inside at the passive cooling stage.

Figure 2: Hot platinum crucible and dish after casting



IMP Innovative Solutions

## Results- Calibration

For XRF analysis we used a PANalytical Epsilon 3XL ED XRF. Calibration of the device was set up by using JCA-601B RM with 15 samples. Total measurement time for the XRF was set to 5 minutes for each sample.

Figure 3: Calibration curves for Si, Ca, Al, Fe, Mg und S

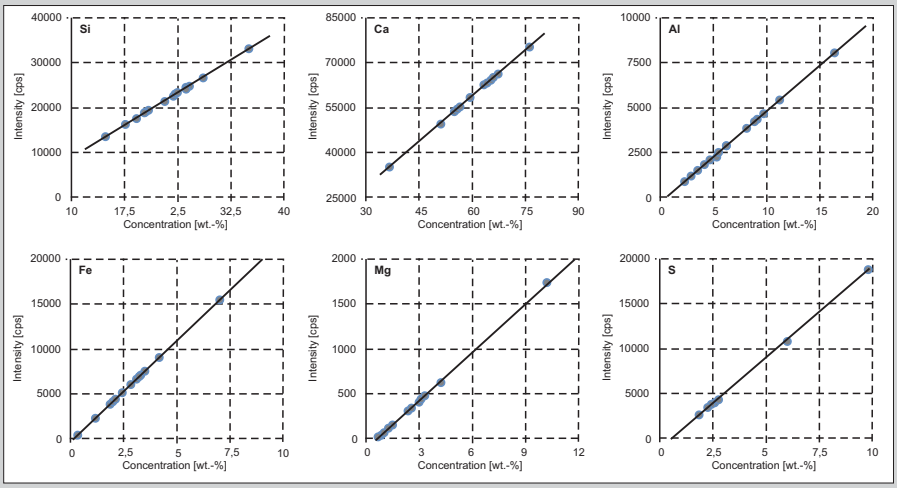



Table 2: Measured elements for cement calibration and squared correlation coefficient

| Element | Emission line | R | Correction |
|---|---|---|---|
| Al | Kα | 0,9995 | - |
| Ca | Kα | 0,9999 | - |
| Fe | Kα | 0,9999 | Fixed α |
| K | Kα | 0,9972 | - |
| Mg | Kα | 0,9999 | - |
| Mn | Kα | 0,9997 | Fixed α |
| Na | ROI | 0,9804 | - |
| P | Kα | 0.9994 | - |
| S | Kα | 0,9998 | - |
| Si | Kα | 0,9995 | - |
| Sr | Kα | 0,9988 | Fixed α |
| Ti | Kα | 0,9994 | Fixed α |

## Validation of method

The fusion method was validated according to ISO 29581-2. For assessment of accuracy and precision, the reference material JCA-601B RM was used. For industrial applications, standard limits are sufficient. For scientific use, expert limits are recommended.

Table 3 shows the results for accuracy and precision using JCA 601B no.14. Both tests show that nearly all elements are not only within the ISO standard but also expert limits. The only exception is sodium with an accuracy level only within the standard limit

Table 3: Results of accuracy and precision test using JCA 601B 14

| Analyte | Na2O | MgO | Ai203 | SiO2 | P2O5 | K2O | CaO | Ti2O | MnO | Fe2O3 | SrO |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Maximum Difference | 0,023 | 0,047 | 0,045 | 0,112 | 0,017 | 0,004 | 0,095 | 0,006 | 0,004 | 0,023 | 0,002 |
| Expert Limit | 0,023 | 0,096 | 0,116 | 0,175 | 0,023 | 0,023 | 0,175 | 0,023 | 0,023 | 0,023 | 0,023 |
| Standard Limit | 0,057 | 0,240 | 0,290 | 0,437 | 0,057 | 0,057 | 0,437 | 0,057 | 0,057 | 0,057 | 0,057 |

| Analyte | Na2O | MgO | Ai203 | SiO2 | P2O5 | K2O | CaO | Ti2O | MnO | Fe2O3 | SrO |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Maximum Difference | 0,050 | 0,086 | 0,147 | 0,168 | 0,015 | 0,013 | 0,125 | 0,010 | 0,003 | 0,015 | 0,004 |
| Expert Limit | 0,020 | 0,120 | 0,150 | 0,200 | 0,020 | 0,020 | 0,200 | 0,020 | 0,020 | 0,020 | 0,020 |
| Standard Limit | 0,050 | 0,300 | 0,350 | 0,500 | 0,050 | 0,050 | 0,500 | 0,050 | 0,050 | 0,050 | 0,050 |

## Discussion

This study on cement samples demonstrates that borate fusion using the Bead One R leads to highly consistent and accurate results. All elements but sodium are within the ISO expert limit.

The deviation of sodium can be explained by its low atomic number and the low EDXRF sensitivity towards small sodium concentrations. Using a WDXRF will certainly meet the requirements for analysis of small sodium concentrations.

The Bead One R is the optimal fusion device for a wide range of applications requiring the highest degree of precision and accuracy. The high concentration range of e.g. silicon and calcium of the calibration allows its application on many different materials.

## References


1. ISO 29581-2 Cement Test methods Part 2: Chemical analysis by X-ray fluorescence
2. ISO 26845 Chemical analysis of refractories - General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods



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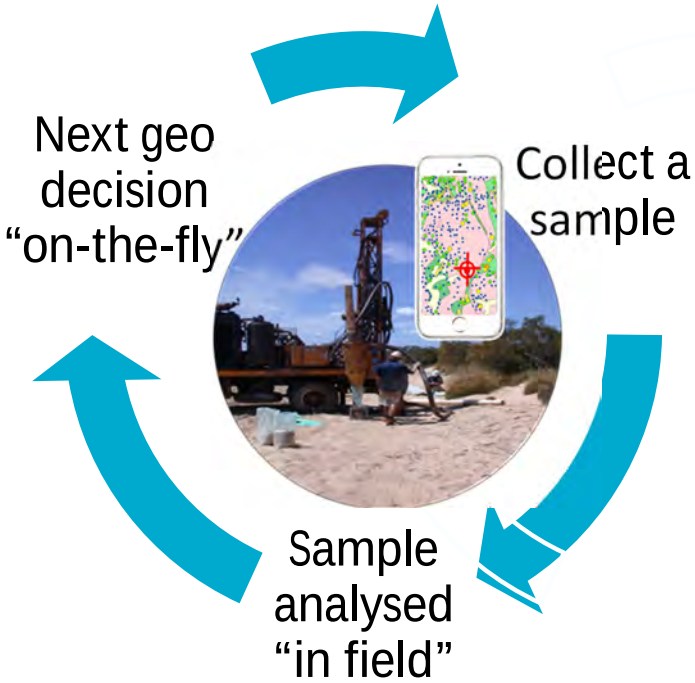


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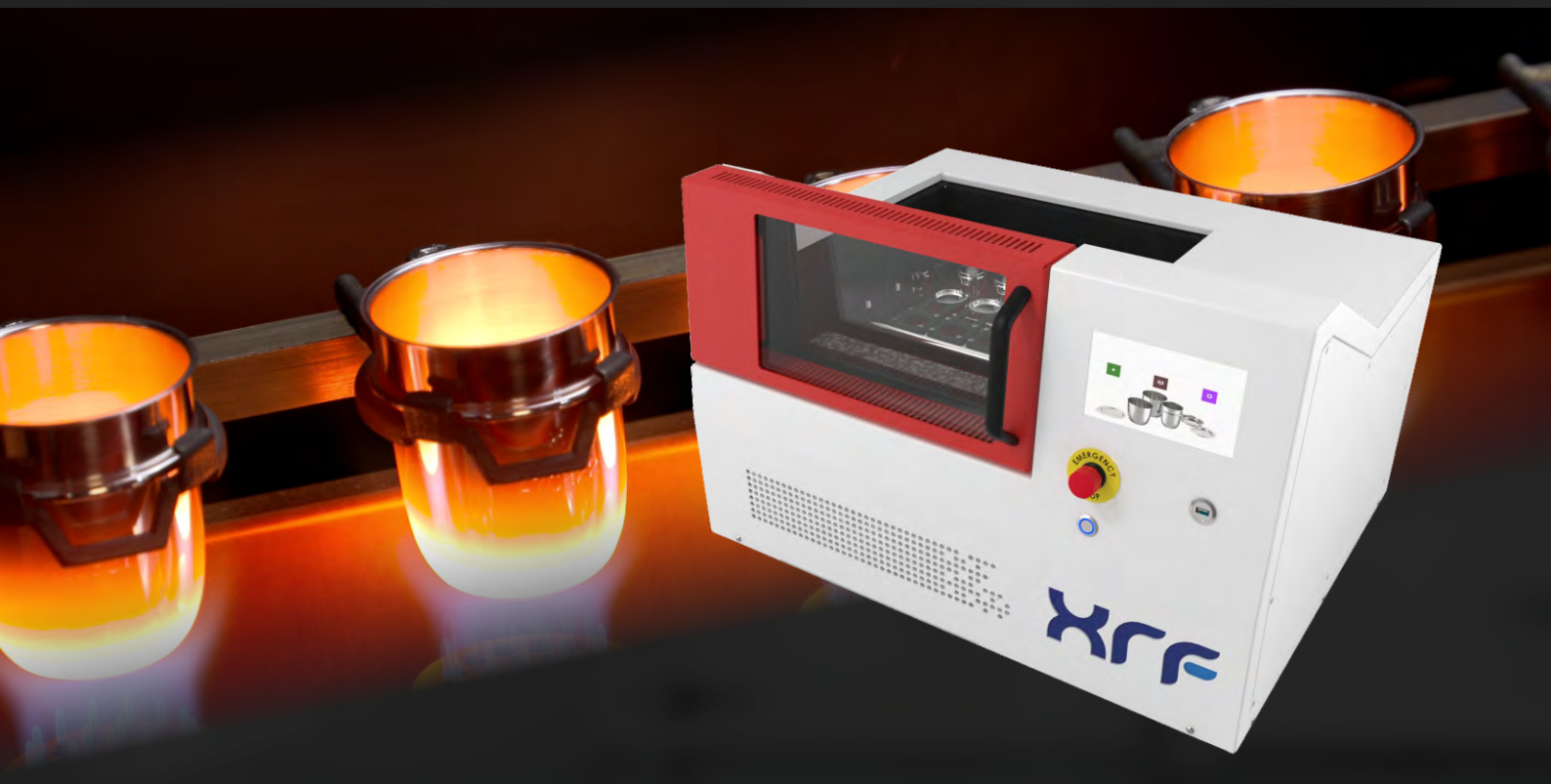

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## New Rigaku XRD for The University of Auckland

**Sydney, Australia, September 4, 2017** - AXT is pleased to announce that they have recently installed a Rigaku Ultimate IV X-ray diffractometer at the University of Auckland in the Faculty of Engineering. The new system will serve as a replacement and upgrade to their ageing system, facilitating researchers involved in a wide variety of endeavours.

Rigaku is recognised as a leader in X-ray analytical instrumentation. The Ultima IV is a highly versatile instrument capable of performing a range of anlaytical experiments to determine composition and phase assemblage. It includes Rigaku's patented Cross Beam Optics (CBO) which allow the user to quickly switch between parallel and focused beam geometries to suit their specific experiments.

The diffractometer is a fundamental piece of research infrastructure that services a large user base. As such, the Ultima IV acquisition was funded internally by the University.

The bulk of the users are postgraduates carrying out high-end research projects, primarily in the area of materials science and advanced materials. The Ultima IV is currently being heavily used with undergraduate project students booking up capacity as they complete their projects. The system will also be used to carry out consulting work for commercial clients.

Dr. Alec Asadov, Technical Officer at the University of Auckland was key to the instrument selection process. "The Ultima IV satisfied our technical requirements and came in at a price we could afford. In particular, this mid-sized system fits the space we had available and it also offered us the flexibility to purchase other attachments and accessories down the track to expand its capabilities", said Dr. Asadov.

Richard Trett, Managing Director at AXT commented, "Rigaku XRD technologies have had a rapid uptake Australia as they have shown unmatched performance and flexibility. This is the first of the current generation of instruments that we have installed in New Zealand and we are confident that the Ultima IV it will provide a high quality platform for University of Auckland researchers to generate publication quality data and perform cutting edge research. We look forward to working with the University of Auckland into the future as they join a host of larger SmartLab XRD installations in the Australia/New Zealand region."

AXT distributes a range of Rigaku instruments in Australia and New Zealand ranging from XRD, XRF and radiographic testing. XRDs constitute part of AXT's portfolio of analytical instruments. For more information on AXT's range of materials testing solutions, please visit www.axt.com.au.

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## University of Queensland to get Unique Single Crystal Diffractometer

**Sydney, Australia, November 20, 2017** - AXT is proud to announce that they will soon be installing a Synergy-S single crystal X-ray diffraction system at the University of Queensland (UQ). This high-end system is manufactured by Rigaku Oxford Diffraction (ROD), the leaders in the field of single crystal and small molecule diffractometers.

The Synergy-S is the latest generation of single crystal diffractometers, and the first instrument to be produced by ROD, who combine the expertise of X-ray analytical powerhouses Rigaku and Agilent, following Rigaku's acquisition of the latter. It features their PhotonJet-S X-ray sources which delivers high intensity flux to a small focal point. The air-cooled design also provides advantages including simplicity over water-cooled sources.


The system specified by the University of Queensland is a world first combining a dual wavelength source (Molybdenum and Silver) and the PILATUS CdTe detector. The detector offers shutterless operation, fast 20Hz sampling rate while providing excellent dynamic range and sensitivity.

Associate Professor Jack Clegg is the Lead Chief Investigator on the ARC LIEF grant application that facilitated the acquisition. When asked why he chose the Synergy-S system, he said, "the system satisfied all our performance requirements. The dual wavelength configuration will allow us to carry out high resolution studies and probe changes in charge density. The large enclosure offers us the flexibility to house lasers and other ancillary devices also allowing high pressure diamond anvil experiments *in situ*. Furthermore, with the ability to integrate N-Helix, we can carry out studies all the way down to 30K (-243°C). With this system we will be able to perform anything from basic single crystal experiments, all the way up to detailed structural analysis of complex metal-organic compounds as well as unique photo crystallography experiments".

Assoc. Prof. Clegg went on to explain that the internally housed lasers will permit dynamic studies to be carried out which will provide a deeper understanding of how changes occur and accelerate their research outcomes through direct observations as opposed to inferred results. It will also enable them to become less reliant on the synchrotron which will also help them to prove hypotheses more quickly.

While the instrument will be utilised by researchers from UQ, QUT and USC ranging from honours students to post doctoral researchers, it is also expected that the unique configuration and capabilities of this instrument will attract users and collaborators from across Australia and around the world. Assoc. Prof. Clegg also remarked that the user-friendly software would enable novice users to easily produce publication quality data.

The appeal of the system is also further reaching with industry interest also expected from areas such as the power industry who deal with sulfide build up in transformers and the mining industry who are interested in the structure of minerals.

Assoc. Prof. Clegg is grateful to the Australian Research Council who provided funding for the instrument through their LIEF (Linkage, Infrastructure, Equipment and Facilities) program.

Richard Trett, Managing Director at AXT also commented, "We pride ourselves in bringing cutting edge solutions to local research facilities supporting Australia's global position as a world leader in scientific research. The unique configuration chosen by UQ goes further than this and is internationally significant for ROD and crystallography. We are excited and honoured to be working with Assoc. Prof. Clegg and supporting UQ's research endeavours."

Single crystal diffractometers from ROD are part of AXT's vast array of analytical instrumentation. For more information on other solutions sourced from their network of suppliers around the, please visit www.axt.com.au.

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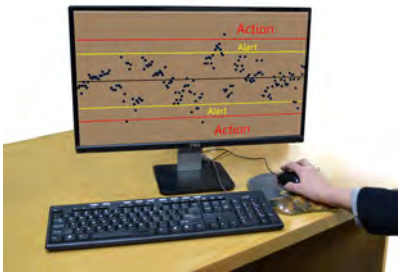
# Premium Standards Produced for Handheld, Desktop and Laboratory XRF's


Resource Reference Materials (Pty) Ltd (RRM) is an international organization focused on and committed to producing the best possible standards for handheld, desktop and laboratory XRF's for the compliance needs of the global resources industries.




## Why Use RRM's XRF Standards?

- RRM provides a comprehensive service to end users of XRF instruments; from hand-held, desktop and more advanced laboratory spetrometers.
- Manufacture project specific standards and/or certified reference materials, from your materials, for calibration and performance monitoring of your instrument.
- 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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