



Australian X-ray Analytical Association

Newsletter Issue 3 2019

President's Address

Dear AXAA Members and Friends,

As the end of 2019 rapidly approaches, so does the deadline (13th December) for [abstract submission](#) for AXAA-2020! Now is the time to finalise your abstract(s) to make sure that you are part of a stimulating and diverse scientific programme. In addition to our normal conference sessions which include an outstanding line-up of Plenary and Invited presentations, AXAA-2020 will include a memorial session for the late Keith Norrish (speakers by arrangement only), and a Technology Forum showcasing the latest developments and products from leaders in the X-ray industry. Sponsorship and exhibition opportunities are still available; please contact the [AXAA-2020 Conference Secretariat](#) if your organisation is interested in one of these exciting opportunities.

The AXAA-2020 programme will also include the presentation of three prestigious awards; the Keith Norrish AXAA Award for Excellence in X-ray Fluorescence Analysis, the Bob Cheary AXAA Award for Excellence in Diffraction analysis, and the Malvern Panalytical Award for Excellence in Analysis by an Early Career Scientist. Now is the time for nominating one or more of your colleagues; a list of previous winners is available [here](#). Please read on for further details of these awards and the nomination and selection process. Nominations are due by 31st January 2020.

One of the highlights of AXAA's activities each year is the line-up of Student Seminars events held in various states. In 2019 events were held in VIC ("Something to Bragg About"), NSW ("Scattering Matters"), WA

("Bright Scatterers") and QLD ("Braggers"). Please read on for a wrap-up of each event. First prize at each event was a full bursary to attend and present at AXAA-2020, and so we will all have the opportunity to learn about the high quality work of the up-and-coming stars of X-ray and neutron analysis.

Our General Meeting was held on 14th November at CSIRO Mineral Resources in Clayton, VIC. Thank you to all of the AXAA members who participated. Meeting minutes will be circulated before the next General Meeting, to be held 30th April 2020 on the Gold Coast (final venue TBC). One of the agenda items at this April General Meeting will be the election of the next AXAA Council for the period 2020-2023. Now is the time to be thinking about nominations for members of the next Council; this is your chance to be intimately involved in the organisation and direction setting of AXAA and the greater number of dynamic and enthusiastic people involved, the better to ensure a vibrant and diverse community. The AXAA National Council consists of a President, Vice-President, Secretary, Treasurer and not less than two other people. Three nominations will be made by the outgoing Council, which will be confirmed at a National Council Meeting to be held early in 2020 and subsequently communicated to members. Nominations are also accepted from the AXAA membership, by email to the AXAA Secretary, until 23rd April 2020.

Finally, on behalf of the AXAA National Council I wish all AXAA members and friends a safe and enjoyable festive season.

Nathan Webster
AXAA President

AXAA-2020
29 APRIL – 1 MAY 2020
AUSTRALIAN X-RAY ANALYTICAL ASSOCIATION
CONFERENCE & EXHIBITION
Gold Coast, QLD, Australia

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AXAA QLD Student Seminar Day – “Braggers”

The inaugural AXAA QLD Student Seminars sponsored by AXT took place on the 21st November 2019 at the University of Queensland. Presentations were given by five PhD students on topics ranging from conversion of mining waste to zeolites and their characterisation by quantitative X-ray diffraction, to optimising layer thickness in flexible OLEDs and characterisation using X-ray reflectometry.



Figure 1. Organisers and presenters at the AXAA QLD Student Seminar event.

Prizes were awarded to Saiqa Muneer (School of Chemistry and Physics, Queensland University of Technology), Maggie-Anne Harvey (Centre for Mined Land Rehabilitation, The University of Queensland) and Dumindu Siriwardena (School of Chemistry, Queensland University of Technology) for their presentations on application of XRD in the analyses of Meropenem in prepared dry powder inhaler formulation, use of μ XRF to describe the distribution of selenium in *Neptunia amplexicaulis* from Central Queensland, and characterisation of electrochemical and structural properties of iron-substituted sodium ruthenate cathodes for sodium-ion batteries, respectively. Many thanks to Associate Professor Kevin Jack (The University of Queensland) and Dr Tony Wang (Queensland University of Technology) for their assistance in the organisation and running of the event.

WA – “Bright Scatterers”

Eleven students from three WA universities presented on a range of subject matters. Judging was very tough and in the end the winners were;

- 1st prize: Maria Sofia Hazarabedian (Curtin Uni) - "Phase identification of Intergranular precipitates in Precipitation Hardening Nickel Alloys"
- 2nd prize: Kyran Williamson (Curtin Uni) - "Metal carbonates as Thermochemical Energy Storage Materials"
- 3rd prize: Ashley Hollings (Curtin Uni) - "Spectroscopic Studies of Brain Zinc Homeostasis and Its Role During Cognitive Decline and Ageing"



Figure 2. Above: Organisers and presenters at the AXAA WA Student Seminar event. Bottom left to right: AXAA National Council members Talitha Santini and Will Rickard presenting awards to winners Maria Sofia, Kyran, and Ashley.

A big thanks to AXT for sponsoring the afternoon tea and sundowner!

Special thanks also to UWA PhD student Grace Scullet-Dean for assisting with the organisation of the event.

AXAA VIC Student Seminar Day– “Something to Bragg About”

The AXAA VIC Student Seminars took place on the 31st October at the CSIRO Laboratories in Clayton. Presentations were given by six PhD students to an audience of 20 members of the AXAA community, on topics ranging from mineral crystallography to catalysis to interactions of nanoparticles with cells.

The standard of each presentation was outstanding, with a diverse range of analysis techniques being used at a very high level. A comment from the judges was that every year it gets harder to judge the best presentations.

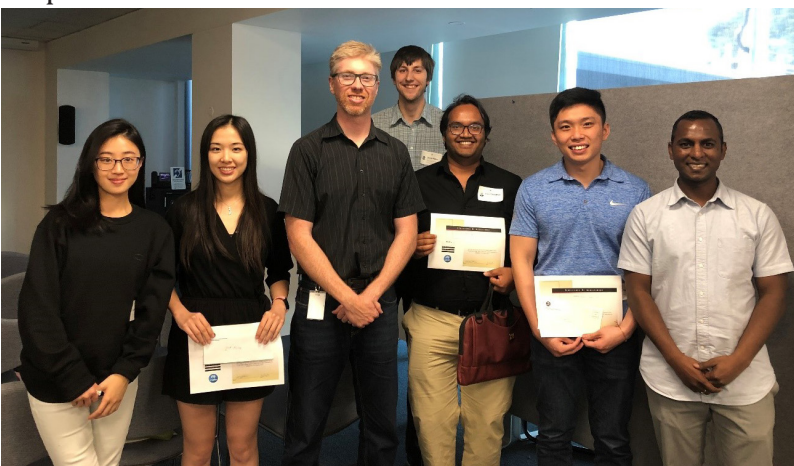


Figure 3. AXAA President Nathan Webster with prize winners and presenters at the AXAA VIC Student Seminars event.

Prizes were awarded to Alok Chaudhari (School of Earth, Atmosphere and Environment, Monash University), Cindy Xiao (Monash Institute of Pharmaceutical Sciences) and Wen Liang Tan (Department of Materials Engineering, Monash University) for their presentations on understanding complex mineral replacement mechanism and kinetics, engineering elongated polymeric nanocapsules for prolonged circulation and improved controlled release, and oriented attachment as the mechanism for microstructure evolution in chloride-derived hybrid perovskite thin films, respectively.

First prize recipient Alok Chaudhari summarises his research below:

Shining a light on mineral transformations using in-situ and ex-situ experimental methods

Alok Chaudhari (Monash University)

Mineral transformation, where one mineral is replaced by another mineral, has led to the formation of many important economic mineral deposits in nature. Many of these reactions are fluid-driven and proceed via a coupled dissolution and reprecipitation reaction^{1,2} (CDR) mechanism. My research is focused on investigating mineral replacement reactions in the copper-sulfide system under mild hydrothermal conditions. Understanding the mechanism and kinetics of complex CDR reactions can be broken down in two



Figure 4. Classical experimental high – pressure and high temperature vessels, titanium pressure reactors (left) and gold bag (right) used in rocking hydrothermal autoclaves.

parts: a) investigating the evolution in mineralogy during the CDR reaction and b) tracking changes in the fluid chemistry at hydrothermal conditions, including metal concentrations and redox state. During my PhD project, I have adopted a laboratory-based experimental approach (Fig. 4) coupled with *in-situ* experiments at the beamlines of the Australian Synchrotron to gain a comprehensive understanding on the nature of CDR mineral replacement reaction copper sulfide system.

One of the key and often ignored aspects of mineral replacement reactions is the role of intermediate reaction species which are generated during the reactions but are not quenchable.

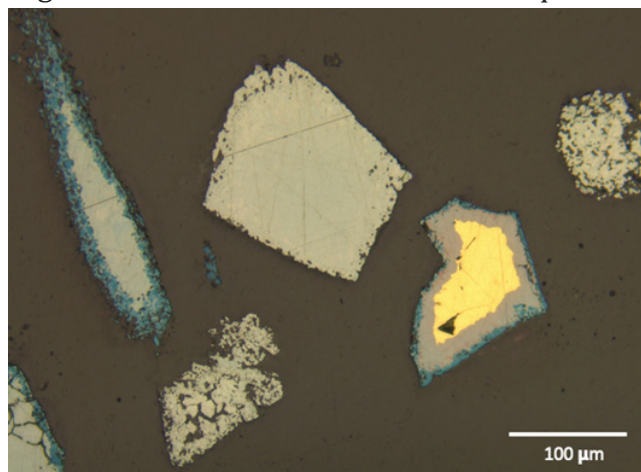


Figure 5. Reflected light microscopy images showing the fluid driven replacement texture generated during the replacement of chalcopyrite (yellow) by digenite (light blue)/covellite (dark blue) under hydrothermal conditions (200 °C – 4 hours).

Ex-situ laboratory quenched experiments provide limited information about the fate of these intermediate reaction species, as some intermediate species are

quenched, and other metastable species revert back to their lower energy state thus requiring high-sensitivity *in-situ* techniques to characterize these species. Synchrotron-based powder diffraction (XRD) is one of the few techniques that allows to investigate such reaction *in-situ* under hydrothermal conditions³ (T up to 240 °C, pressure to 40 bar). The method also can provide high quality, high resolution information about the kinetics of hydrothermal reactions. For example, a number of phases in the copper sulfide system undergo phase transition at higher temperatures^{4,5}; digenite ($\text{Cu}_{1.8}\text{S}$) transforms to high-digenite⁶ at about 83 °C and chalcocite undergoes a phase transition to form high-chalcocite⁴ at 103 °C. These intermediate phases could play a decisive role during the reaction and control the nature and composition of the final products.

Another important aspect of CDR reactions is the role of dissolved ionic species in catalyzing these reactions. While *in-situ* XRD provides the necessary information on the mineralogical evolution during the mineral replacement, it does not effectively tell us about the concentrations of metals in the fluid and their oxidation state. Therefore, we performed the first *in-situ* X-ray Absorption Spectroscopy (XAS) experiments aimed at providing us with direct evidence for the evolution of Fe and Cu concentrations and oxidation state during a complex mineral replacement reaction, and to decipher the role of solution chemistry in controlling these transformations. While the *in-situ* XRD experiments were conducted using capillaries, high-quality XAS data were obtained using the high-volume mAESTRO⁷ autoclave system developed by our group (Fig. 6).



Figure 6. High pressure – temperature mAESTRO cell for XAS spectroscopy at the Australian Synchrotron.

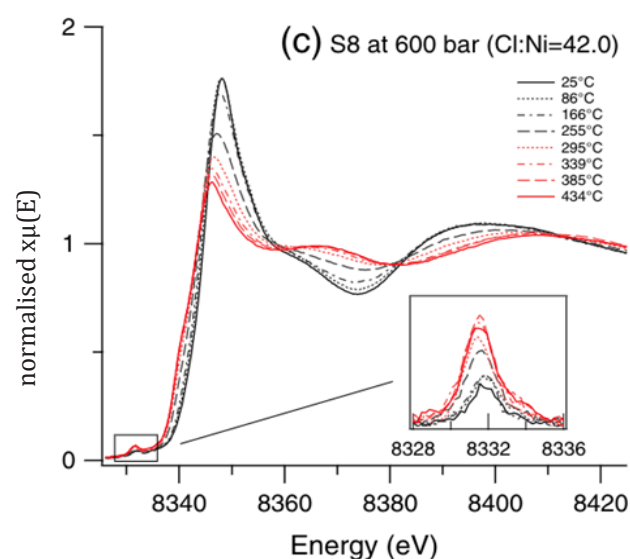


Figure 7. Spectral evolution of nickel complexes⁷ as a function of temperature (25 °C – 483 °C) acquired using mAESTRO cell at the XAS beamline Australian Synchrotron.

The mAESTRO autoclave is demonstrated in Tian *et al.* (2012).⁷ The evolution of nickel complexes is investigated in hydrothermal brines from 25 °C - 439 °C and from 400 bar – 600 bar. A change in coordination geometry of Ni(II) from octahedral to tetrahedral (Fig. 7) was observed around 200 °C. The change in Ni (II) coordination at higher temperatures promotes the remobilization of Ni from mafic and ultramafic rocks leading to the formation of economic deposits of Ni sulfides⁸ (e.g., Auebury deposits, Tasmania). Similarly, like Ni, the ability to determine the speciation and coordination geometry of Fe and Cu, at high temperatures proved to be essential for investigating the mechanism of fluid-driven mineral replacement reactions in the copper sulfide.

The combination of *in-situ* XRD and *in-situ* XAS techniques is a powerful tool to investigate the reaction mechanism and kinetics of complex fluid-driven CDR reactions or a wide variety of fluid-mediated natural or engineered systems.

References

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NSW – “Scattering Matters”

The NSW Student Seminars took place on November 8th, at ANSTO Lucas Heights. There was an excellent turnout with 9 presenters and about 25 attendees.



Figure 8. Organisers and presenters at the AXAA NSW Student Seminar event.

The NSW prizes were awarded as follows:

- 1st prize: Daniel Fanna, Western Sydney
- 2nd prize: Bryce Mullins, University of Sydney, “Disorder by Design: Energy, Pyrochlores and the Art of ‘Stuffing’”
- 3rd prize: Emily Yap, University of New South Wales, “Micro X-ray Generators for Portable X-ray Analysis”

First prize winner Daniel Fanna shares his research below:

Crystal structure diversity of Zn²⁺ complexes with a multi-dentate flexible Tris base salicylic ligand

Daniel J. Fanna^{a,b} and Feng Li^a

^aSchool of Science and Health, Western Sydney University

^bAdvanced Materials Characterisation Facility, Western Sydney University

Introduction

A common approach for the synthesis of supramolecular architectures is metal-directed self-assembly. This method utilises carefully selected metal ions and ligand donor groups to promote the construction of larger metallo-supramolecular assemblies. As the preferred coordination environment of many metal ions are well-understood, it is possible to promote the assembly of a desired architecture by predicting the interactions that will take place between a metal and a coordinating ligand. Subsequently, this

well-established approach has allowed the synthesis of many unique materials, especially when using metal ions with strict preferred coordination environments and ligands with rigid and fewer binding sites. On the other hand, coordination interactions become difficult to predict when using multi-dentate ligands with several flexible binding groups and metal ions with numerous stable coordination conformations. Investigating metal-directed self-assembly interactions with systems that are difficult to predict is important as this arms future researchers with knowledge on the potential complexations that may take place, which in turn helps chemists promote the formation of a desired metallo-supramolecular assembly.

Herein, the complexation between a multi-dentate ligand with flexible coordination groups and various Zn²⁺ salts (chloride, acetate and perchlorate) was investigated by single-crystal X-ray diffraction (SC-XRD). Zinc was selected as a metal centre because it is known for producing stable complexes in a variety of coordination geometries and additionally, due to its d¹⁰ electron configuration, it is popular in the synthesis of optical materials. This work also incorporates the salicylic group as it is well-known for its strong metal ion binding and favourable optical properties, making it an ideal group to be incorporated in a chemosensor for metal detection applications.

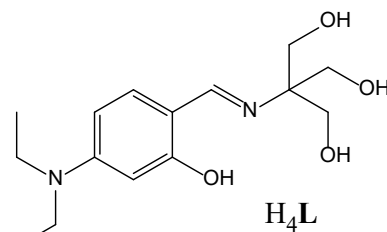


Figure 9. The ligand (E)-2-((4-(diethylamino)-2-hydroxybenzylidene)-amino)-2-(hydroxymethyl)propane-1,3-diol (H₄L).¹

Experimental section

A brief overview of the synthetic methods employed is given below, while detailed experimental procedures for this work along with several characterisation techniques can be found in the published work by Li, et al. 2019.¹ The ligand (E)-2-((4-(diethylamino)-2-hydroxybenzylidene)-amino)-2-(hydroxymethyl)propane-1,3-diol (H₄L, Figure 9) was synthesised by a Schiff base condensation between 4-(diethylamino) salicylaldehyde and Tris base with synthesis confirmed by ¹H and ¹³C NMR as well as electrospray ionisation high-resolution mass spectrometry (ESI-HRMS) techniques. Complexes **1-4** were synthesised using a

similar synthetic procedure where H_4L and various Zn^{2+} salts (**1** from $ZnCl_2$, **2** from $Zn(CH_3COO)_2$, **3** and **4** from $Zn(ClO_4)_4$) were combined as a 1:1 stoichiometry in MeOH, and where appropriate H_4L was deprotonated with trimethylamine (complexes **1**, **3** & **4**). Crystals of complexes **1**, **2** & **4** were grown by slow evaporation of the methanolic reaction mixture, while complex **3** was grown by the vapour diffusion of diethyl ether into the methanolic reaction mixture. Single-crystal XRD was then conducted on the MX1 and MX2 beamlines of the Australian Synchrotron at 100(2) K using double-crystal silicon (111) monochromated synchrotron radiation.^{2,3}

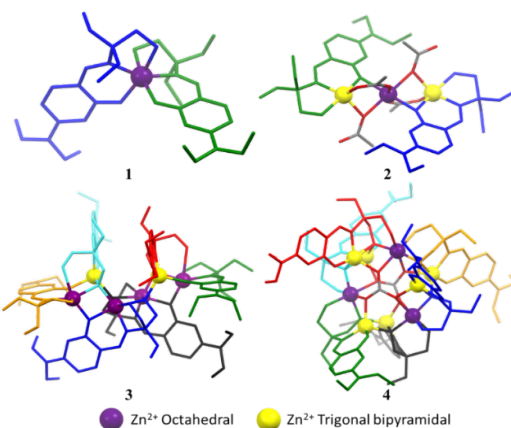
Results discussion

Despite utilising a similar synthetic procedure, complexes **1-4** gave four different crystal structures in which the resulting assemblies are influenced by the anion identity and the crystallisation method used (Figure 10). The simplest structure, **1**, gave $(H_3L)_2Zn$, a mononuclear assembly comprised of two H_3L ligands coordinating to an octahedral Zn^{2+} centre with chiral coordination giving a tetragonal $P4_12_12$ space group.

Complex **2** gave $[(H_3L)_2Zn_3(CH_3COO)_4]$ a trinuclear assembly comprised of two H_3L ligands coordinating to three Zn^{2+} metal centres in a linear manner with acetate bridging between Zn^{2+} centres. In this complex Zn^{2+} metal centres were observed in two different coordination geometries. The central Zn^{2+} metal centre is in an octahedral coordination geometry, while the two external Zn^{2+} metal centres are trigonal bipyramidal.

In the case of H_4L complexed with $Zn(ClO_4)_4$, two different structures were observed, and this was determined by the crystallisation method.

Figure 10. Crystal structures for complexes **1-4**. For clarity, H-atoms, non-coordinating anions and solvent molecules have been removed, individual H_4L ligands have been also coloured. Zinc centres in an octahedral geometry are shown in purple, while trigonal bipyramidal geometries are shown in yellow. Adapted from Li, et al. 2019.¹



Crystallisation via vapour diffusion in a low air environment yielded complex **3** $[(H_2L)_4(H_3L)_2Zn_6](ClO_4)_4$ a hexanuclear assembly comprised of six ligands in two different protonation states coordinated to four octahedral and two trigonal bipyramidal Zn^{2+} metal centres. In contrast, crystallisation conducted via a slow evaporation of a similar methanolic experimental mixture to **3** gave complex **4** $[(H_2L)_6Zn_9(CO_3)_2](ClO_4)_2$ a nonanuclear architecture where six H_2L ligands coordinate to nine Zn^{2+} metal centres, again with Zn^{2+} metal centres in octahedral and trigonal bipyramidal geometries. Interestingly, in complex **4** two molecules of CO_3^{2-} were observed coordinating to the central Zn^{2+} metal centres, it is likely that these CO_3^{2-} molecules originated from atmospheric CO_2 as the slow evaporation was undertaken in air.

Conclusion

The structural diversity observed in this work stems from the ability of the hydroxyl and phenolic groups of H_4L to coordinate various numbers of metal centres. This, in conjunction with the ability for Zn^{2+} metal ions to adopt several different coordination geometries and numbers, allows for minor synthetic variations such as the anions used and the presence or absence of atmospheric components (CO_3^{2-}) to cause significant crystal structure variations. From this investigation we have been able to understand the molecular interactions that mediate coordination of octahedral and trigonal bipyramidal coordination systems. This, in turn, has allowed our group to develop a salicylic based chemosensor for applications in Cu^{2+} sensing of drinking water⁴ (Figure 11) and soils.⁵

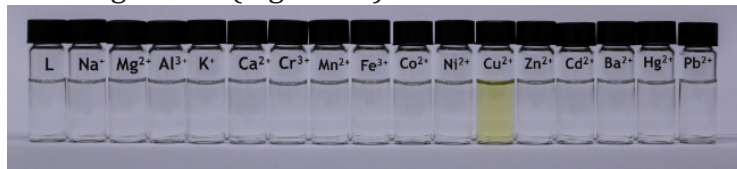


Figure 11. The colourimetric detection of Cu^{2+} in a MeOH/ H_2O (1:1, v/v; HEPES 20 mM; pH = 7.00) mixture using a salicylaldehyde diamionaphthalene based ligand by the Li group.⁴

References

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Study of battery materials on a laboratory diffractometer using high energy X-rays

Olga Narygina (Malvern Panalytical)

Introduction

Currently Li-ion batteries are present in any portable electronics device and in electrical vehicles. Lithium provides a large part of the currently available electrode materials. The availability of Li on Earth remains small and thus alternative elements such as Na or Mg are being considered to replace Li. Development of low-cost electrode materials using alternative elements is quickly becoming a topic of high interest in the scientific community.

X-ray diffraction and scattering techniques are powerful tools for the study of battery materials (Liu et al., 2014; Talaie et al., 2015). X-ray diffraction (XRD) allows to identify and quantify the different

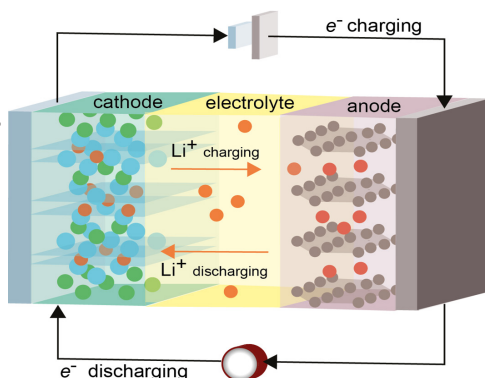


Figure 12. Simplified schematic drawing of Li-ion battery cell.

crystallographic phases present in battery electrodes, refine their structural properties, determine the average crystallite size and lattice strain. It is also possible to perform in situ and in operando XRD measurements of the complete battery cells to monitor the chemical reactions during charge/discharge (Buchberger et al., 2015; Sharma et al., 2015; Talaie et al., 2017). In operando battery experiments can be done in either reflection or transmission geometry, however, the latter is preferred as it allows to probe full sample volume. To ensure sufficient transmission through a battery cell a high energy radiation is required (e.g. Mo or Ag X-ray anodes). Here we present an example of such in operando XRD study of Li-NMC (lithium-manganese-cobalt oxide) prismatic battery cell in transmission geometry.

Methods

In operando XRD measurements were performed using a Malvern Panalytical Empyrean diffractometer equipped with Ag X-ray anode, focusing mirror, sample stage with XYZ translations and GaliPIX3D solid-state hybrid pixel detector with CdTe sensor with 100% efficiency for corresponding photon.

Four complete charge/discharge cycles between 3.2 V and 4.2 V were monitored using 5 minutes XRD scans in the 5-30 2Theta range.

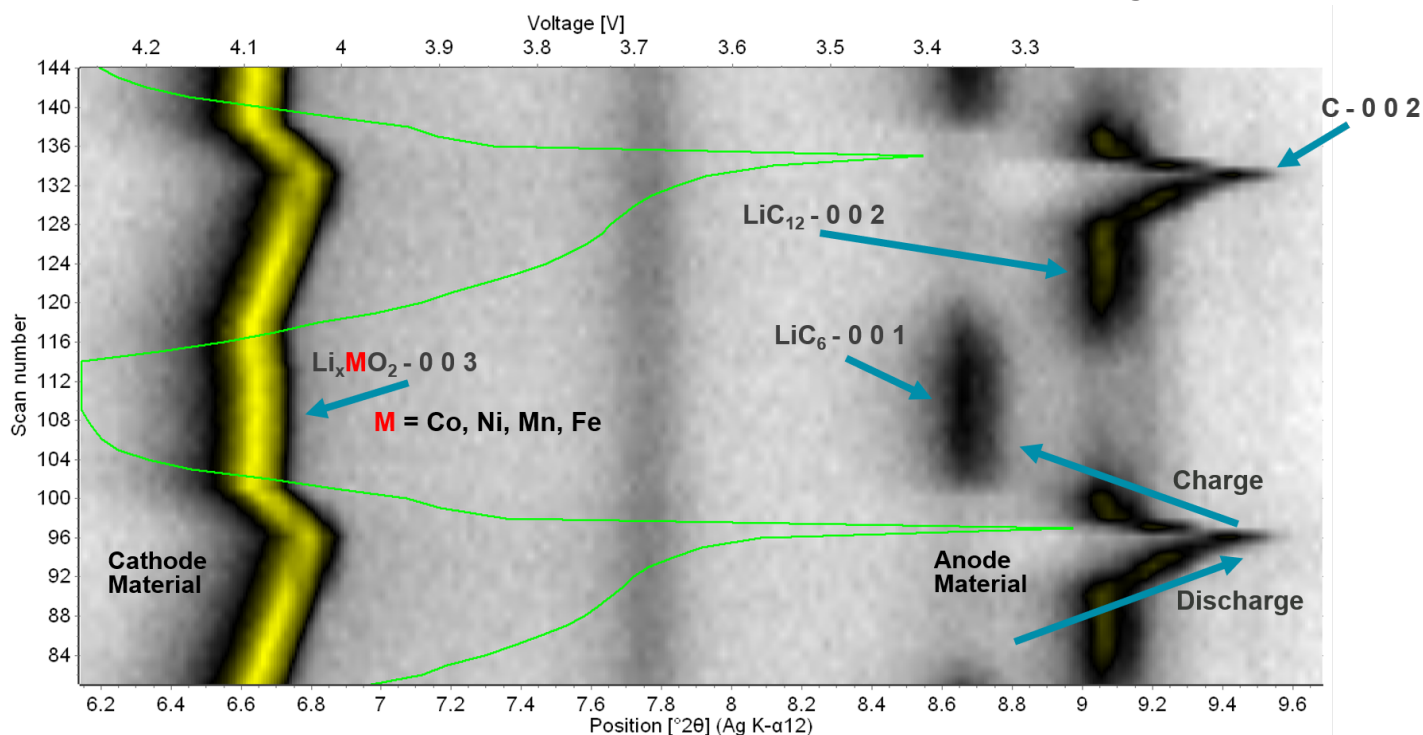


Figure 13. Zoom-on XRD data set recorded during two cycles. The green line shows applied voltage. Characteristic Bragg reflections of cathode and anode materials are marked.

The total time of the experiment was 14 hours with 166 scans measured in total. HighScore Plus software (Degen et al., 2014) was used for the data analysis.

Results and discussion

The XRD data set recorded during two charge/discharge cycles of Li-NMC prismatic cell is shown in Figure 12. The main Bragg reflections of cathode and anode materials are marked. The low intensity XRD reflection at ~ 7.75 deg 2Theta corresponds to a polymer separator between the cathode and anode. The position of (003) reflection of Li_xMO_2 ($M = \text{Co, Ni, Mn, Fe}$) varies between ~ 6.6 and 6.8 deg 2Theta during charge/discharge, signifying change of the Li_xMO_2 unit cell as Li^+ ions move in and out the cathode.

Anode material undergoes several phase transformations, starting from a pure carbon in the beginning of the charging cycle, which first transforms into Li-depleted carbide, LiC_{12} , and then into Li-enriched carbide, LiC_6 . The reversed sequence of phase transformations is observed on the following discharge cycle.

For the quantitative description of the observed phase transformations the data set was further analyzed using HighScore Plus suite. The entire data set was reduced to a single XRD pattern by summation. The resulting representative of the set XRD pattern was used for identification of all crystalline phases detected during the in operando experiment.

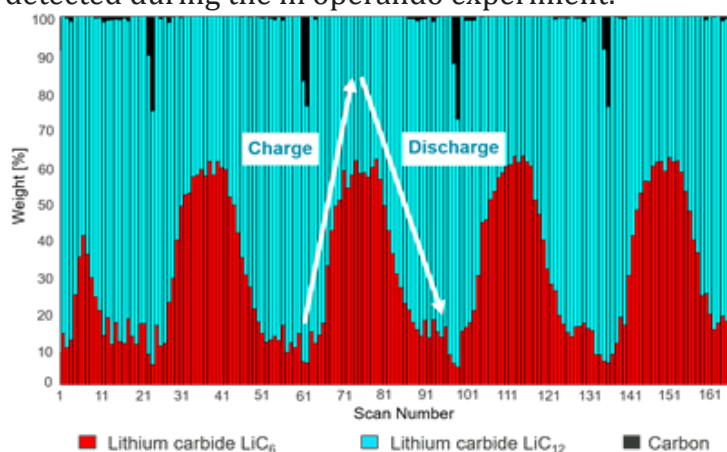


Figure 14. Results of the quantitative phase analysis performed using mixed fit model.

In total seven phases were identified. Apart from Li_xMO_2 , LiC_{12} , LiC_6 and C, marked in Figure 13, copper and aluminium current collectors as well as polymer separator between the electrodes were identified. For the quantitative model we used mixed fit approach.

The amounts of copper, aluminium and Li_xMO_2 do not change during the experiment, those phases were fitted using Pawley fit (Pawley, 1981), whilst anode phases (LiC_{12} , LiC_6 and C) were fitted using Rietveld method (Rietveld, 1969). The quantitative fitting model, optimized using single XRD pattern, representing the entire data set, was applied to the 166 recorded XRD patterns using sequential automatic fitting approach. The overview of the obtained quantitative results is shown in Figure 14. The relative amounts of C, LiC_6 and LiC_{12} can be followed over the four charge/discharge cycles performed in the experiment. No significant changes of the relative phase amounts for a given stage of the cycle was observed. This signifies consistent performance of the analyzed prismatic cell during the experiment.

Conclusion

Phase transformations occurring during charge/discharge of commercially available prismatic battery cell were monitored using in operando XRD in transmission geometry performed on a laboratory diffractometer. The high energy of Ag X-ray anode provides sufficient transmission through the sample and allows to record high quality XRD data suitable for the accurate quantitative analysis. Using mixed fitting approach, the phase transformation sequence undergone by the battery anode was quantitatively described. No significant changes in the relative amounts of the anode phases (LiC_6 , LiC_{12} and carbon) were observed over the four performed cycles, suggesting reproducible performance of the prismatic cell during the experiment.

Further studies of the prismatic battery cells as well as pouch cells is planned, including both in operando and *in situ* experiments at variable temperatures.

If you want to learn more about investigation of battery materials using X-rays, watch the educational (free) webinar "Solving the challenges of Na-ion battery electrodes" - <http://bit.ly/34mKJz9>

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Inviting Nominations for AXAA Awards for Excellence

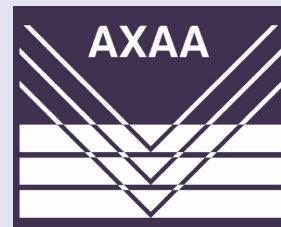
Please consider nominating your colleagues for the following awards, to be presented at AXAA-2020:

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

XRD – Bob Cheary AXAA Award for Excellence in Diffraction Analysis

[Click here for details.](#)

These two awards, one for XRF and one for diffraction, are for significant long-term contributions to analysis, rather than say a single paper, and will perpetuate the contribution of the person after whom the award is named.



In addition, a Malvern Panalytical-sponsored Early Career Researcher award will be awarded for excellence in analytical science of interest to AXAA membership. The award will celebrate “outstanding application of laboratory and/or major radiation facility technique(s) so as to achieve significant impact in a field of endeavour”. The recipient will not be limited to having used any specific brand of instrument.

[Click here for details.](#)

2020 Reynolds Cup Competition

The Clay Minerals Society's 10th Biennial Reynolds Cup (2020) competition for quantitative mineral analysis is now open!

The competition is open to anyone interested in quantitative mineral analysis, with particular emphasis on clay mineralogy.

Information about the competition including guidelines, previous winners, key recommendations, and how to participate can be found [here](#).



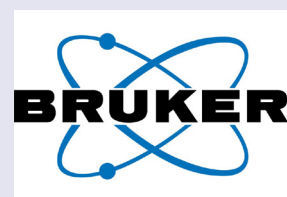
XAS workshop - remote access

The X-ray Absorption Spectroscopy (XAS) team at the Australian Synchrotron will host a workshop on XAS Data Analysis on December 12-13th. While registrations have closed, we are running a remote access option via Zoom link. A number of institutions will be hosting group ZOOM sessions across Australia. If you have XAS data, and would like to know more about how to analyse it, you may contact as-xas@ansto.gov.au to request access. To be considered, please include a paragraph detailing your XAS experience and an explanation of how this workshop will directly benefit your work.

TOPAS Workshop at the AXAA 2020 Meeting, Brisbane, 27-28 April 2020

We are planning a 2-day TOPAS workshop in conjunction with the upcoming AXAA 2020 Meeting. Topics covered will include data quality, quantitative phase analysis and more. This workshop would be more suited for entry-level / intermediate TOPAS users.

Register your interest [here](#), now.



Bruker SPECTRAPlus V3/V4 Training

Customers from the cement and mining industries recently attended a comprehensive 4-day training on XRF with the SPECTRAPlus software package at the Bruker office.

On the first day, participants received an introduction to the theory of XRF, and learnt the basics and benefits of fusions for XRF analysis. The following days were more focused on the SPECTRAPlus software package, which enables XRF users to achieve maximum analytical performance from the wavelength dispersive X-ray fluorescence spectrometers and provides the best support for all kind of analytical tasks.

Participants completed the training equipped with detailed knowledge of methods and practices of XRF, and gained an optimum understanding of both Bruker AXS XRF hardware and software. They also benefited from interacting with our XRF experts, Elvy Grigolato from Bruker Australia and Danny Verbeteen from XRF Scientific, who have both accumulated decades of experience on XRF and analytical chemistry.

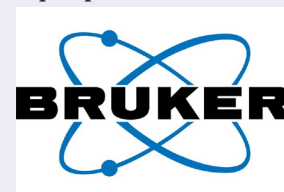


IDEAL Lunchtime Workshop at AsCA 2019 in Singapore, 17 December, 12:00 – 14:00

Meet us at the upcoming AsCA 2019, held at U Town, National University of Singapore from 16-19 December 2019. We are organizing a lunchtime workshop on IDEAL. The new IDEAL routine, the extension of the independent atom model leading to better crystallographic models, is available as part of the APEX3 package. This workshop is to familiarize existing and new users with the package.

Developers and application scientists will introduce you to this exciting new tool with examples. In addition, the workshop will provide an excellent opportunity to process your own data. Bring your laptop with the latest APEX3 version installed. Requests for a demo software license can be made [here](#).

Please [register here for the IDEAL workshop](#) to secure your seat. This workshop is complimentary, but limited seats are available.



Workshop – Single Crystal Data Processing Using CrysAlisPro

If you are involved with the determination of single crystal structures, you should attend this workshop. Dr. Pierre Le Magueres, Director of Scientific Support at Rigaku will lead a workshop and demonstrations on determining the structure of complex and difficult single crystals using CrysAlisPro.

Date: Tuesday December 17, Time: 1-3pm

Venue: Rm 67-101/University of Wollongong

Topics covered:

- 'Regular' data processing
- Indexing/processing of a twinned data set
- Diagnosing/indexing a modulated data set
- Dealing with a data set with heavy icing
- Handling data from a crystal that moved
- Importing data from other programs

Register at <https://www.axt.com.au/pardot-form/crystalispro-workshop-ic19/>



AXAA Website and Contacts

Please visit our website, www.axaa.org, for further information, or follow us on Twitter [@axaa_org](https://twitter.com/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 send the membership form from the [AXAA website](http://www.axaa.org), and a short statement about how they intend to contribute to the organisation, to the National Council Secretary Mark Styles.

AXAA Resource Centre

There are a range of resources available on the [AXAA website](http://www.axaa.org), 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 2020. Please feel free to send contributions for the newsletter to Jessica Hamilton 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 Jessica Hamilton at ausxray@gmail.com.

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



Combined Small spot Elemental XRF and Phase XRD Analysis of Meteorite

Introduction

Meteorites are records of the solar system formation. Several meteorites have not gone through significant geological transformations over billions of years and have remained intact since the formation of the solar system. Knowing more about the composition and mineral phases of meteorites bring us a step closer to understanding how the solar system was formed.

In this study, small spot analysis and elemental distribution mapping of the Northwest-Africa 2086 (NWA 2086) CV3 meteorite were conducted. Qualitative mapping and semi-quantitative spot elemental composition analyses were compared to phase identification using X-ray diffraction for cross verification. The qualitative mapping results show that elements such as Al and Ca are abundant in the inclusion region while Fe and Cr are more prominent in the meteorite body. Combined XRF analysis and XRD phase identification show the presence of sodalite in addition to variations of olivine, pyroxene, and spinel in the rim and the core of calcium-aluminum rich inclusions (CAIs). The sodalite is an indication of early Na-Cl metasomatic processes in the solar nebula.

The studied slice as shown in figure 1 has a maximum dimension of 28 x 13 x 2 mm. NWA 2086 was found in 2003 with a total known weight of 780 g. The meteorite was classified as a carbonaceous chondrite of the CV3 group (Russel et al., 2005). This group of meteorites is characterized by the presence of chondrules and calcium-aluminum rich inclusions (CAIs) in a dark colored matrix.

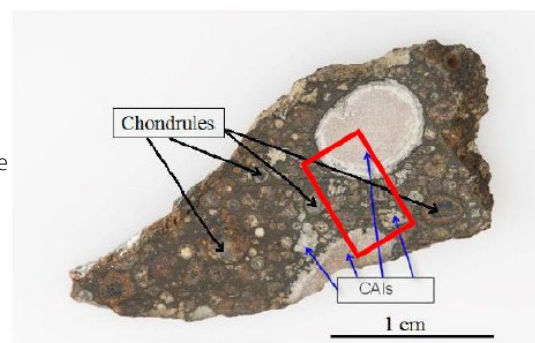


Figure 1. Photograph showing studied part of the meteorite Northwest-Africa 2086

X-ray fluorescence (XRF) analysis

For small spot mapping (SSM) analysis, measurements were performed using a Malvern PANalytical Zetium WDXRF spectrometer equipped with a 4kW, Rh-anode SST R-mAX tube, a high performance ED core with a silicon drift detector, and SuperQ software. In this study, the ED capabilities of the spectrometer are used. For all measurements in this study, net intensities were derived from the acquired spectra by deconvolution. The measurement spot size was 500µm with a step size of 250µm, producing a total of 600 spots to map and area of 5 x 7.5mm as shown in red in Figure 1.

With just a measurement time of 60 seconds per spot (total measure time of 12.3 hours), 15 elements have been simultaneously analyzed and results are shown in Figure 2. It can be clearly seen that the intensities of these 15 elements have been distinctly mapped out in the sample. The 3D intensity contour maps can be generated for each element as shown for Al in the figure. It can be observed that the elemental composition of the CAIs is enriched in Al, Ca, Ni, and Zn, while these elements are less abundant in the dark matrix of the sample. Vice versa, the elemental composition of the dark matrix shows enrichment in Fe, Mn, and Cr, which are less prominent in the inclusions. These results illustrate that elemental mapping of the elements in the sample can be clearly shown in both 2D and 3D.

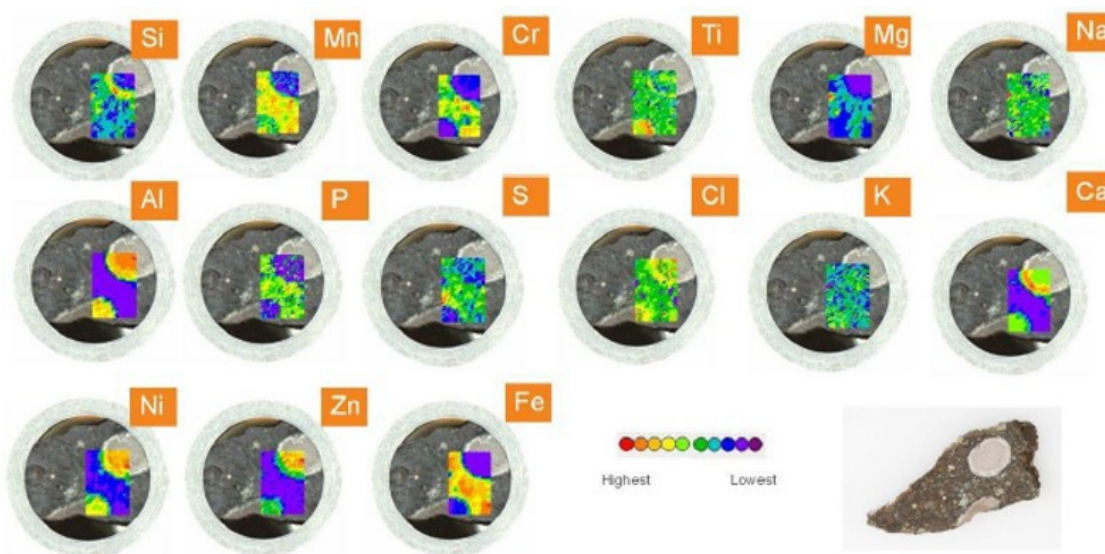
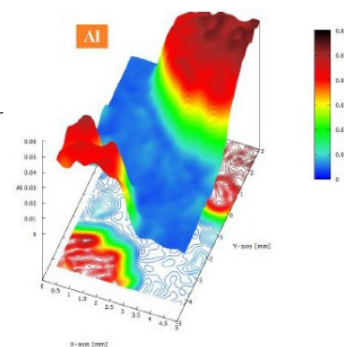
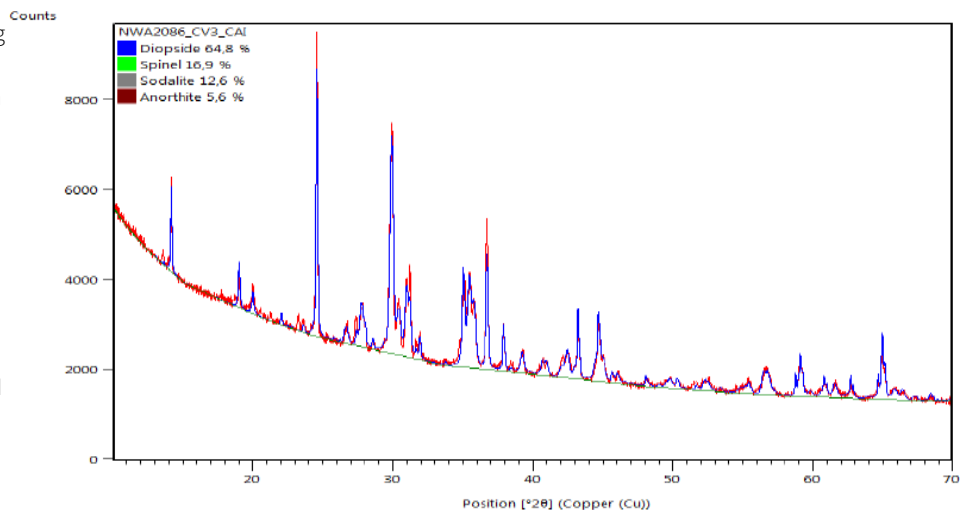


Figure 2. Small spot elemental mapping of the NWA 2086 meteorite

X-ray diffraction (XRD)

For phase identification and spot mapping a Malvern Panalytical Empyrean Diffractometer was used setup with Co K α radiation, Programmable XYZ stage, Focussing Lens (300 μ m spot) and PIXCEL Detector. The complete surface of the fragment was measured simultaneously by X-ray diffraction using Bragg-Brentano geometry. Additionally, small amounts of the matrix, chondrules and a CAI of a neighboring fragment of the same slice were prepared, pulverized, and measured separately on a silicon zero-background sample holder.



The matrix and the chondrules are composed of olivine, (Mg,Fe) $_2$ SiO $_4$, and Ca-poor pyroxene, (Mg,Fe,Ca)Si $_2$ O $_6$. The CAIs are comprised of Ca-rich pyroxene (diopside), CaMgSi $_2$ O $_6$, spinel, MgAl $_2$ O $_4$, anorthite, CaAl $_2$ Si $_2$ O $_8$, and sodalite, Na $_8$ (Al $_6$ Si $_6$ O $_24$)Cl $_2$.

Conclusion

This study shows that the Zetium wavelength dispersive spectrometer equipped with an ED and small spot mapping functionality can perform practical elemental mapping in the analysis of meteorites. Combining ED with the small-spot optics provides a mapping capability that can distinguish important compositional variations in a relatively short period of time, with minimum effort and without calibration setup

Looking for the details in a sample?

The small spot analysis and mapping module can accommodate samples with diameters up to 35mm. Sample preparation is simple, and samples are mounted in a dedicated holder, specially designed for irregularly shaped samples of varying size.



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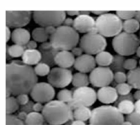
Solids / irregulars

Phase
Crystallinity
Tomography



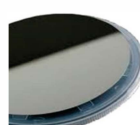
Powders

Phase
Crystallinity
Quantification



Nanomaterials

Phase
Particle size
Particle shape



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Thickness
Roughness
Orientation
Defects, Strain,
Texture, Spatial
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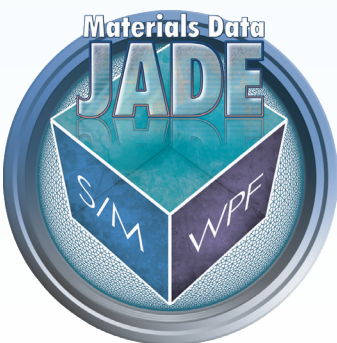
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