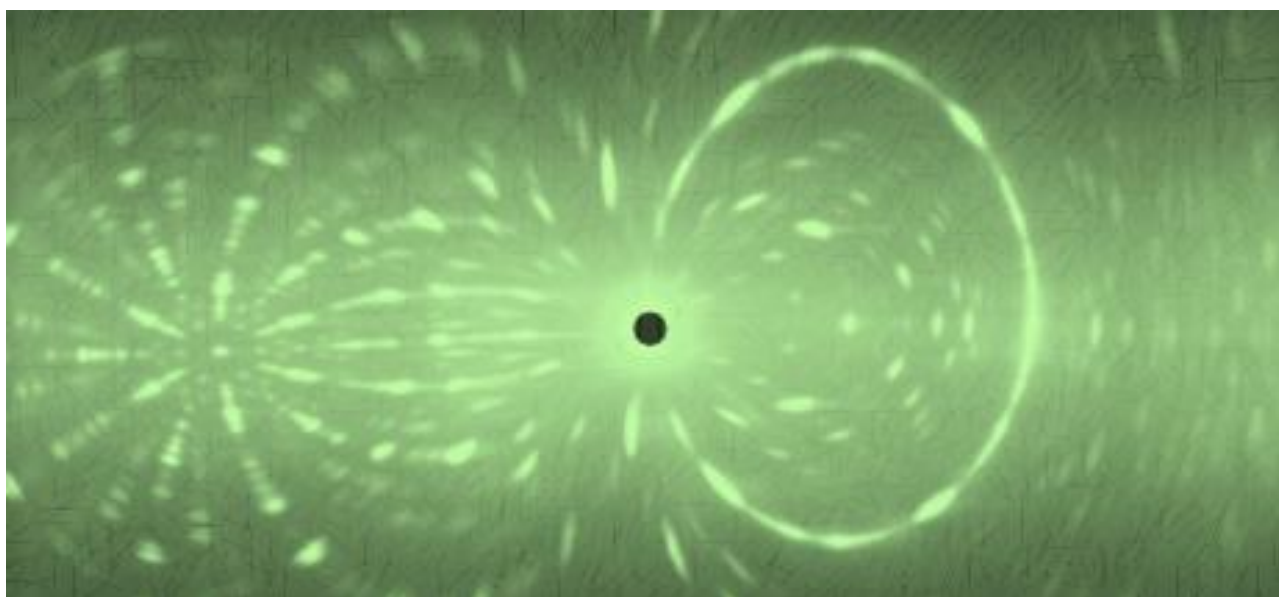




# AINSE-ANBUG NEUTRON SCATTERING SYMPOSIUM (AANSS 2016)

## Abstract Handbook

AINSE, Lucas Heights, Sydney  
29 – 30 November 2016



# AANSS ABSTRACT HANDBOOK

## 13<sup>th</sup> Annual AINSE-ANBUG Neutron Scattering Symposium (AANSS2016)

*Hosted By*

Australian Institute of Nuclear Science and Engineering  
(AINSE Limited)

Australian Neutron Beam Users Group  
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New Illawarra Road, Lucas Heights NSW 2234 Australia  
Tel +61 2 9717 3376 Email [ainse@ainse.edu.au](mailto:ainse@ainse.edu.au)  
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## A short history of ANBUG

The Australian Neutron Beam Users Group was first established on 19 April, 1979. Professor Ken Taylor (UNSW) was the first Chairman with Dr Frank Moore (AINSE) the first Honorary Secretary. The initial group comprised around 35-40 members, growing steadily to around 80 members by 1990. During this period ANBUG was instrumental in maintaining neutron science in the forefront of the scientific political arena and provided documentation to several government agencies and review processes with a view to enhancing HIFAR facilities (e.g. submissions to the Minister for National Development and the Australian Science and Technology Council). ANBUG and its engagements with the scientific community and political agencies continued in this way to around 1993. However, the lack of funding for HIFAR, combined with major developments and improvements to neutron facilities overseas (which contributed in part to the development of “suitcase science”) led to a dwindling of ANBUG in the second half of the 1990s.

The decision by the Australian Government in the late 1990s to fund a new Research Reactor, combined with related funding and staffing appointments in neutron science for OPAL, led to the new ANBUG constitution being established in 2001. ANBUG has gone from strength to strength since then and currently has over 330 members.

Open Constitution: ANBUG welcomes members from throughout the World who “... have an interest in neutron beam research and are interested in furthering and assisting the achievement of the objectives of the Society.” The success of ANBUG’s constitution is indicated by the growth in membership with members drawn from throughout the World. The openness and accessibility of ANBUG is demonstrated by the fact that members of the Executive Committee include members from Australia and overseas, including a current member from New Zealand.

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## Welcome to Delegates

On behalf of AINSE and ANBUG it is with great pleasure that we extend a warm welcome to our conference speakers and delegates.

This symposium is the annual meeting of neutron scattering community in Australia and New Zealand, and also welcomes the many international users of the Australian OPAL research reactor. The AANSS meetings are unique points on the annual conference calendar because they bring together a diverse community of scientists, engineers, mathematicians and programmers to explore the future of neutron-based science in Australia in an informal, collegial environment.

It has been written that [1]:

“In science, if you know what you are doing then you should not be doing it.  
In engineering, if you don't know what you doing, then you should not being doing it.  
Of course, you seldom, if ever, see either in a pure state.”

Neutron scattering science thrives from the intersection of subfields, by using the known to explore the unknown. The desire to solve some of the biggest questions of our time is the impetus behind the new generation of neutron sources emerging world-wide. Clever engineering, better instrumentation and revolutionary sample environments are key to exploring these unanswered questions. Multidisciplinary meetings like the AANSS ensure that there is something for everyone. A meeting like this may be the ideal opportunity for you to discover new solutions for existing problems, and also new problems looking for a solution.

The meeting also includes the ANBUG AGM, which all members are encouraged to attend. The meeting encompasses a range of presenters from those who are well-established in the Australian (and global) neutron scattering community to students and ECRs who are just beginning their careers. What all of these talks and posters have in common is that they highlight excellent scientific achievements that have primarily resulted from OPAL and showcase the wide array of neutron scattering techniques and applications.

We gratefully acknowledge the financial support from our sponsors Ezzi Vision, Nu Scientific and Radiation Saunders. Please show your gratitude to the sponsors by visiting their displays during the course of the meeting. We wish you a pleasant and scientifically inspiring meeting.

David Cortie and Rachel Caldwell

[1] R. Hamming, *The Art of Doing Science and Engineering: Learning to Learn* (1975), 5

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## Organising Committee

David Cortie (Australian National University)  
Rachel Caldwell (AINSE)  
Stephen Holt (ANSTO)  
Moeava Tehei (University of Wollongong)  
Anna Paradowska (ANSTO)  
Vanessa Peterson (ANSTO)  
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## Plenary Speaker

**E/Professor Brian O'Connor**

Department of Physics and Astronomy, Curtin University, Perth

### ***The Legacy of Dr Hugo Rietveld (1932 – 2016) - Australian Neutron Diffraction Pioneer***



The monumental contribution of the late Hugo Rietveld to powder diffraction science, through his method for optimising crystal structure models against diffraction data, is widely recognised as evidenced there being by more than 14,000 citations to his method plus many international awards which he received.

Little is known, however, about his significant role in the first neutron diffraction research performed with the HIFAR research reactor at Lucas Heights which went critical in 1958 and by 1960 was working at full power producing neutrons for researchers. Shortly after, in 1961, two papers appeared in Nature describing the initial use of HIFAR neutrons in research. Terry Sabine, Arthur Prior and Brian Hickman of the Australian Atomic Energy Commission (now ANSTO) published a paper 'Scattering of long-wavelength neutrons by irradiation beryllium oxide' in September 1961 and two weeks later the first HIFAR neutron diffraction paper appeared: Clews C J B, Maslen E N, Rietveld H M and Sabine T M (1961) 'X-ray and neutron diffraction examination of p-diphenylbenzene' Nature 192 154. Through this work Hugo became the first PhD student to author a HIFAR neutron diffraction study. He was at that time a PhD student in the Crystallography Group at the University of Western Australia led by the late Dr Ted Maslen. Importantly, the work was funded by the fledgling AINSE organisation.

On completing his PhD, Hugo maintained a strong connection with his adopted Australia after returning to The Netherlands to join the neutron diffraction group of the Reactor Centrum Nederland in Petten where he focussed on working with powders for which large single crystals were not available. This was most challenging due to overlapping reflections making crystal structure solution and refinement unduly difficult. Hugo moved quickly in developing what was to become the Rietveld Method. The method was first described in two papers published in 1967 and 1969 after he had presented a paper at the IUCr Congress in Moscow in 1966.

While the power of the method for crystal structural research is widely appreciated, its growing importance in materials characterization in the industrial setting is not well known yet. Brian O'Connor will give an overview of the broad impact of the method in fundamental research and in industrial uses such as process control in mineral processing. Applications of the method have recently included the in-situ processing of powder x-ray diffraction data at the Mars Science Laboratory which landed on Mars in 2012.



## Speaker Abstracts

### Session 1

#### Remembering Rietveld: Powder diffraction

##### *Characterising new planetary materials with neutron diffraction.*

**H.E. Maynard-Casely<sup>a</sup>**, H.E.A. Brand<sup>b</sup>, M.L. Cable<sup>c</sup>, R. Hodyss<sup>c</sup>

<sup>a</sup> Australian Centre for Neutron Scattering, ANSTO.

<sup>b</sup> Australian Synchrotron, ANSTO.

<sup>c</sup> Jet Propulsion Laboratory, NASA, USA.

There's a lot of hydrogen in the outer solar system; locked up with water on the icy Galilean moons of Jupiter, within the small organic molecules that rain down on Saturn's moons Titan or even in an elusive metallic form within the centers of the gas giants. The intrinsic hydrogen-domination of planetary ices, makes studying these materials with laboratory powder diffraction very challenging. Insights into their crystalline phase behavior and the extraction of a number of thermal and mechanical properties is often only accessible with high-flux synchrotron x-ray diffraction or with neutron diffraction. Here, we will present how both the ECHIDNA and WOMBAT instruments at ACNS have been used to gain insights into new materials that have been found to exist under planetary conditions.

## Hydration as a trigger for new properties in inorganic materials

**S.G. Duyker**<sup>a</sup>, V.K. Peterson<sup>b</sup>, G.J. Kearley<sup>b</sup>, A.J. Studer<sup>b</sup>, C.J. Kepert<sup>a</sup>, J.A. Hill<sup>c</sup>, C.J. Howard<sup>d</sup> and A.L. Goodwin<sup>c</sup>

<sup>a</sup> School of Chemistry, University of Sydney, NSW, Australia.

<sup>b</sup> Australian Centre for Neutron Scattering, Lucas Heights, NSW, Australia

<sup>c</sup> Inorganic Chemistry Laboratory, Department of Chemistry, University of Oxford, UK

<sup>d</sup> School of Engineering, University of Newcastle, NSW, Australia

The humble water molecule binds to metal ions strongly enough that it can have a significant distortive influence on the coordination geometry, yet weakly enough that it can be readily removed, thus providing scope for reversible chemical switching between structural forms. When this principle is applied in 3D coordination frameworks, the unique topological constraints of the framework can lead to new behaviours. Examples from our work will be presented, including anomalous mechanical properties enabled by unsaturated coordination spheres,[1] and new kinds of symmetry breaking transformations triggered by (de)hydration[2] (see figure).

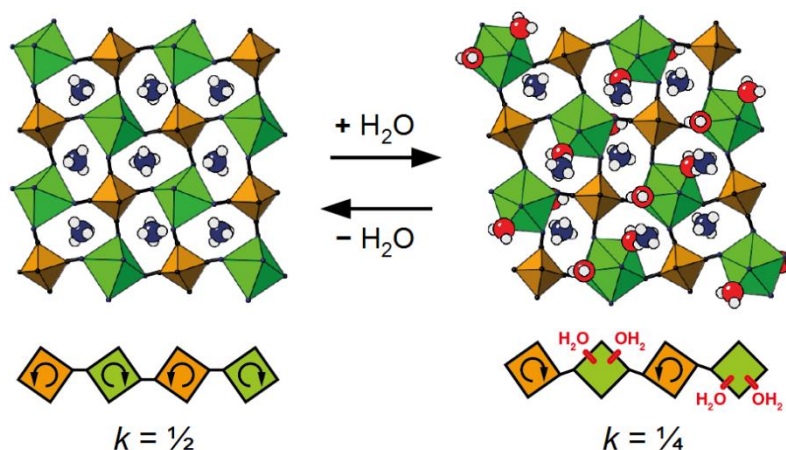


Figure:  $(NH_4)_2SrFe(CN)_6$  has a cyanide-bridged structure analogous to that of a conventional double perovskite, which, upon hydration of the Sr ions (green), distorts to yield a polyhedral tilt system that is forbidden under the corner-sharing regime of oxide-bridged perovskites.[2]

[1] S.G. Duyker, V.K. Peterson, G.J. Kearley, A.J. Studer, C.J. Kepert, *Nature Chem.*, **8**, 270 (2016).

[2] S.G. Duyker, J.A. Hill, C.J. Howard, A.L. Goodwin, *J. Am. Chem. Soc.*, **138**, 11121 (2016).



## **Pressure-induced valence transitions in metal oxides: squeezing the electrons out of lone pairs**

**C.D. Ling**<sup>a</sup>, **M. Avdeev**<sup>b</sup> and B.J. Kennedy<sup>a</sup>

<sup>a</sup> School of Chemistry, The University of Sydney, Sydney 2006, Australia.

<sup>b</sup> Australian Centre for Neutron Scattering, ANSTO, Menai 2232, Australia.

While bonds in solid-state compounds always have some degree of covalent character, the ionic approximation is usually sufficient to understand their “crystal chemistry” using concepts like the effective ionic radius (IR). IR predicts that an atom will shrink as its oxidation state increases. This occurs gradually as electrons are removed within a shell (e.g., IR(Ir<sup>3+</sup>) = 0.68, IR(Ir<sup>4+</sup>) = 0.625, IR(Ir<sup>5+</sup>) = 0.57 Å in 6-fold coordination), but removing the last electron of a shell produces a much more pronounced change (e.g., IR(Bi<sup>3+</sup>) = 1.03, IR(Bi<sup>5+</sup>) = 0.76 Å). For a compound with a suitable combination of cations, it should therefore be possible to effect a net reduction in volume by transferring an electron from one to the other. Temperature and/or pressure could drive such a valence state transition; but in practice, this is extremely rare, with only three cases reported until recently. We tested this idea systematically in a series of high-pressure X-ray and neutron diffraction and spectroscopy experiments on six candidate materials containing Bi<sup>3+</sup> with 4d or 5d metal cations. We observed a valence state transition in every case, suggesting that they are far more common than previously thought. This talk will present both published [1,2] and unpublished experimental results, as well as *ab initio* calculations that shed light on the finely balanced electronic states of these compounds. The potential for tuning these transitions closer to ambient pressures, and of inverting the effect to give a volume change with an electronic stimulus, will be discussed.

[1] W. Müller, M. Avdeev, Q. Zhou, B.J. Kennedy, N. Sharma, R. Kutteh, G.J. Kearley, S. Schmid, K.S. Knight, P.E.R. Blanchard, C.D. Ling, *Journal of the American Chemical Society* **134**, 3265-3270 (2012).

[2] Z. Huang, J.E. Auckett, P.E.R. Blanchard, B.J. Kennedy, W. Müller, Q. Zhou, M. Avdeev, M.R. Johnson, M. Zbiri, G. Garbarino, W.G. Marshall, Q. Gu, C.D. Ling, *Angewandte Chemie – International Edition* **53**, 3414-3417 (2014).

## **Using Neutron-based Techniques to Investigate Battery Behaviour**

**James C. Pramudita**<sup>a,b</sup>, Damian Goonetilleke<sup>a</sup>, Vanessa K. Peterson<sup>b,c</sup> and Neeraj Sharma<sup>a</sup>

<sup>a</sup> School of Chemistry, UNSW Australia

<sup>b</sup> Australia Nuclear Science and Technology Organisation

<sup>c</sup> Institute for Superconducting & Electronic Materials, University of Wollongong

The extensive use of portable electronic devices has given rise to increasing demand for reliable high energy density storage in the form of batteries. Today, lithium-ion batteries (LIBs) are the leading technology as they offer high energy density and relatively long lifetimes.[1] Despite their widespread adoption, Li-ion batteries still suffer from significant degradation in their performance over time.[1] The most obvious degradation in lithium-ion battery performance is capacity fade – where the capacity of the battery reduces after extended cycling. This talk will focus on how *in situ* time-resolved neutron powder diffraction (NPD) can be used to gain a better understanding of the structural changes which contribute to the observed capacity fade. The commercial batteries studied each feature different electrochemical and storage histories that are precisely known, allowing us to elucidate the tell-tale signs of battery degradation using NPD and relate these to battery history. Moreover, this talk will also showcase the diverse use of other neutron-based techniques such as neutron imaging to study electrolyte concentrations in lead-acid batteries, and the use of quasi-elastic neutron scattering to study Na-ion dynamics in sodium-ion batteries.

[1] Tarascon, J. M.; Armand, M., Issues and challenges facing rechargeable lithium batteries. *Nature* **2001**, 414 (6861), 359-367.

## Structural Transformative Behaviour in Barium Thorium Oxides: A Neutron and Synchrotron X-ray Diffraction Study

**G.L. Murphy**<sup>ab</sup>, **B. J. Kennedy**<sup>a</sup>, **M. Avdeev**<sup>b</sup> and **Z. Zhang**<sup>b</sup>

<sup>a</sup> *School of Chemistry, University of Sydney, New South Wales, 2006, Australia.*

<sup>b</sup> *Australian Nuclear Science and Technology Organisation, Lucas Heights, 2234 Australia.*

The increasing interest in nuclear energy, as a response to demands for a transition to non-fossil fuel energy sources, has reinvigorated attempts to explore a thorium fuel cycle. Thorium is seen as a more attractive option to uranium as a nuclear fuel, due to its enhanced proliferation resistance and reduced transuranic generation. Thorium reactors face several challenges particularly related to the large paucity of information of fuel fission by product material phases. A pertinent structure in this regard is BaThO<sub>3</sub>, due its potential to host the fission daughter Ba-137m. The radioactivity of thorium coupled with the unusual decomposition behaviour of BaThO<sub>3</sub>, has prevented detailed analysis of the material. The present study has utilized a combination of neutron and synchrotron x-ray diffraction in order to establish the structure of BaThO<sub>3</sub> and elucidate its high temperature structural behaviour. Our results indicate BaThO<sub>3</sub> undergoes a reversible second order continuous phase transformation at 975 K from the space group *Pbnm* to *Imma*. Neutron and synchrotron X-rays were key to this, successfully resolving the characteristic superlattice X-point and M-point reflections present in *Pbnm* and absent in *Imma*. Considering the decomposition behaviour of BaThO<sub>3</sub> attempts were made to include Th(IV) partially on the *B* site of other ABO<sub>3</sub> perovskites including BaZrO<sub>3</sub> and SrZrO<sub>3</sub> through the formation of appropriate solid solutions. This resulted in maximum Th solubilities of less than 10 %. Surprisingly greater solubilities are known for Th(IV) on the *A* site of in layered perovskites such as Na<sub>2/3</sub>Th<sub>1/3</sub>TiO<sub>3</sub> [1]. The ability of Th to occupy the perovskite *A*-site is in contrast to that what is typically observed for uranium and other nuclear fuel relevant perovskites.

[1] Zhu, W. J.; Hor, P. H., A new titanium perovskite oxide, Na<sub>2/3</sub>Th<sub>1/3</sub>TiO<sub>3</sub>. *J. Solid State Chem.* **1995**, *120* (1), 208-20

## Session 2 Nature and History

### ***Imaging the past: recent applications of neutron imaging in archaeometry***

**Filomena Salvemini**, Australian Nuclear Science and Technology Organisation

The study of metal artefacts of archaeological, historical and cultural interest can shed light on the most advanced manufacturing processes developed by different cultures over time. Scientist need to treat them with care and must avoid damage, including the acceleration of any natural ageing process, so that we can pass on the artefacts to future generations.

The cultural heritage community is well aware of the benefits of non-invasive scientific methods. This approach has been progressively established as common practice in archaeometry and conservation science. Neutron imaging is playing a significant role in expanding the technical limits and investigation capabilities of standard analytical methods due to its well-known features of high a penetration power and its different interaction with matter, compared, for example, with the analogous X-ray imaging.

While traditional analytical techniques might fail to preserve the integrity of the objects, neutron imaging methods can be successfully used to characterize the structure, morphology and composition of metal artworks three-dimensionally without the need for sampling or invasive procedures. These physical properties of an artefact are the imprint of its manufacturing process and of its life cycle. They can be convincingly reconstructed through a careful analysis of the material evidence.

In collaboration with museum institutions and university research groups, archaeometric investigations have been recently conducted by using the neutron imaging beamline DINGO at ANSTO. A selection of case studies will be presented.

### ***Neutron micro-CT as a non-destructive tool for palaeontology***

**Joseph Bevitt**, Australian Nuclear Science and Technology Organisation

The physical extraction of fossilised remains from rocks enables quantitative physiological investigation of bone-dimensions, volume, and porosity, however leads to the destruction of valuable contextual information and soft-tissue remains within the matrix. Conventional and synchrotron-based X-ray computed tomography (XCT) have been utilised for many years as critical tools in uncovering valuable 3-D internal and surface renderings of scientifically important fossils, however poor contrast and X-ray penetration often prevents thorough tomographic analysis.

DINGO, Australia's first and only neutron micro-computed tomography (nCT) instrument is located at the OPAL nuclear research reactor. It is being used to obtain unprecedented reconstructions of extraordinary fossilised anatomical features not visible with conventional imaging techniques. This presentation will outline the physical capabilities of DINGO, the limitations and results to-date in the field of palaeontology. Drawing upon specimens scanned from across Australia, Antarctica, New Zealand, China and Mongolia, this presentation will demonstrate the complementarity of nCT to classic XCT methods, and its limitations. Evidence of extraordinary soft-tissue fossilisation in Jurassic fauna will be shown, along with insights into ancient and modern-day animals, achieved through the use of neutron CT scanning.

## Residual stress in mollusc shells

**M.B. Cortie**<sup>a</sup>, A. Dowd<sup>a</sup>, K.Y.H He<sup>a</sup>, A. Choi<sup>a</sup> and V. Luzin<sup>b</sup>

<sup>a</sup>School of Mathematical and Physical Sciences, University of Technology Sydney,

<sup>b</sup>Australian Centre for Neutron Scattering

The aragonite-protein composite material out of which the shells of most molluscs are made has a fracture toughness of about  $8 \text{ MPa}\sqrt{\text{m}}$ . This is surprisingly high considering that the aragonite phase has a bulk fracture toughness of only  $0.9 \text{ MPa}\sqrt{\text{m}}$ . One reason for the improved performance of the shell relative to pure aragonite is that it is comprised of a strongly textured array of aragonite platelets in an organic matrix. It is well known that cracks that initiate in an aragonite platelet are deflected, blunted or arrested when they reach the more ductile organic phase. We speculate that a compressive residual stress at strategic locations in the shell may further improve its resistance to crack propagation.

To investigate this hypothesis we used neutron diffraction, X-ray diffraction and Raman spectroscopy in an attempt to identify whether a detectable stress distribution exists in a large mollusc shell and, if so, whether this stress state can provide enhanced fracture toughness. Freshly collected shells of the gastropod *Ninella torquata* (family Turbinidae), which has a diameter of about 10 cm, were used. The texture of the samples was readily extracted using neutron diffraction and an apparent residual stress gradient of several MPa identified. This effect was not evident in X-ray diffraction of powder samples taken in layers spaced through the wall thickness. The possible existence and implications of a non-uniform stress distribution through the shell are analyzed and discussed.

## The Impact of pH on Packing in Tethered Lipid Bilayers

C.G. Cranfield<sup>a</sup>, T. Berry<sup>a</sup>, **S.A. Holt**<sup>b</sup>, A.P. Le Brun<sup>b</sup>, S.M. Valenzuela<sup>a</sup>, H. Coster<sup>c</sup>, and B. Cornell<sup>d</sup>

<sup>a</sup>School of Life Sciences, University of Technology Sydney, Ultimo, Australia.

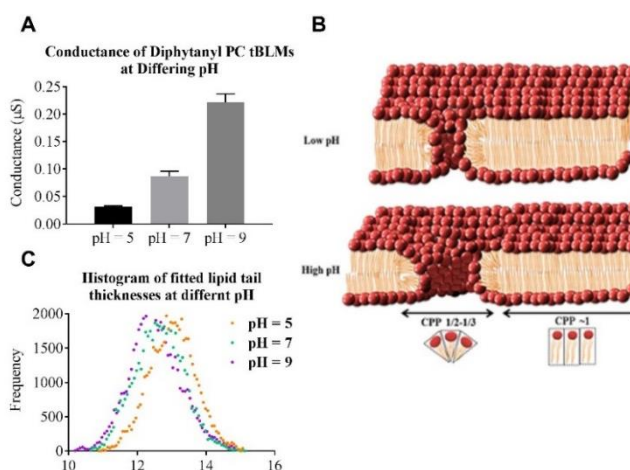
<sup>b</sup>Australian Centre for Neutron Scattering, ANSTO, Australia.

<sup>c</sup>School of Chemical and Biomolecular Engineering, University of Sydney, Australia.

<sup>d</sup>SDx Tethered Membranes Pty Ltd, Roseville, Australia.

We report that increasing the  $\text{H}_3\text{O}^+$  concentration when lowering the pH reduces the intrinsic ionic conduction through phospholipid bilayers (Fig 1A), which is counter to what might be expected from increasing the  $\text{H}_3\text{O}^+$  concentration. We attribute the conduction decrease to a reduction of the molecular area per lipid ( $a_0$ ) [1]. These effects are seen at  $\text{H}_3\text{O}^+$  concentrations in the range nM to  $\mu\text{M}$  despite these being very low concentrations compared to that of a typical bathing electrolyte solution of 135mM ionic concentration. We present

a model, in which the pH dependent reduction in  $a_0$  favours an increase in lipid packing. To support this model, we provide evidence of the effects of the hydronium ion on lipid geometry using *neutron reflectometry* (Fig 1C). Further examples will be given of the impact of the  $\text{H}_3\text{O}^+$  ion concentration on the hydrogen bonding within the polar groups of lipid.



[1] J. Israelachvili, S. Marčelja and R. G. Horn, *Quarterly reviews of biophysics*, **13**, 121 (1980)

## **Chemical Deuteration and Neutrons for Structure Function Applications**

**T.A. Darwish**, N.R. Yepuri, A.K. Heuer, M. Cagnes and P. Holden

*The National Deuteration Facility, Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, 2232, Australia.*

In small angle neutron scattering and neutron reflectometry studies, the use of mixtures of deuterated and hydrogenated solvents to manipulate scattering length density and achieve contrast variation is widespread. This approach, while useful is less effective for multicomponent organic systems containing molecules of similar scattering length densities. In such systems molecular deuteration is necessary to achieve contrast between the different components and it significantly increases the options in structure function investigations.

There have been limited global initiatives in the field of molecular deuteration where the majority of these programs focus on biological deuteration of proteins and lipids, while more complex deuterated small molecules haven't been widely available to the neutron community. This has limited the experiments that can be performed, and formed a bottleneck for advancing the applications of neutron scattering.

In this paper we will discuss the recent advancements and the impact of deuteration on the research outcomes achieved by using deuterated molecules produced by the chemical deuteration laboratories at ANSTO's National Deuteration Facility. Recent high-impact case studies will be presented which reveal the exciting and diverse characterisation studies which are now available for the neutron community. The chemical deuteration of surfactants, sugars, heterocyclic and aromatic compounds has made possible a wide range of investigations systems in the fields of molecular electronics, structural biology, and biotechnology.

## **Session 3 Soft Matter**

### **Kinetic Small Angle Neutron Scattering to Study Large Biomolecular Complexes**

R. Inoue<sup>a</sup>, **K. Wood**<sup>b</sup> and M. Sugiyama<sup>a</sup>

<sup>a</sup> *Research Reactor Institute, Kyoto University, Osaka 590-0494, Japan.*

<sup>b</sup> *Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, Australia.*

Proteins are the structural building blocks that make us up but also the functional molecules that drive us. Amazing advances over the last 60 years in structural biology mean we are now determining the 3D structure of over eight thousand proteins per year. Proteins rarely function as isolated entities however, and one of the next major challenges in structural biology is to examine the structure-function relationship in larger complexes made up of several individual proteins.

Small angle scattering is particularly well suited to probe the space scale of large biomolecular complexes. The protein  $\alpha$ -Crystallin makes up around a third of the structural proteins of mammalian eye lenses as well as displaying chaperone function. Here we examine the dynamical structure of  $\alpha$ -Crystallin complexes by following exchange between the complexes using deuteration and small angle neutron scattering [1].

The work described is a result of collaborative work between all the authors listed below.

[1] R. Inoue, T. Takata, N. Fujii, K. Ishii, S. Uchiyama, N. Sato, Y. Oba, K. Wood, K. Kato, N. Fujii and M. Sugiyama, *Sci. Rep.* **6**: 29208 (2016)



## Watching photo-sensitive surfactant micelles morph in real time using SANS

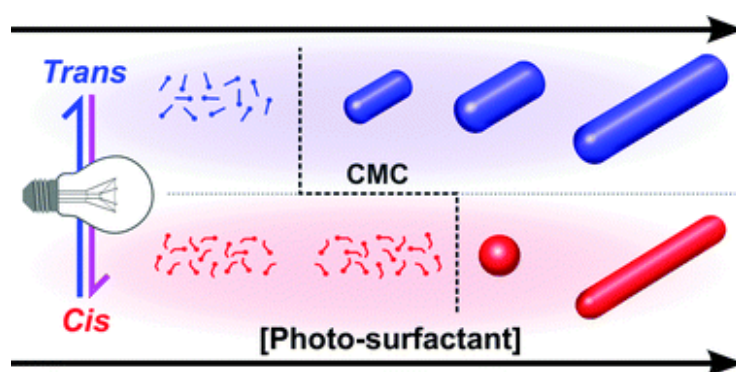
Rico F. Tabor<sup>a</sup>, Brendan L. Wilkinson<sup>b</sup> and Christopher J. Garvey<sup>c</sup>

<sup>a</sup> School of Chemistry, Monash University, VIC 3800, Australia.

<sup>b</sup> University of New England, Armidale, NSW 2351, Australia.

<sup>c</sup> ACNS, ANSTO, Lucas Heights, NSW 2234, Australia.

Recent interest has surged in colloidal and self-assembling systems that can respond to internal and external stimuli. Of the possible response mechanisms, light is seen as a clean and low energy 'reagent' for stimulating physical and chemical changes. We have recently synthesized a library of light-addressable surfactant molecules based around the photo-isomerisable azobenzene core, coupled to sugar head-groups [1]. Depending on their molecular geometry, these molecules self-assemble into a wide variety of micellar structures, from small spheres to worms, fibres and liquid crystals, and the diversity of this self-assembly is uncovered using small-angle neutron scattering. By using time-resolved SANS, we can watch as the micelles change their shape in real time as the molecules undergo photo-isomerisation, pointing to applications in the delivery of sensitive cargoes, and the modulation of biological and cellular processes.



[1] R. F. Tabor, D. D. Tan, S. S. Han, S. A. Young, Z. L. E. Seeger, M. J. Pottage, C. J. Garvey, and B. L. Wilkinson, *Chem. Eur. J.*, **20** (2014) 13881-13884.

[2] R. F. Tabor, M. J. Pottage, C. J. Garvey and B. L. Wilkinson, *Chem. Comm.* **51** (2015) 5509-5512.

## The Specific Ion Response of Polyelectrolyte Brushes Depends on Polymer and Anion Hydrophilicity

**T.J. Murdoch<sup>a</sup>**, J.D. Willott<sup>a</sup>, W.M. de Vos<sup>b</sup>, A. Nelson<sup>c</sup>, S.W. Prescott<sup>d</sup>, E.J. Wanless<sup>a</sup> and G.B. Webber<sup>a</sup>

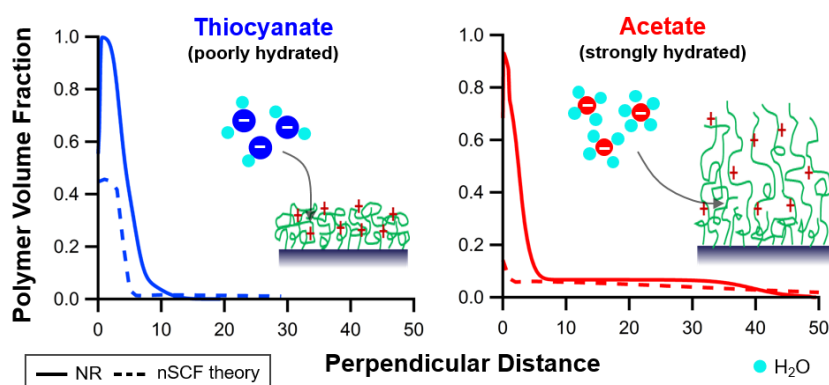
<sup>a</sup> Priority Research Centre for Advanced particle Processing and Transport, University of Newcastle, NSW 2234, Australia.

<sup>b</sup> Membrane Science and Technology, Mesa+ Institute for Nanotechnology, University of Twente, Enschede 7500 AE, Netherlands.

<sup>c</sup> Australian Nuclear Science and Technology Organisation, NSW 2234, Australia.

<sup>d</sup> School of Chemical Engineering, UNSW Australia, NSW 2052, Australia

We have previously shown that the pH and ionic strength response of weak, polybasic brushes is dependent on the location of the counterion within the Hofmeister series and the relative hydrophobicity of the polymer.[1-3] Recent measurements of a hydrophobic poly(2-diisopropylamino) ethyl methacrylate (PDPA) brush on *Platypus* reflectometer show that a collapsed conformation independent of anion identity is formed at low ionic strength, 0.1 mM. At higher ionic strengths, up to 500 mM, extended conformations are observed in potassium acetate solutions, while potassium thiocyanate solutions collapse the brush. Numerical self-consistent field (nSCF) calculations allowed previous hypotheses regarding the accumulation and hydration strength of the counterions to be tested. Addition of a single Flory-Huggins interaction parameter ( $\chi$ ) analogous to the anion hydrophilicity was sufficient to replicate the measured brush behaviour.



[1] J.D. Willott et al., *Langmuir*, **31**, 3707 (2015)

[2] J.D. Willott et al., *Macromolecules*, **49**, 2327 (2016)

[3] J.D. Willott et al., *PCCP*, **17**, 3880 (2015)

## **Hydrogen bond dynamics, conformational flexibility and polymorphism in antipsychotics**

**José E.M. Pereira**<sup>a</sup>, Juergen Eckert<sup>b</sup>, Dehong Yu<sup>c</sup>, Richard Mole<sup>c</sup> and Heloisa N. Bordallo<sup>a,d</sup>

<sup>a</sup> Niels Bohr Institute, University of Copenhagen, DK-2100, Copenhagen, Denmark.

<sup>b</sup> Department of Chemistry, University of South Florida, Florida 33620, United States.

<sup>c</sup> Australian Nuclear Science and Technology Organisation, Lucas Heights, 2233 NSW.

<sup>d</sup> European Spallation Source ESS AB, PO Box 176, SE-22100 Lund, Sweden.

This work is related to the investigation of three different antipsychotics, one of each generation: aripiprazole ( $C_{23}H_{27}Cl_2N_3O_2$ ), haloperidol ( $C_{21}H_{23}ClFNO_2$ ) and quetiapine hemifumarate ( $C_{23}H_{27}N_3O_4S$ ) using a combination of Inelastic Neutron Scattering (INS) and Density Functional Theory (DFT). These substances were selected because their crystalline structure and the concerns related to their polymorphism are somehow known [1]. We report on data obtained using the direct geometry spectrometer PELICAN, located at the Australian Centre for Neutron Scattering (ACNS, formerly the Bragg Institute) at the Australian Nuclear Research and Technology Organisation (ANSTO). Polymorphic transformations and purity of the samples were determined by calorimetric studies, while their structures were verified by X-rays diffraction. Furthermore, the origin of each of the observed modes is supported by theoretical data provided by Density Functional Theory calculations (DFT).

[1] G.L. Destri et al, J. Pharmaceutical Sciences 2011, 100, 4896; K. Ravikumar and B. Sridhar, Acta Cryst. 2005, E61, o3245 & JB Nanubolu et al, CrystEngComm., 2012, 14, 4677.

## **Deuterium effects in Human Serum Albumin with Nanoparticle Silica Kinetics**

**John White**<sup>\*</sup>, Jared Raynes<sup>§</sup>, Jitendra P. Mata<sup>#</sup>, Elliot Gilbert<sup>#</sup>, Robert Knott and Liliana de Campo<sup>#</sup>

<sup>\*</sup>Research School of Chemistry, Australian National University

<sup>§</sup>CSIRO Agriculture & Food, Werribee, Australia

<sup>#</sup>ACNS, ANSTO, Sydney, Australia

Light scattering shows three stages of the interaction of 80Å radius silica nanoparticles with human serum albumin in buffered solutions. The structures formed in the fast stage, twenty minutes after mixing, have been identified in a “stopped flow” neutron small angle scattering experiment. Good scattering functions were obtained at two-minute time resolution for this phase of the interaction in D<sub>2</sub>O and H<sub>2</sub>O. pH dependent changes in structure are analysed using standard fitting programs with a minimum number of parameters.

This experiment was aimed to find the structural signature of nanoparticle-protein interaction, possibly the “protein corona” supposed to be formed as a means to promote entry of nanoparticles into cells. Here we use small “engineered” nanoparticles where the indications of toxicity are strong. We show with nanometric resolution that for our system, the association is largely a form of protein-induced aggregation distinct from the protein corona hypothesis. The corona might well be the mode of interaction for small proteins and nanoparticles 10-100 times larger than we have studied, but measurements on widely used commercial products may be more relevant.

## Session 4

### Technique development for academia and industry

#### ***Residual stress in self-piercing riveting (SPR) joints: Industrial application***

**Rezwanul Haque**

School of Science and Engineering

University of the Sunshine Coast, Australia - rhaque@usc.edu.au

Self-Piercing Riveting (SPR) is a high-speed mechanical fastening technique used to join sheet materials which does not require pre-drilling. This technique is increasingly adopted by the automotive industry because of the growing use of light weight or dissimilar metals (galvanized steel, aluminium and magnesium alloys) that are difficult to spot weld. During SPR, a mechanical interlock is created by the rivet through piercing the top sheet and flaring in the bottom sheet under the guidance of the rivet internal geometry and a die. However, the rivet material should have enough hardness to pierce the sheets, and necessary ductility to deform plastically in the bottom sheet without cracking. As now-a-days more and more high strength lightweight alloy materials are introduced, the ability of a rivet to pierce and deform in a ductile manner becomes more limited. Also, residual stress is developed inside the joint due to the plastic deformation. During service, this residual stress may lead to premature failure of the joint. Hence, it is important to know the residual stress distribution arising from the riveting process.

Residual stress distribution in SPR joints of high strength materials was investigated by neutron strain scanner Kowari at ANSTO. It is evident that neutron diffraction technique can successfully predict the position of the rivet legs after flaring inside the joint without having to cross-section. Moreover, this technique can also help to predict any cracks in top and bottom sheets. The study also shows that neutron diffraction technique enabled a crack in the rivet tip to be detected which was not apparent from a cross-section. This information can provide guidance for the optimum design of SPR joining conditions in industrial application, especially for high strength low ductility materials also can be used to push the operating window of the process.

#### ***Neutrons for Structural Integrity Applications of Engineering Components***

**A.M. Paradowska\***, U. Garbe, M. Reid, P. Bendeich, M. Law

Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW 2232, Australia

\*Corresponding author ([anna.paradowska@ansto.gov.au](mailto:anna.paradowska@ansto.gov.au))

The Australian Nuclear Science and Technology Organisation (ANSTO) at Lucas Heights, have two neutron instruments: KOWARI and DINGO that can be useful tool in modern welding assessment. Both instruments provide non-destructive characterisation techniques for modern materials, engineering and structural integrity investigations. The primary function of the KOWARI instrument is the determination of residual stresses within the interior of bulk engineering components and test samples, in particular for the development of modern engineering welding processes as well as post processing (e.g. post weld heat treatment, peening) and variety of structural integrity investigations. DINGO is a neutron radiography and imaging beamline used to assessing defects and dimensional tolerance of internal features engineering components well suited for thick and complex's weldments. In this paper an overview of possibilities and recent developments on both instruments are presented.

## ***Large-Scale Structure Investigations via Neutron Scattering Techniques***

**C. Rehm**, and L. de Campo

*Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, Lucas Heights NSW 2234, Australia.*

Modern materials science and engineering relies increasingly on detailed knowledge of the microstructure and interactions in soft and hard materials. Contemporary research areas comprise, e.g., biology and the life sciences, porosity, particle sizes as well as complex fluids. At the Australian Nuclear Science and Technology Organisation (ANSTO) we apply small-angle neutron scattering (SANS) as a major technique for probing structures and interfaces of bulk samples of such substances on length scales ranging between approximately 0.001  $\mu\text{m}$  and 1  $\mu\text{m}$  using the QUOKKA [1] instrument, whereas the ultra-small-angle neutron scattering (USANS) instrument KOOKABURRA [2] advances large-scale structure determination of complex systems of interest in the size range of 0.1  $\mu\text{m}$  to 10  $\mu\text{m}$ . Both techniques provide information on bulk properties with minimum sample preparation, and can be used to analyse material in a non-destructive manner. This presentation will discuss SANS and USANS techniques available at ANSTO for the study of large-scale structures, and present combined SANS/USANS data sets measured on selected samples.

[1] E.P. Gilbert, J.C. Schulz and T.J. Noakes, *Physica B* **385-386**, 1180 (2006).

[2] C. Rehm, A. Brûlé, A.K. Freund and S. Kennedy, *J. Appl. Cryst.* **46**, 1699 (2013).



## Ionic Liquid Based Nanoparticle Emulsions as a Corrosion Inhibitor

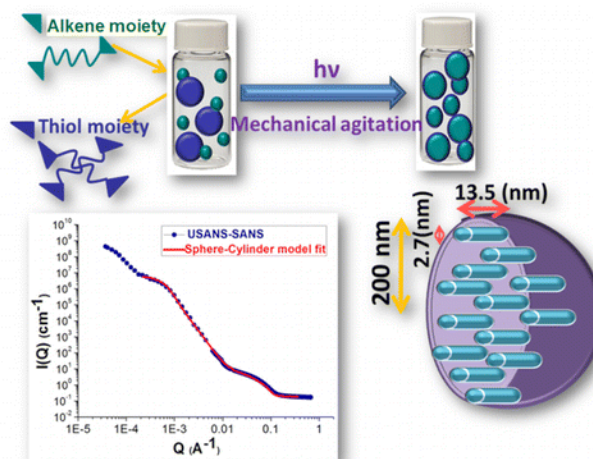
**Jitendra P. Mata**<sup>a</sup>, Mona Taghavikish<sup>b</sup>, Surya Subianto<sup>b</sup>, Naba Kumar Dutta<sup>b,c</sup>, Liliana de Campo<sup>a</sup>, Christine Rehm<sup>a</sup>, and Namita Roy Choudhury<sup>b,c</sup>

<sup>a</sup> ACNS, ANSTO, New Illawarra Rd, Lucas Heights, Sydney, NSW 2234, Australia.

<sup>b</sup> Future Industries Institute, Mawson Lakes Campus, University of South Australia, Mawson Lakes, Adelaide, SA 5095, Australia.

<sup>c</sup> School of Chemical Engineering, University of Adelaide, Adelaide, SA 5005, Australia

In this contribution, we report the facile preparation of cross-linked polymerizable ionic liquid (PIL)-based nanoparticles via thiol–ene photopolymerization in a miniemulsion. The synthesized PIL nanoparticles with a diameter of about 200 nm were fully characterized with regard to their chemical structures, morphologies, and properties using different techniques, such as Fourier transform infrared spectroscopy, thermogravimetric analysis, scanning electron microscopy, and transmission electron microscopy [1].



To gain an in-depth understanding of the physical and morphological structures of the PIL nanoparticles in an emulsion, small-angle neutron scattering and ultra-small-angle neutron scattering were used. Neutron scattering studies revealed valuable information regarding the formation of cylindrical ionic micelles in the spherical nanoparticles, which is a unique property of this system. Furthermore, the PIL nanoparticle emulsion was utilized as an inhibitor in a self-assembled nanophase particle (SNAP) coating. The corrosion protection ability of the resultant coating was examined using potentiodynamic polarization and electrochemical impedance spectroscopy. The results show that the PIL nanoparticle emulsion in the SNAP coating acts as an inhibitor of corrosion and is promising for fabricating advanced coatings with improved barrier function and corrosion protection [1].

[1] M. Taghavikish et al., *ACS Omega* 1 (2016) 29–40.

## ***The Time-of-Flight SANS Instrument BILBY: design, construction, commissioning and first results***

**A. Sokolova<sup>a</sup>**, A. Whitten<sup>a</sup> and L de Campo<sup>a</sup>

<sup>a</sup>*Australian Centre for Neutron Scattering, ANSTO, NSW 2234, Australia*

ANSTO successfully operates one SANS instrument Quokka and in January of year 2016 commenced user operation of the second SANS instrument, Bilby [1]. The Bilby is utilizing both Time-of-Flight and monochromatic capabilities. The design (in particular, set-up of four choppers which uses idea of that for D33 instrument [2] at ILL) opens possibility to vary wavelength resolution in the wide range (from 4% to 30%). Two arrays of position sensitive detectors in combination with utilizing of wide wavelength range (from  $\sim 3\text{\AA}$  to  $\sim 18\text{\AA}$ ) provide capability to collect scattering data of wide angular range without changing experimental set-up (the most common settings used by now allow simultaneous data collection in the range between  $1 \cdot 10^{-3} \text{\AA}^{-1}$  ÷  $0.6 \text{\AA}^{-1}$ ). Offered instrument design opens possibility to collect scattering from a wide range of samples, with a unique capability to record fast kinetics data.

The presentation will be focused on general concept and unique features of the new SANS instrument, as well as some results of instrument commissioning and operation. Data reduction algorithms implemented in Mantid software [3] will be presented. Also, some principal difficulties in use of ToF mode comparing to monochromatic set-up for soft matter samples will be discussed.

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[2] C.D. Dewhurst, *Measurement Science and Technology*. **19** (2008).

[3] O. Arnold, et al., *Nuclear Instruments and Methods in Physics Research Section A*. **764** (2014).

## Session 5

# Structure and dynamics in Novel Materials

### **Multiferroic Thin Films (A Comprehensive Neutron Scattering Investigation)**

**Clemens Ulrich<sup>a</sup>**

<sup>a</sup> *School of Physics, University of New South Wales, Kensington 2052, Australia.*

Artificially grown thin film heterostructures of transition metal oxides exceed the capabilities of current semiconducting technology as they offer further functionalities such as metal-insulator transitions, magnetism, superconductivity, or multiferroicity. Bismuth ferrite ( $\text{BiFeO}_3$ ) is the rare case of a room temperature multiferroic material and opens as such the most promising pathway for spintronics applications. The existence of a spin cycloid is a mandatory requirement to establish a direct magnetoelectric coupling. Thus far, internal strain in epitaxial grown films has limited the stability of the spin cycloid for  $\text{BiFeO}_3$  films with less than 300 nm thickness when grown on  $\text{SrTiO}_3$ . Our neutron diffraction experiments have demonstrated that we were able to realize a spin cycloid in films of just 100 nm thickness through improved electrostatic and epitaxial constraints [1]. Further fascinating examples are  $\text{SrCoO}_3$  thin films. Theoretical calculations have predicted ferromagnetic (FM) to antiferromagnetic (AFM) phase transitions induced by epitaxial strain [2]. With the proper choice of substrate material we were able to confirm the FM-AFM transition by neutron diffraction. As such,  $\text{SrCoO}_3$  would constitute a new class of multiferroic material where magnetic and electric transitions can be driven through external strain [3]. This opens new avenues for fundamental research and technical applications in spintronic or magnonic devices.

[1] J. Bertinshaw, R. Maran, S.J. Callori, V. Ramesh, J. Cheung, S. A. Danilkin, W. T. Lee, S. Hu, J. Seidel, N. Valanoor, and C. Ulrich, *Nature Communications* **7**, 12664 (2016).

[2] J. H. Lee and K. M. Rabe, *Phys. Rev. Lett.* **107**, 067601 (2011).

[3] S. J. Callori, S. Hu, J. Bertinshaw, Z. J. Yue, S. Danilkin, X. L. Wang, V. Nagarajan, F. Klose, J. Seidel, and C. Ulrich, *Phys. Rev. B* **91**, 140405(R) (2015).

## Experimental and theoretical approaches to understanding selective gas adsorption in metal-organic frameworks

**J.E. Auckett<sup>a</sup>**, S.G. Duyker<sup>b</sup> and V.K. Peterson<sup>a</sup>

<sup>a</sup> Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, New South Wales 2234, Australia.

<sup>b</sup> School of Chemistry, The University of Sydney, New South Wales 2006, Australia.

Porous solids such as metal-organic frameworks (MOFs) are considered promising candidates for many industrial gas-separation applications, especially due to their structural and chemical versatility with respect to traditional solid sorbents such as zeolites [1]. Rational tuning of such materials for improved performance requires that the interactions between the host framework and guest molecules be well-understood at the atomic level. Our research targets this detailed understanding of framework-guest systems using *in situ* and *operando* neutron scattering experiments, in which structure and dynamics are probed as a function of guest loading and temperature, along with comprehensive atomistic density functional theory-based (DFT) calculations from which various physical and dynamical properties can be extracted.

We are currently investigating several MOFs which display interesting sorption behaviours, such as shape-dependent binding, “reverse sieving” (i.e. selectively absorbing larger gas molecules while rejecting smaller ones) [2], and guest-responsive negative thermal expansion (NTE). This talk describes the suite of conventional and unconventional tools we have used to explore the structural and dynamic properties of these frameworks, yielding highly detailed information about their behaviour. Many of these methods can also be applied to a wide variety of systems involving host-guest interactions.

[1] J.R. Li, R.J. Kuppler and H.C. Zhou, *Chem. Soc. Rev.* **38**, 1477 (2009).

[2] L.J. McCormick, S.G. Duyker, A.W. Thornton, C.W. Hawes, M.R. Hill, V.K. Peterson, S.R. Batten and D.R. Turner, *Chem. Mater.* **26**, 4640 (2014).

## Type II $\text{Bi}_{1-x}\text{W}_x\text{O}_{1.5+1.5x}$ : a (3 + 3) – dimensional commensurate modulation that stabilises the fast ion conducting delta phase of bismuth oxide

**J. Wind<sup>a</sup>**, J. E. Auckett<sup>b</sup>, R. L. Withers<sup>c</sup>, R. O. Piltz<sup>b</sup>, R. Mole<sup>b</sup> and C. D. Ling<sup>a</sup>

<sup>a</sup> School of Chemistry, The University of Sydney, NSW 2006, Australia.

<sup>b</sup> Australian Centre for Neutron Scattering, ANSTO, NSW 2234, Australia.

<sup>c</sup> Research School of Chemistry, Australian National University, ACT 0200, Australia.

The high temperature cubic polymorph of bismuth oxide,  $\delta\text{-Bi}_2\text{O}_3$ , is the best intermediate-temperature oxide ionic conductor known. Unfortunately,  $\delta\text{-Bi}_2\text{O}_3$  is only stable from 750-830°C, limiting its use as an ionic conductor. In order to stabilise its average fluorite-type structure to room temperature, while preserving a large part of its conductivity, higher valent transition metals such as  $\text{Nb}^{5+}$ ,  $\text{Ta}^{5+}$ ,  $\text{Cr}^{6+}$ ,  $\text{Mo}^{6+}$  or  $\text{W}^{6+}$  can be introduced, resulting in a variety of complex modulated structures based on the fluorite-type subcell.

We have identified a new member of this class of (3+3)-dimensional modulated phases in the  $\text{Bi}_{1-x}\text{W}_x\text{O}_{1.5+1.5x}$  system, in which the modulation vector  $\epsilon$  ‘locks in’ to a commensurate value of 1/3 [2]. The structure was refined in a 3x3x3 supercell against single-crystal Laue neutron diffraction data. Detailed *ab initio* calculations were used to test and optimise the local structure of the oxygen sublattice around a single mixed Bi/W site. The underlying crystal chemistry was shown to be based on a transition from fluorite-type to pyrochlore-type via the formation of  $\text{W}_4\text{O}_{18}$  ‘tetrahedra of octahedra’. The full range of occupancies on this mixed Bi/W site give a solid-solution range bounded by  $\text{Bi}_{23}\text{W}_4\text{O}_{46.5}$  ( $x = 0.148$ ) and  $\text{Bi}_{22}\text{W}_5\text{O}_{48}$  ( $x = 0.185$ ).

AC impedance measurements show promising results with ionic conductivities comparable to yttria stabilized zirconia. *Ab initio* molecular dynamics simulations combined with quasi-elastic (QENS) and inelastic neutron scattering (INS) experiments give first insights into the dynamics of the conduction process and diffusion mechanisms in these materials.

[1] C. Ling et al., *J. Am. Chem. Soc.* **135**: 6477-6484 (2013).

[2] J. Wind et al., *Acta Cryst.* **B71**: 679-687 (2015).

## **Magnetic structures of magnetocaloric ( $Mn_{1-x}Ni_x$ )CoGe and $Mn(Co_{1-x}Ni_x)Ge$ alloys**

**Q.Y. Ren**,<sup>1</sup> W.D. Hutchison,<sup>1</sup> J.L. Wang,<sup>2</sup> A.J. Studer,<sup>3</sup> W.T. Lee,<sup>3</sup> J.M. Cadogan<sup>1</sup> and S.J. Campbell<sup>1</sup>

<sup>1</sup>School of Physical, Environmental and Mathematical Sciences, The University of New South Wales, Canberra, ACT 2600, Australia

<sup>2</sup>Institute for Superconductivity and Electronic Materials, University of Wollongong, NSW 2522, Australia

<sup>3</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW 2234, Australia

The magnetocaloric effect (MCE) - a significant temperature change around the magnetic transitions in materials driven by magnetisation or demagnetisation - has emerged as an increasingly important topic in condensed matter physics in the past two decades. This development is due primarily to potential applications in refrigeration as an alternative to gas-based compression-expansion refrigeration [1]. A large MCE occurs generally around a magnetic transition, especially when the magnetic transition coincides with a structural transition (magneto-structural transition) [1].

MnCoGe-based compounds offer particular scope for MCE applications, particularly for cooling around room temperature with previous studies having shown that it is relatively straightforward to engineer the structural transition temperature and thereby produce a magneto-structural transition [2]. In the present work, a series of  $(Mn_{1-x}Ni_x)CoGe$  ( $x = 0.02-0.07$ ) and  $Mn(Co_{1-x}Ni_x)Ge$  ( $x = 0.14-1.00$ ) samples have been prepared in order to investigate the effects of doping the Mn and Co sites of MnCoGe with Ni. The crystal structures and magnetisation were measured using XRD (20-310 K) and PPMS (5-320 K) with the magneto-structural transitions studied using neutron powder diffraction and polarised neutron diffraction (5-450 K; WOMBAT, OPAL). Magneto-structural transitions from ferromagnetic-orthorhombic (FM-Orth) structure to paramagnetic-hexagonal (PM-Hex) structure were obtained in both  $(Mn_{1-x}Ni_x)CoGe$  and  $Mn(Co_{1-x}Ni_x)Ge$  around room temperature. A spiral antiferromagnetic (SP-AFM) structure was also observed in the orthorhombic structure of  $Mn(Co_{1-x}Ni_x)Ge$  ( $x \geq 0.55$ ) at low temperature, following by a magnetic transition from SP-AFM to FM at higher temperature. In addition, the influence of magnetic field on the FM-Orth/PM-Hex magneto-structural transition was studied using field-dependent neutron diffraction (5-320 K; 0-9 T). Our investigations show that normal (inverse) MCE are obtained around the FM-Orth/PM-Hex (SP-AFM/FM) transitions in  $(Mn_{1-x}Ni_x)CoGe$  and  $Mn(Co_{1-x}Ni_x)Ge$ .

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[2] Q. Y. Ren, W. D. Hutchison, J. L. Wang, A. J. Studer, M. F. M. Din, S. M. Pérez, J. M. Cadogan and S. J. Campbell, *J. Phys. D: Appl. Phys.* **49**, 175003 (2016)



## **Inelastic neutron scattering of lanthanoid single molecule magnets**

M. Vonci<sup>a</sup>, M.J. Giansiracusa<sup>a</sup>, **R. A. Mole**<sup>b</sup>, A. Soncini<sup>a</sup> and C. Boskovic<sup>a</sup>

<sup>a</sup> School of Chemistry, University of Melbourne, VIC, 3010, Australia

<sup>b</sup> Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2225, Australia

With their signature energy barrier to magnetic relaxation and quantum tunnelling through this barrier, single-molecule magnets (SMMs) are important candidate molecules for future applications in molecular spintronics and quantum computation. Among the most promising SMMs are those based on trivalent lanthanoid ions (Ln-SMMs). One of the most powerful experimental techniques for elucidating the electronic structure of SMMs is inelastic neutron scattering (INS), which provides a direct probe of the relevant energy levels. INS is very sensitive to the electronic structure of the lowest lying energy levels of Ln(III) ions, which are dominated by crystal field (CF) splitting effects. Despite these advantages, relatively few INS spectra with well-defined magnetic scattering have been reported for Ln-SMMs.

We have recently completed a study of two structural families of Ln(III)-polyoxometalates:  $\text{Na}_9[\text{Ln}(\text{W}_5\text{O}_{18})_2]$  (Ln = Nd, Tb, Ho, Er) and  $\text{Na}_{11}[\{\text{Ln}(\text{OH}_2)\}_3\text{CO}_3(\text{PW}_9\text{O}_{34})_2]$ , (Ln = Ho, Er). In both cases the INS measurements have been analysed using both a conventional crystal field and a more comprehensive *ab initio* approach. In the current contribution I will address the issues related to getting high quality neutron scattering data and summarise what has been learnt about the rational design of SMMs from this series of experiments.

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- [2] Vonci, M.; Giansiracusa, M.J.; Gable, R.W.; Van den Heuvel W.; Latham, K.; Moubaraki, M.; Murray, K.S.; Yu, D.; Mole, R.A.; Soncini, A.; Boskovic *Submitted*
- [3] Giansiracusa, M.J.; Vonci, M.; Van den Heuvel, W.; Gable, R.W.; Moubaraki, B.; Murray, K.S.; Yu, D.; Mole, R.A.; Soncini, A.; Boskovic, C. *Inorg. Chem.* **2016**, 55, 5201

## Session 6 Magnetism and Spectroscopy

### **Neutron Scattering Study in Breathing Pyrochlore Antiferromagnet $Ba_3Yb_2Zn_5O_{11}$**

**T. Masuda<sup>a</sup>**

<sup>a</sup> *Institute for Solid State Physics, the University of Tokyo, Kashiwanoha, Kashiwa, 277-8581, Japan*

Comprehensive study on breathing pyrochlore antiferromagnet  $Ba_3Yb_2Zn_5O_{11}$  [1] is presented. To identify the energy scheme of crystalline electric field (CEF), we performed inelastic neutron scattering (INS) measurement in high energy range. The observed dispersionless excitations are explained by a CEF Hamiltonian of Kramers ion  $Yb^{3+}$  of which the local symmetry exhibits  $C_{3v}$  point group symmetry. The magnetic susceptibility is consistently reproduced by the energy scheme of the CEF excitations [2]. To identify the spin Hamiltonian we performed INS experiment in low energy range and thermodynamic property measurements at low temperatures. The INS spectra are quantitatively explained by spin-1/2 single-tetrahedron model having  $XXZ$  anisotropy and Dzyaloshinskii-Moriya interaction. This model has a two-fold degeneracy of the lowest-energy state per tetrahedron and well reproduces the magnetization curve at 0.5 K and heat capacity above 1.5 K. At lower temperatures, however, we observe a broad maximum in the heat capacity around 63 mK, demonstrating that a unique quantum ground state is selected due to extra perturbations with energy scale smaller than the instrumental resolution of INS. Possible mechanisms for the ground state selection are discussed [3].

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## The Frustrated Quantum Spin Chain, Linarite, In High Magnetic Fields

B. Willenberg<sup>1,2</sup>, S. Nishimoto<sup>3</sup>, M. Schaeppers<sup>3</sup>, M. Reehuis<sup>1</sup>, A.U.B. Wolter<sup>3</sup>, S.-L. Drechsler<sup>3</sup>, B. Buechner<sup>3,4</sup>, A. Studer<sup>5</sup>, **K.C. Rule**<sup>5</sup>, B. Ouladdiaf<sup>6</sup>, and S. Suellow<sup>2</sup>

<sup>1</sup>Helmholtz Center Berlin for Materials and Energy, D-14109 Berlin, Germany

<sup>2</sup>Institute for Condensed Matter Physics, TU Braunschweig, D-38106 Braunschweig, Germany

<sup>3</sup>Leibniz Institute for Solid State and Materials Research IFW Dresden, D-01171 Dresden, Germany

<sup>4</sup>Institute for Solid State Physics, D-01069 TU Dresden, Dresden, Germany

<sup>5</sup>Australian Centre for Neutron Scattering, ANSTO, Kirrawee DC NSW 2234, Australia

<sup>6</sup>Institute Laue-Langevin, F-38042 Grenoble Cedex, France

Linarite,  $\text{PbCuSO}_4(\text{OH})_2$  is a natural mineral ideally suited to the study of frustration in  $J_1$ - $J_2$  systems due to an accessible saturation field and the availability of large single crystals well suited to neutron investigations. In this one dimensional  $J_1$ - $J_2$  model, competing ferromagnetic nearest-neighbour interactions ( $J_1 > 0$ ) and antiferromagnetic next-nearest-neighbours ( $J_2 < 0$ ) can give rise to novel phenomena such as multiferroicity for spiral spin states. It is also predicted that materials which exhibit such frustrated magnetic interactions are likely to display evidence of spin-nematic states. The magnetic spin-nematic phase can be likened to the arrangement of molecules in nematic liquid crystal displays (LCD). The magnetic form of the spin-nematic state, involves the ordering of spin-quadrupole moments in the absence of conventional spin-dipole order such that the magnetic spins align spontaneously along a chosen axis while still fluctuating dynamically.

In Linarite, the  $\text{Cu}^{2+}$  ions form spin  $S = 1/2$  chains along the  $b$  direction with dominant nearest-neighbour FM interactions and a weaker next-nearest-neighbour AFM coupling, resulting in a magnetically frustrated topology [1, 2].

We present a neutron scattering and magnetic property study of linarite revealing a helical magnetic ground state structure with an incommensurate propagation vector of  $(0 \ 0.186 \ \frac{1}{2})$  below  $T_N = 2.8\text{K}$  in zero magnetic field [3]. From detailed measurements in magnetic fields up to 12 T ( $B \parallel b$ ), a very rich magnetic phase diagram will be presented (Fig. 1) [4]. A two-step spin-flop transition is observed, transforming the helical magnetic ground state into a collinear structure. As well, a magnetic phase with sine-wave modulated moments parallel to the field direction was detected, enclosing the other long-range ordered phases, and which exhibits phase separation in high magnetic fields. Theoretical calculations imply that linarite possesses an xyz exchange anisotropy. Our data establish linarite as a model compound of the frustrated one-dimensional spin chain, with ferromagnetic nearest-neighbour and antiferromagnetic next-nearest-neighbour interactions. We shall also discuss the high field phase (marked “?” in the phase diagram of Fig. 1) in terms of the spin-nematic physics as well as the hard to access regions of the phase diagram, namely Region II.

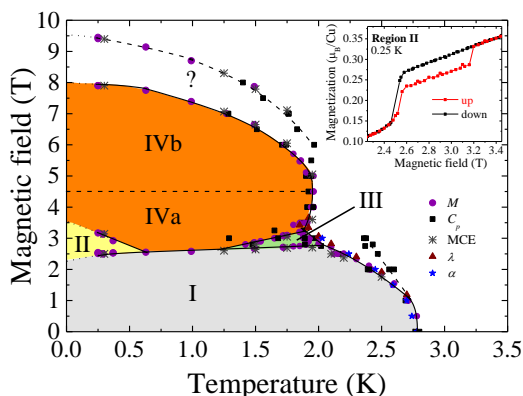


Figure 1: Complex magnetic phase diagram of Linarite including sample magnetization curve measured within Region II at 0.25K from [4].

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## Spin-reorientation in Quaternary $Dy_2Fe_2Si_2C$

R.A. Susilo<sup>a</sup>, **J.M. Cadogan**<sup>a</sup>, W.D. Hutchison<sup>a</sup>, G.A. Stewart<sup>a</sup>, M. Avdeev<sup>b</sup> and S.J. Campbell<sup>a</sup>

<sup>a</sup> School of Physical, Environmental and Mathematical Sciences, UNSW Canberra at the Australian Defence Force Academy, Canberra BC, ACT 2610, Australia.

<sup>b</sup> Bragg Institute, ANSTO, Lucas Heights, NSW 2234, Australia.

The low temperature magnetic properties of  $Dy_2Fe_2Si_2C$  have been investigated by magnetisation, specific heat, neutron powder diffraction and  $^{57}Fe$  Mössbauer spectroscopy measurements. In contrast to other  $R_2Fe_2Si_2C$  compounds [1-4], we found that  $Dy_2Fe_2Si_2C$  undergoes two successive magnetic transitions at low temperatures. The first magnetic transition at  $T_N = 26(2)$  K is associated with the transition from paramagnetic to antiferromagnetic states, whereas our neutron diffraction and  $^{57}Fe$  Mössbauer spectroscopy studies reveal that the second magnetic transition at  $T_t = 6(2)$  K is likely related to a spin-reorientation of the Dy moments rather than the independent ordering of the Fe sublattice. The magnetic structure above  $T_t$  can be described with a propagation vector  $\mathbf{k} = [0\ 0\ 1/2]$  with the ordering of the Dy magnetic moments along the monoclinic b-axis, whereas on cooling below  $T_t$  the Dy moment tips away from the b-axis towards the ac-plane. Magnetocrystalline anisotropy energy calculations show that a canted magnetic structure is more energetically favourable below  $T_t$  than b-axis order due to the important influence of higher-order crystal field terms at low temperatures, thus explaining the unique occurrence of spin reorientation in  $Dy_2Fe_2Si_2C$  compared with other  $R_2Fe_2Si_2C$  compounds.

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## ***Magnetization enhancement by exchange coupling of antiferromagnetic nanoparticles embedded in ferromagnetic matrix***

Xiang Ding<sup>a</sup>, Li-Ting Tseng<sup>a</sup>, Wai Tung Hal Lee<sup>b</sup> and **Jiabao Yi**<sup>a</sup>

<sup>a</sup> *School of Materials Science and Engineering, University of New South Wales, New South Wales 2052, Australia.*

<sup>b</sup> *Australian Centre of Neutron Scattering, Australian Nuclear Science and Technology Organisation, Lucas Heights, New South Wales 2234, Australia.*

We have observed enhanced magnetisation as a consequence of the exchange-coupling of antiferromagnetic nanoparticle embedded in ferromagnetic matrix. Magnetic behaviour in nanostructure has been found to be significantly different from the magnetic behaviour in bulk materials. As an example, Ni cluster has a saturation moment of 2  $\mu\text{B}$  compared to 0.6  $\mu\text{B}$  in bulk form [1]. In our previous work, we found NiO nanoparticles to be a ferromagnet with a saturation magnetization of 105 emu/g at 5 K, corresponding to 1.16  $\mu\text{B}/\text{Ni}$  [2]. It should be noted that bulk NiO is an antiferromagnet. However, the high saturation magnetization in these materials can only be observed at very low temperature. In 2003, Skumryev et. al. found that nanostructured Co had an enhanced blocking temperature when coated with antiferromagnetic CoO due to the exchange coupling between Co core and CoO surface [3]. Exchange coupling has therefore been proposed to be a mechanism that can lead to room-temperature high-magnetisation materials. To test this hypothesis, we fabricated Ni/NiO composite thin films using laser-MBE method. Magnetisation measurement using SQUID and layer-by-layer chemical and magnetic structure determination using polarized neutron reflectivity both showed that nanostructured NiO embedded in Ni matrix has led to an enhancement of room-temperature magnetisation higher than that of pure Ni films, thus providing evidence that supports the hypothesis.

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## Session 7

# Neutron Reflectometry to explore the Nanoworld

### ***The Influence of Glass Transitions on Diffusion in OLED Stacks***

Jake A. McEwan<sup>1</sup>, **Andrew J. Clulow**<sup>1</sup>, Paul E. Shaw<sup>1</sup>, Andrew Nelson<sup>2</sup>, Nageshwar Rao Yepuri<sup>3</sup>, Tamim Darwish<sup>3</sup>, Paul L. Burn<sup>1</sup> and Ian R. Gentle<sup>1</sup>

<sup>1</sup>Centre for Organic Photonics & Electronics, School of Chemistry and Molecular Biosciences, The University of Queensland, St Lucia QLD 4072, Australia

<sup>2</sup>Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia

<sup>3</sup>National Deuteration Facility, Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia

Of all of the organic electronic devices thus far conceived, organic light emitting diodes (OLEDs) have been the most successfully applied in a commercial setting. With OLED displays now available in the television and portable device markets, the appetite for their continued development continues to garner considerable research interest. Optimised OLED device architectures typically comprise a number of organic layers with thicknesses between 10 nm and 100 nm sandwiched between inorganic electrodes. Each of the organic layers used in the device is sequentially deposited in an order that optimises charge transport and capture, and light emission from the devices. The fidelity and stability of these multilayer organic stacks is therefore of paramount importance in determining the efficiencies and operational lifetimes of OLED devices.

Neutron reflectometry is a powerful technique for probing the layered structures found within OLEDs by utilising selective deuteration to provide contrast between or within the layers.<sup>1-3</sup> Modelling the changes in the neutron reflectivity profiles of the OLED stacks deposited onto smooth substrates allows for the visualisation of changes in the layered structure in a non-destructive manner. In this talk we will outline our recent efforts to relate the thermal properties of the organic materials used in OLED devices with their diffusion behaviour under thermal stress. Our collaboration with the National Deuteration Facility has led to the synthesis of a number of previously unobtainable deuterated analogues of semiconducting molecules typically used in OLEDs and that have a range of thermal characteristics.<sup>3-5</sup> These molecules were used in time-resolved reflectometry experiments that have allowed us to systematically build up an understanding of the importance of glass transitions for the stability of OLED stacks.

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## ***X-ray and Neutron Reflectivity Study of the Membrane-bound CLIC1 Protein at the Air-Water Interface***

**Khondker Rufaka Hossain<sup>a</sup>**, Stephen A Holt<sup>b</sup>, Heba Al Khamici<sup>a</sup> and Stella M Valenzuela<sup>a</sup>

<sup>a</sup> *School of Life Sciences, University of Technology Sydney, Sydney, New South Wales, Australia,*

<sup>b</sup> *Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, New South Wales, Australia.*

The CLIC proteins are a ubiquitous family of Chloride Intracellular Ion Channel proteins that are evolutionarily conserved across species and exist as a soluble form and an integral membrane-bound form <sup>1</sup>. The X-ray structure of the soluble form of a number of CLIC proteins has been solved and their putative transmembrane domain identified <sup>2-5</sup>. However, the factors which facilitate the membrane insertion, the structural transition between these two forms and the structural features of the membrane-bound form for this class of proteins remain largely unknown. In an attempt to answer these questions, we have employed biophysical techniques to study the interaction of wild-type and mutant versions of the protein CLIC1 with monolayers prepared using various mixtures of different phospholipids and sterol molecules. Our findings have demonstrated that cholesterol plays a crucial role for the penetration of CLIC1 into the hydrophobic tails of the lipid monolayer with the protein occupying an area per molecule between 5-7 nm<sup>2</sup>. We have also demonstrated for the first time that CLIC1 interaction with cholesterol is dependent on the intact 3 $\beta$ -OH group in the sterol ring and that the GXXXG motif in CLIC1 acts as the cholesterol-binding site used by the protein for its initial recognition and binding to membrane cholesterol.

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## **SPATZ: The Second Time-of-Flight Neutron Reflectometer at the OPAL Research Reactor**

**A.P. Le Brun**<sup>a</sup>, S. Pullen<sup>a</sup>, P. Constantine<sup>a</sup>, J. Spedding<sup>a</sup>, D. Roach<sup>a</sup>, A. McGregor<sup>a</sup>, J. Affleck<sup>a</sup>, J. Christoforidis<sup>a</sup>, M. Trapp<sup>b</sup> and R. Steitz<sup>b</sup>

<sup>a</sup> Australian Centre for Neutron Scattering, ANSTO, Lucas Heights, NSW, Australia.

<sup>b</sup> Helmholtz Zentrum Berlin, Germany.

In September 2015, an agreement was signed between HZB and ANSTO to transfer the V18 'BioRef' time-of-flight neutron reflectometer [1], currently situated at the 10 MW BER-II Research Reactor, to the OPAL Research Reactor. During 2016 preparations have been made to carry out the transfer of a neutron-scattering instrument halfway around the globe. This has involved a joint team of ANSTO and HZB personnel spending four weeks carefully disassembling BioRef and packing it into shipping containers for transport to ANSTO. Once the instrument arrives it will be known as SPATZ (German for Sparrow) and will be the 15<sup>th</sup> neutron-scattering instrument at OPAL.

SPATZ has a vertical sample geometry, which complements the current reflectometer, PLATYPUS, which has a horizontal sample geometry. The vertical sample geometry will allow for use of sample environments which cannot be currently used on PLATYPUS due to geometry constraints and allows for wide-angle diffraction from multilayers and lamellar stacks. SPATZ will also be equipped for simultaneous infra-red spectroscopy and reflectometry experiments, and will come with equipment for upgrades for polarisation and spin-echo techniques.

The instrument will view the OPAL cold neutron source (CNS) by taking the end position of the CG2B guide. Currently, the CG2B guide is installed between the primary and secondary shutters and part of the project scope is to complete the installation of the CG2B guide beyond the secondary shutter into the Neutron Guide Hall. The CG2B guide will accommodate SPATZ and an additional upstream instrument to be determined in the future. This presentation will provide an overview of the project, its current status, and future direction. Feedback from the neutron scattering community is encouraged.

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***The influence of controlled burning on the remobilizing of arsenic and selected metals from soils in the historic mining districts of Central Victoria and the possible health impacts***

**J. Abraham**, K. Dowling and S.K. Florentine  
*School of Applied & Biomedical Science,  
Faculty of Science and Technology, Federation University Australia,  
Mt. Helen Campus, Victoria 3350, Australia.*

Among the types of forest fire, controlled burning is the burning of naturally accumulated leaf litter on forest floor include bushes, which is a standard practice to reduce the fuel levels and hence minimize the likelihood of severe fires. It is usually conducted in cooler months, especially in spring and autumn in Australia. Controlled burning has the potential to affect the physical, chemical, biological and geological properties of the soils of the burned area. This may affect metals in the soil include its release from organic matter, particularly happen in mining affected landscapes. Fire increases erosion rate by as much as 2 to 100 fold, which facilitate rapid transport of remobilized metals by winds and running water raising contaminant levels, which increase its bioavailability. This is of concern as it may affect surface and groundwater quality and human and ecosystem health, particularly given that controlled burns are standard fire risk reduction strategy in Australia. Toxicity of metals can affect an organism's survival, activity, growth, metabolism and reproduction, particularly on children, who becomes exposed to metals to a greater extend than adults. It is reported that climate change and land use management practices have resulted in the increasing intensity and frequency of fires, required more number of controlled burns. This study aims to find the concentrations of selected metals and arsenic in the soils and waters of historic mining districts of Central Victoria in order to understand it's post-burn spatio-temporal variations. The result will be useful for post-burn metal related human and ecosystem health risk assessment in the region.

## ***Eight years in the life of ECHIDNA***

**M. Avdeev** and J. Hester

*Australian Centre for Neutron Scattering, ANSTO*

It has been eight years since the ECHIDNA instrument was open to users. Since then, more than 500 proposals have been completed on the instrument and more than 250 papers published as a result. In addition to scientific output, the accumulated experience of running ECHIDNA also provides interesting insights into its capabilities and usage. We will present statistics of various aspects of user experiments (including counting time distribution, instrument configuration and sample environment usage), examples of pushing the instrument limits (for example smallest samples, most absorbing materials, weakest magnetic moments), and an overview of research done on the instrument and in the ECHIDNA user community.

## ***A Peltier Controlled Sample Changer for SANS***

S. Lee<sup>a</sup>, N. Booth<sup>a</sup>, P. Imperia<sup>a</sup> and G. Davidson<sup>a</sup>

<sup>a</sup> *Australian Nuclear Science and Technology Organisation, Australian Centre for Neutron Scattering, New Illawarra Road, Lucas Heights, Australia*

A twelve position sample changer for SANS instruments at ACNS has been developed and will be available for users in the near future. The design philosophy for this project was to make the system as modular and scalable as possible with an emphasis on ease of use for the user and reduced background. The Peltier control system allows each sample to be controlled at different temperatures and allows the possibility of producing very fast temperature changes over a limited range. The system can be controlled via a local Watlow™ touchscreen and remotely via the SICS based neutron instrument control system used at ACNS. The liquid cooled heatsink for the Peltiers provides the support for the spring loaded sample holders, allowing rapid sample changes. The width of the sample changer has been reduced compared to the existing 20 position and with Borated aluminium shielding the background with blocked beam has been improved. The temperature sensors used are K-type thermocouples. Performance and stability tests will be presented. A design for uSANS is being developed.



## ***Inelastic Neutron Scattering of YbMn<sub>2</sub>Si<sub>2</sub> – Magnetic Interplay of Mn and Yb Sites***

R. A. Mole<sup>1,2</sup>, D. H. Yu<sup>1</sup>, M. Hofmann<sup>2,3</sup>, J. L. Wang<sup>2,4</sup> and **S. J. Campbell**<sup>2</sup>

<sup>1</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW2232

<sup>2</sup>School of Physical, Environmental and Mathematical Sciences, UNSW Canberra at the Australian Defence Force Academy, PO Box 7916, Canberra, BC 2610, Australia

<sup>3</sup>Heinz Maier Leibnitz Zentrum (MLZ), Technische Universität München, Lichtenbergstrasse 1, Garching, 85747, Germany

<sup>4</sup>Institute for Superconductivity and Electronic Materials, University of Wollongong, Wollongong, NSW 2522

The layered RT<sub>2</sub>X<sub>2</sub> series of compounds (R = rare-earth, T = 3d, 4d transition metal, X = Si, Ge) of tetragonal body centred ThCr<sub>2</sub>Si<sub>2</sub>-type structure (*I4/mmm*) is one of the most widely studied systems in condensed matter and materials science [e.g. 1]. Among the RT<sub>2</sub>X<sub>2</sub> family, Yb- and Eu-based intermetallics continue to attract strong scientific interest, mainly as a result of their intermediate valence character and the related wide range of unusual physical and magnetic properties [e.g. 2].

Having delineated the spectral features of YbMn<sub>2</sub>Si<sub>2</sub> in the region of the layered antiferromagnetism from T<sub>N1</sub> = 526(4) K to T<sub>N2</sub> = 30(5) K [3], we have investigated the crystal field splitting of YbMn<sub>2</sub>Si<sub>2</sub> by inelastic neutron scattering using PELICAN over the temperature range 5-65 K. The high resolution - 800 μeV and dynamic range ~14 meV - available at λ = 2.375 Å has enabled the additional excitations observed below T<sub>N2</sub> – the temperature below which the magnetic cell is doubled along the *c*-axis - to be investigated in detail. The results have been interpreted in terms of a crystal field model in which Yb<sup>3+</sup> ions have a unique environment above T<sub>N2</sub> with the doubled magnetic cell below T<sub>N2</sub> leading to inequivalent sites for the Yb<sup>3+</sup> ions. The calculated excitation spectra show good agreement with the observed spectra both above and below T<sub>N2</sub>. In particular the low temperature model describes a molecular field with components in the x, y and z directions with the x and y components related to the significant contraction of ~ 0.1 % of the *c* lattice parameter at T<sub>N2</sub>.

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**Rigid unit modes dynamics in the molecular sieve material  $\text{AlPO}_4\text{-5}$** D. L. Cortie<sup>a,b</sup>, B. R. McBride<sup>a</sup>, R.A. Mole<sup>b</sup>, R. Withers<sup>a</sup>, D. Yu<sup>b</sup>, G. McIntyre<sup>b</sup> and Y. Liu<sup>a</sup><sup>a</sup> *Research School of Chemistry, Australian National University*<sup>b</sup> *The Australian Nuclear Science and Technology Organisation*

The web-like ring structure composed of corner-sharing tetrahedra in  $\text{AlPO}_4\text{-5}$  has received considerable attention over the years because its unusual topology enables entrapment and diffusion of guest molecules within the extended micropores<sup>1</sup>. Recent work on this molecular sieve reported clear evidence for strong sub-picosecond tetrahedral dynamics attributed to rigid-unit modes (RUMS). In order to preserve the bond-lengths during these motions, some chemical species undergo restricted rotational diffusion within relatively large volumes around their average lattice positions. These motions drive fluctuations in the effective pore-window sizes, potentially acting to filter absorption and diffusion of guest species. Here we report a quasi-elastic neutron scattering investigation of the dynamics over the temperature range 1.5 – 300 K using the PELICAN instrument. This is compared with *ab initio* molecular dynamics to gain a clear microscopic picture of the timescales of the entangled motions within the tetrahedra network.

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## **Application of Linear Spin Wave Theory to the Cr<sub>8</sub> Antiferromagnetic Heisenberg Ring**

**T. D'Adam**<sup>a</sup>, R. Mole<sup>a</sup> and J. Stride<sup>b</sup>

<sup>a</sup> ACNS, Australian Nuclear Science and Technology Organisation, Lucas Heights, Australia.

<sup>b</sup> School of Chemistry, University of NSW, Kensington, Australia.

The investigation of single molecule magnets (SMMs) has proven to be a focal point of magnetism research for over three decades, leading to the discovery of structures which may find applications in data storage, quantum information processing (QIP) and spintronics. Though molecular magnetism is not a new field, there are still many complexes to investigate and understand, including a range chains, rings, discs and cages. Amongst the considerable number of structures, particular interest has been shown to antiferromagnetic Heisenberg rings (AFHR) such as Cr<sub>8</sub>, CsFe<sub>8</sub> and Fe<sub>18</sub>.

These structures have been investigated due to their interesting magnetic behaviours which include quantum tunnelling of the Neel vector (QTNV) and a long magnetic relaxation time below their blocking temperature TB [1]. The Cr<sub>8</sub> homometallic AFHR is one of the most well understood structures of its type having been extensively investigated since its initial synthesis using techniques including high-field EPR, cantilever torque magnetometry [2] and INS [3]. Through application of Linear Spin Wave Theory (LSWT) using the SpinW Matlab library [4] it has been possible to calculate the dynamic structure factor of the Cr<sub>8</sub> ring; this agrees well with both the INS data collected for this structure as well as models produced using alternate methods [3]. This demonstrates that LSWT is applicable to the Cr<sub>8</sub> ring and we plan to use this method to analyse more complex structures which also do not exhibit long range magnetic ordering.

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## Temperature Effects in Lattice Dynamics of SnSe

**S. Danilkin**<sup>a</sup>, D. Yu<sup>a</sup>, G. Deng<sup>a</sup>, E. Pomjakushina<sup>b</sup>, H. Liu<sup>c</sup>, G. Kearley<sup>a</sup>

<sup>a</sup> ANSTO, Lucas Heights, Australia

<sup>b</sup> Paul Scherrer Institut, Villigen, Switzerland

<sup>c</sup> Shanghai Institute of Ceramics, Shanghai, China

Tin selenide demonstrates a record high thermoelectric figure of merit [1]. The distinctive feature of this material is very low thermal conductivity directly related to peculiarities of SnSe lattice dynamics. We studied the temperature dependence of the crystal structure, phonon dispersion curves and vibrational density of states (VDOS) of SnSe in temperature range from 300 to 750 K using neutron scattering instruments at ANSTO. We found that frequencies of longitudinal and transverse acoustic phonons in SnSe have remarkably low frequencies, in particular the TA [100] phonon branch. Another feature of SnSe is the presence of low-frequency optic phonons in close proximity to acoustic branches and softening of TO<sub>b</sub> [100] mode. Measurements of VDOS during heating reveal rather complex modifications of the spectrum showing a significant softening of phonon groups centred at ~ 7.5 and 16 meV related to displacive Pnma – Cmcm phase transition at 830 K. The high density of low-energy modes along with observed strong phonon damping is probably responsible for the low values of lattice component of thermal conductivity in this material.

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## Dynamical Mechanism of Phase Transitions in A-site Ferroelectric Relaxor (Na<sub>1/2</sub>Bi<sub>1/2</sub>)TiO<sub>3</sub>

**Guochu Deng**<sup>a\*</sup>, Sergey Danilkin<sup>a</sup>, Haiwu Zhang<sup>b</sup>, Paolo Imperia<sup>a</sup>, Xiaobing Li<sup>b</sup>, Xiangyong Zhao<sup>b</sup>, Haosu Luo<sup>b</sup>

<sup>a</sup> Bragg Institute, Australian Nuclear Science and Technology Organization, Locked Bag 2001, Kirrawee DC, NSW 2232, Australia

<sup>b</sup> Shanghai Institute of Ceramics, Chinese Academy of Sciences, 215 Chengbei Road, Jiading, Shanghai 201800, China

\*Email: guochu.deng@ansto.gov.au

The dynamical phase transition mechanism of (Na<sub>1/2</sub>Bi<sub>1/2</sub>)TiO<sub>3</sub> (NBT) was studied using inelastic neutron scattering. Softening of multiple phonon modes were observed to correlate with the phase transition sequence of NBT. As usual, the softening of the zone centre transverse optic (TO) modes Δ<sub>5</sub> and Σ<sub>3</sub> was observed in (200) and (220) zone, showing the Ti vibration instabilities in TiO<sub>6</sub> octahera for both cubic-tetragonal (C-T) and tetragonal-rhombohedral (T-R) phase transitions. In this two phase transitions, however, Ti<sup>4+</sup> has different preferential displacement directions. Surprisingly, the longitudinal optic (LO) mode also soften significantly toward zone centre in the vicinity range of the transition temperature, indicating the Na<sup>+</sup>/Bi<sup>3+</sup> vibration instability against TiO<sub>6</sub> octahera during the R-T phase transition. Strong inelastic diffuse scattering shows up near M(1.5, 0.5, 0) and R(1.5, 1.5, 0.5) in the tetragonal and rhombohedral phases, respectively, indicating the condensations of the M<sub>3</sub> and R<sub>25</sub> optic modes for the corresponding phase transitions. This reveals the rotation instabilities of TiO<sub>6</sub> in the corresponding phase transition temperature range. Bottleneck or waterfall features were observed in the dispersion curves at certain temperatures, but did not show the close correlations to the formation of polar nanoregions (PNRs). Additional instabilities are the origin of the complexity of phase transitions and crystallographic structures in NBT.<sup>[1]</sup>

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[1] G. Deng et al. Phys. Rev. B V90 134104 (2014)

***Structural characterization by Small Angle Scattering suggests models for monomeric and dimeric forms of full-length ezrin.***

Phang JM, Harrop SJ, **Duff AP**, Sokolova AV, Crossett B, Walsh JC, Beckham SA, Nguyen CD, Davies RB, Glöckner C, Bromley EH, Wilk KE, Curmi PM.

Ezrin is member of the ERM (Ezrin-Radixin-Moesin) family of proteins that have been conserved through metazoan evolution. These proteins have dormant and active forms, where the latter links the actin cytoskeleton to membranes. ERM proteins have three domains: an N-terminal FERM (band Four-point-one ERM) domain comprising three subdomains (F1, F2 and F3); a helical domain; and a C-terminal actin-binding domain. In the dormant form, FERM and C-terminal domains form a stable complex. We have determined crystal structures of the active FERM domain and the dormant FERM:C-terminal domain complex of human ezrin. We observe bistable array of phenylalanine residues in the core of subdomain F3 that is mobile in the active form and locked in the dormant form. As subdomain F3 is pivotal in binding membrane proteins and phospholipids, these transitions may facilitate activation and signaling. Full-length ezrin forms stable monomers and dimers. We used small-angle x-ray scattering to determine the solution structures of these species. As expected, the monomer shows a globular domain with a protruding helical coiled-coil. The dimer shows an elongated dumbbell structure that is twice as long as the monomer. By aligning ERM sequences spanning metazoan evolution, we show that the central helical region is conserved, preserving the heptad repeat. Using this, we have built a dimer model where each monomer forms half of an elongated anti-parallel coiled-coil with domain-swapped FERM:C-terminal domain complexes at each end. The model suggests that ERM dimers may bind to actin in a parallel fashion.



## ***An In Vitro Model to Investigate the Interactions Between Antimicrobial Peptides and the Outer Membrane of Gram-negative Pathogens***

**Mei-Ling Han**<sup>1</sup>, Hsin-Hui Shen<sup>2</sup>, Yan Zhu<sup>2</sup>, Anton P. Le Brun<sup>3</sup>, Stephen, A. Holt<sup>3</sup>, Kade Roberts<sup>1</sup>, Jiang-Ning Song<sup>2</sup>, Matthew A. Cooper<sup>4</sup>, Samuel M. Moskowit<sup>5</sup>, Tony Velkov<sup>1</sup>, Jian Li<sup>2</sup>

<sup>1</sup>Monash Institute of Pharmaceutical Sciences, <sup>2</sup>Faculty of Medicine, Nursing & Health Sciences, Monash University, Melbourne, Australia

<sup>3</sup>Bragg Institute, Australia Nuclear Science and Technology Organisation, Sydney, Australia

<sup>4</sup>Institute of Molecular Bioscience, the University of Queensland, Brisbane, Australia

<sup>5</sup>Vertex Pharmaceuticals, Boston, MA, United States

Increasing antibiotic resistance in Gram-negative bacteria led to polymyxins as the last-therapy. Polymyxins present their antimicrobial activity through an initial electrical interaction with lipid A in the outer membrane (OM) of GNB, and the most common mechanism of polymyxin resistance is through modifications of lipid A with positively charged groups, such as 4-amino-L-arabinose (L-Aar4N) or phosphoethanolamine (pEtN). However, it is notable that Gram-negative bacteria employ a combination of charge-charge repulsion mechanism and the modification to fatty acyl chains of lipid A to obtain high-level polymyxin resistance. Hence, we designed hydrophobic polymyxin-related lipopeptides in order to overcome modified lipid A to insert into the outer membrane of Gram-negative bacteria. In this study, we employed neutron reflectometry (NR) study to investigate the interactions between lipid A and polymyxins.

Lipid A was extracted from polymyxin-susceptible and -resistant *pseudomonas aeruginosa* strains, and analysed using ESI-MS in the negative ion mode. The asymmetric lipid A: deuterated DPPC bilayers were deposited on SiO<sub>2</sub> surfaces by combined Langmuir-Blodgett and Langmuir-Schaefer disposition methods, and characterised by neutron reflectometer.

Our results showed L-Ara4N modified lipid A was observed in polymyxin-resistant PAK*pmrB6* strain, but not in the wild-type PAK strain. The NR data obtained from unmodified lipid A: DPPC bilayer was fitted into a five-layer model. Whereas, a six-layer model containing an extra outer headgroup was established for L-Ara4N modified lipid A: d-DPPC bilayer. Our results showed a dense of PMB (volume fraction of >20%) bound to the surface of both unmodified and modified lipid A: DPPC bilayers. While it is notable that the significant changes in NR profiles obtained from H<sub>2</sub>O contrast indicated about 15.8% and 6.1% of PMB penetrated into the wild-type lipid A headgroup and fatty acyl chains, respectively, but without penetration into L-Ara4N-lipid A: d-DPPC bilayer. However, the employment of octapeptin A3 induced higher hydrophobic interactions with L-Ara4N-lipid A: d-DPPC bilayer. Our study provides an in vitro model to investigate the interactions of polymyxins with OM bilayers in GNB, and confirmed that lipid A modification with L-Ara4N was certainly to reduce the penetration of PMB into bacterial membranes. Remarkably, the higher binding affinity between octapeptin A3 and L-Ara4N modified lipid A indicated its potential to be the new-generation antibiotics for the therapy of infections caused by multi-drug resistant Gram-negative bacteria.

## Specific Anion Effects on Thermoresponsive Polymer Brushes

**B. A. Humphreys, T.J. Murdoch<sup>a</sup>, J.D. Willott<sup>a</sup>, A. Nelson<sup>b</sup>, S.W. Prescott<sup>c</sup>, G.B. Webber<sup>a</sup> and E.J. Wanless<sup>a</sup>**

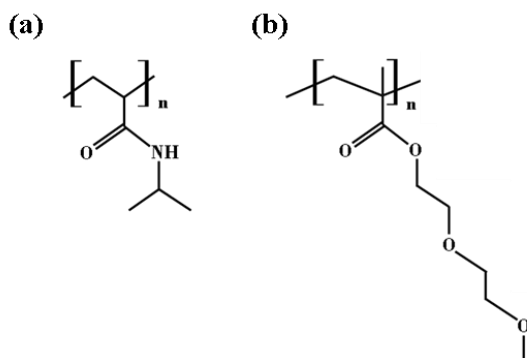
<sup>a</sup> Priority Research Centre for Advanced Particle Processing and Transport, University of Newcastle, NSW 2234, Australia.

<sup>b</sup> Australian Nuclear Science and Technology Organisation, NSW 2234, Australia.

<sup>c</sup> School of Chemical Engineering, UNSW Australia, NSW 2052, Australia

We have recently investigated the influence of anion identity on the physical properties of two different thermoresponsive polymer brushes via neutron reflectometry, atomic force spectroscopy and ellipsometry. [1-3] Polymer brushes of (a) poly(*N*-isopropylacrylamide) (PNIPAM) and (b) poly(2-(2-(methoxyethoxy)ethyl methacrylate) (PMEO<sub>2</sub>MA) were exposed to potassium salts of acetate and thiocyanate. The temperature response of both polymer brushes was shifted to lower temperatures in the presence of the kosmotropic acetate anions and higher temperatures when exposed to chaotropic thiocyanate anions.

The response of the PMEO<sub>2</sub>MA brush was considerably broader as a function of temperature and the shift in temperature in response to the added salt was greater compared to that displayed by the PNIPAM brush.



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- [1] B. A. Humphreys et al., *PCCP*, **18**, 6037 (2016)
- [2] T. J. Murdoch et al., *Macromolecules*, **49**, 6050 (2016)
- [3] T. J. Murdoch et al., *JCIS*, submitted (2016)

**EMU, the high resolution backscattering spectrometer at ANSTO**Nicolas R. de Souza<sup>a</sup>, **Alice Klapproth**<sup>a</sup> and Gail N. Iles<sup>a</sup><sup>a</sup> Australian Nuclear Science and Technology Organisation, New Illawarra Road, Lucas Heights NSW 2234, Australia

The energy range and resolution of backscattering spectrometers are well suited to characterizing relaxations on an atomic and molecular scale, such as diffusion processes occurring in e.g. polymer chains, membranes, proteins, molecular crystals, between interstitial crystal lattice sites. The EMU spectrometer can be used to study the dynamics of water molecules in the confined space of a host structure or ionic diffusion in conductor materials. In addition, quantum rotational tunnelling of functional groups (e.g. -CH<sub>3</sub>, -NH<sub>4</sub>) and hyperfine splitting of nuclear energy levels can be investigated. Relaxation times from a few 10 ps to over 1 ns are accessible.

We will present the first -CH<sub>3</sub> tunneling and diffusional motion spectra, obtained during the instrument commission, as an example of EMU's present capabilities. The experiments have been performed in a temperature range from 3 – 650K, using top- and bottom-loading cryo-furnaces. Other sample environments such as pressure, magnetic fields, controlled gas delivery systems, sub-K cryostats etc. are also available or currently under testing. EMU entered user service in 2016 and we welcome proposals in a wide range of scientific disciplines.

The EMU instrument has the highest energy resolution of the neutron spectrometers at ANSTO and provides a momentum transfer range from as low as 0.1 Å<sup>-1</sup> up to 1.95 Å<sup>-1</sup>. The high energy resolution is obtained by neutron backscattering, which occurs twice, through spherical focusing onto the sample, located between the Si (111) crystal monochromator and the analyser arrays [1]. A linear Doppler drive modulates the incident neutron energies over an energy range of ± 31 μeV. The inelastic scattered neutrons are counted in two <sup>3</sup>He linear-position sensitive detector arrays.

[1] Nicolas R. de Souza, Alice Klapproth & Gail N. Iles (2016), Neutron News, 27:2, 20-21, DOI:10.1080/10448632.2016.1163985, <http://dx.doi.org/10.1080/10448632.2016.1163985>

## Using $^{10}\text{Be}$ to estimate soil residence times (SRT) and tolerable erosion rates in Northern Territory, Australia

**Rajeev Lal**<sup>a,d</sup>, Keith Fifield<sup>b</sup>, Stephen Tims<sup>a</sup>, Robert Wasson<sup>b</sup> and Dave Howe<sup>c</sup>

<sup>a</sup>Department of Nuclear Physics, The Australian National University, ACT 0200, Australia.

<sup>b</sup>Department of Geography, National University of Singapore, Singapore 117570.

<sup>c</sup>Charles Darwin University, Darwin, NT, 0810, Australia.

<sup>d</sup>Fiji National University, Lautoka, Fiji.

We use *meteoric*  $^{10}\text{Be}$  measurements from soil cores to estimate SRT and geological erosion rates of a kandosol (a red loamy soil) in the Daly Basin in Northern Territory, Australia. In addition *in situ*  $^{10}\text{Be}$  in quartz from surface rocks was used to determine the rock erosion rates. The long term soil and rock erosion rates were used as a proxy for soil production rates and hence the tolerable erosion rates.

The kandosols are deep unconsolidated soil-saprolite systems (>2 m) which have developed on ~190 m thick Dolostone, which is a carbonate rock of ooid and stromatolitic dolostone and minor dolomitic sandstone, of Cambrian-Ordovician age and is the main geological formation underlying much of the Daly Basin.

We estimate SRT after correcting *meteoric*  $^{10}\text{Be}$  inventory for  $^{10}\text{Be}$  losses by decay and assuming that loss of  $^{10}\text{Be}$  in solution is minimal in these weakly acidic soils (pH 5.5-6.5). Our estimates of minimum SRT range between 0.3-2.3 Ma, using an estimated  $^{10}\text{Be}$  delivery of  $0.5 \times 10^6$  atoms  $\text{cm}^{-2} \text{yr}^{-1}$ . The corresponding geological erosion rate range is between 1.4-20.1  $\text{m Ma}^{-1}$ . The *in situ*  $^{10}\text{Be}$  concentrations in quartz from surface rocks were found to be in the range of  $1.16\text{-}5.74 \times 10^5$  atoms/g/qtz. The corresponding surface rock erosion rates range from 4.6-30.2  $\text{m/Ma}$ . The average Quaternary erosion rate hence the tolerable erosion rates on the lowlands of the central Daly River catchment was found to be 10.2  $\text{m/Ma}$ .

We find the measured geologic denudation rates are comparable to rates measured in other parts of monsoonal Northern Australia [1] and are at least two orders of magnitude lower than modern erosion rates [2].

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[2] R. Lal, L. K. Fifield, S. Tims, R. Wasson, D. Howe, Nucl. Instrum. Meth. Phys. Res. B **294**, 577 (2013).

## **SPATZ: The Second Time-of-Flight Neutron Reflectometer at the OPAL Research Reactor**

**A.P. Le Brun**<sup>a</sup>, S. Pullen<sup>a</sup>, P. Constantine<sup>a</sup>, J. Spedding<sup>a</sup>, D. Roach<sup>a</sup>, A. McGregor<sup>a</sup>, J. Affleck<sup>a</sup>, J. Christoforidis<sup>a</sup>, M. Trapp<sup>b</sup> and R. Steitz<sup>b</sup>

<sup>a</sup> Australian Centre for Neutron Scattering, ANSTO, Lucas Heights, NSW, Australia.

<sup>b</sup> Helmholtz Zentrum Berlin, Germany.

In September 2015, an agreement was signed between HZB and ANSTO to transfer the V18 'BioRef' time-of-flight neutron reflectometer [1], currently situated at the 10 MW BER-II Research Reactor, to the OPAL Research Reactor. During 2016 preparations have been made to carry out the transfer of a neutron-scattering instrument halfway around the globe. This has involved a joint team of ANSTO and HZB personnel spending four weeks carefully disassembling BioRef and packing it into shipping containers for transport to ANSTO. Once the instrument arrives it will be known as SPATZ (German for Sparrow) and will be the 15<sup>th</sup> neutron-scattering instrument at OPAL.

SPATZ has a vertical sample geometry, which complements the current reflectometer, PLATYPUS, which has a horizontal sample geometry. The vertical sample geometry will allow for use of sample environments which cannot be currently used on PLATYPUS due to geometry constraints and allows for wide-angle diffraction from multilayers and lamellar stacks. SPATZ will also be equipped for simultaneous infra-red spectroscopy and reflectometry experiments, and will come with equipment for upgrades for polarisation and spin-echo techniques.

The instrument will view the OPAL cold neutron source (CNS) by taking the end position of the CG2B guide. Currently, the CG2B guide is installed between the primary and secondary shutters and part of the project scope is to complete the installation of the CG2B guide beyond the secondary shutter into the Neutron Guide Hall. The CG2B guide will accommodate SPATZ and an additional upstream instrument to be determined in the future. This presentation will provide an overview of the project, its current status, and future direction. Feedback from the neutron scattering community is encouraged.

[1] M. Strobl et al., *Rev. Sci. Instrum.* **82**, 055101 (2011)

**Neutron and X-ray studies of TiAl-Nb intermetallics undergone high-pressure torsion**

**Xi Li**<sup>1,2</sup>, Rian Dippenaar<sup>2</sup>, Megumi Kawasaki<sup>3</sup>, Klaus-Dieter Liss<sup>2,1</sup>

<sup>1</sup>School of Mechanical, Materials & Mechatronics Engineering, University of Wollongong, NSW 2522, Australia

<sup>2</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW 2234, Australia

<sup>3</sup>Division of Materials Science and Engineering, Hanyang University, Seoul, South Korea  
Email:cissy346@gmail.com

High-pressure torsion is a severe-plastic-deformation technique rendering the bulk metallic material into an ultrafine microstructure. Not only the application of high pressure in the 6 GPa range bears potential for phase transformation, moreover huge plastic shear strains of 10 to 100 is achieved by torsion processing. In a first study, pellets of  $\gamma$ -based Ti-45Al-7.5Nb have been processed under 6 GPa at room temperature, (i) with pressure loading and unloading only, (ii) with 5 turns of torsion and (ii) 10 turns. The material, which usually is brittle and hard to deform, was successfully processed and showed ductility under these conditions. First investigations by neutron and X-ray diffraction are presented, emphasizing the complementarity of both kinds of quantum beams. While X-rays determine the overall structure, such as close-packing, neutrons are particularly sensitive to the order parameter in the constituting  $\gamma$ -TiAl and  $\alpha_2$ -Ti<sub>3</sub>Al intermetallic phases. It is found that the atomic order decreases on larger amount of processing. Also structural transformations regarding the atomic packing take place. These preliminary examinations open routes for physical understanding and recovery of the occurring crystallographic transformation and microstructural arrangements.



## ***In-Situ Diffraction Studies Related To Thermo-Mechanical Processes in Metals and Alloys***

**Klaus-Dieter Liss**<sup>1,2</sup>, Xi Li<sup>1,2</sup>, Rian Dippenaar<sup>2</sup>

<sup>1</sup>Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW 2234, Australia

<sup>2</sup>School of Mechanical, Materials & Mechatronic Engineering, University of Wollongong, NSW 2522, Australia

Email: kdl@ansto.gov.au, liss@kdliss.de, web site: <http://liss.freeshell.org>

Both neutron and synchrotron high-energy X-rays have penetrating power into metals, and intensities are competitive for ex- and in-situ studies of thermo-mechanical processes. They bear great potential in order to speed up materials design by orders of magnitude. The present contribution will enhance novel pioneering experiments on selected metal systems and showcase the complementarity between neutrons and X-rays and to other in-situ techniques, such as the Laser Confocal Scanning Microscope. Neutrons bear the advantage of averaging over larger volumes and therefore, are less dependent on grain statistics, leading to good, quantitative phase analysis and texture measurements. Phase evolutions are studied upon application of high temperature and high pressure. The neutron contrast, different to X-rays has been employed to investigate order-disorder transitions in titanium-aluminides. Moreover, dynamical theory of diffraction leads to the study of the smallest distortions and their kinetics at high temperature in zirconium and titanium alloys. Synchrotron X-rays allow t focusing on a small number of crystallites, showing up traces of grain evolution in reciprocal space, such as grain rotation, grain growth, phase correlations, dynamic recovery and recrystallization such as in a Materials Oscilloscope.

## **Calibration of temperature controllers and sensors**

**A. Manning**, N. Booth and P. Imperia

*Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW 2234, Australia.*

Reliable temperature measurements are a key part of many sample environment setups for neutron scattering experiments. This typically requires the use of both a temperature sensor and temperature controller. It is often desirable to be able to perform a continuous measurement spanning a wide range of temperatures, however the choice of suitable sensors is limited. Rhodium iron (RhFe) resistive thermometers are best suited to the temperature range of our cryofurnaces (1.5 to 800 K), however their calibration is known to drift when exposed to elevated temperatures.

Here, we will present procedures to ensure that both the controller and sensor used in such experiments are performing optimally. Calibrated resistors, which can mimic the behavior of a temperature sensor fixed at a particular temperature, can be used to check the calibration of the readings given by temperature controllers such as Lake Shore 336/340 or Oxford Instruments iTC units. If the controllers are known to be operating to the tolerances specified by their manufacturer, then we can have confidence in the measurements that they make.

We can then use our newly verified controllers to assess the performance of RhFe sensors over a range of temperatures from 4 to 800 K in a custom-built calibration rig. This allows us to quantify the behavior of the sensors after they are subject to high temperatures, and to account for shifts in their readings. Using this procedure, we can ensure that the sensors remain reliable after prolonged operational use.

## **High-pressure single-crystal neutron diffraction**

**G.J. McIntyre**<sup>a</sup>, J. Binns<sup>b</sup> and S. Parsons<sup>c</sup>

<sup>a</sup> *Australian Nuclear Science and Technology Organisation, Lucas Heights NSW 2234, Australia.*

<sup>b</sup> *Centre for High-Pressure Science & Technology Advanced Research, Shanghai, PRC.*

<sup>c</sup> *Centre for Science at Extreme Conditions, School of Chemistry, University of Edinburgh, Edinburgh EH9 3JJ, UK.*

High-pressure neutron diffraction is always challenging, but it can offer several advantages over high-pressure X-ray diffraction to make meeting those challenges worthwhile. In addition to the usual higher sensitivity to low-X elements, notably hydrogen, and to magnetic moments, the low absorption by many pressure cell materials can yield greater reciprocal-space coverage for single crystals. The low scattering power usually requires considerably larger sample volumes than with X-rays, but for the same reason the cell-wall materials can be quite thick.

Common cell designs include He-gas cells and simple clamp cells, opposed piston cells (e.g. Bloch, McWhan), opposed-anvil cells (e.g. diamond anvil cell, Paris-Edinburgh cell), and multi-anvil cells, each adapted to sample volume, accessibility, pressure, and other external parameters, especially temperature, that suit the scientific question of interest. State-of-the-art experiments using each cell type will be described.

A special challenge in high-pressure diffraction is to perform neutron and X-ray experiments on the same material under the same conditions. Previously, this meant using different cells and samples with achieving identical pressures a hit-or-miss affair. This has all changed with the recent demonstration on KOALA on the OPAL research reactor that modern neutron Laue diffraction can be performed on the same sample in the same diamond-anvil cell as used for laboratory X-ray experiments [1].

[1] J. Binns, K. V. Kamenev, G. J. McIntyre, S. A. Moggach and S. Parsons, *IUCrJ*, 3, 168, (2016).

**Linear spin wave theory calculations for a powder sample: excitations in the distorted kagomé lattice  $Mn_3(1,2,4-(O_2C)_3C_6H_3)_2$** **R. A. Mole<sup>1,2</sup>, D. H. Yu<sup>1</sup>, K.C. Rule<sup>1</sup>, J.A.Stride<sup>2</sup> and P.T.Wood<sup>3</sup>**<sup>1</sup> Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2225, Australia<sup>2</sup> Department of Chemistry, University of New South Wales, Sydney, NSW 2052, Australia<sup>3</sup>Department of Chemistry, Lensfield Rd. Cambridge, CB2 1EW, UK

The measurement of spin waves from magnetic crystals has been a longstanding success story of inelastic neutron scattering. The development of linear spin wave theory [1,2] predates the first neutron scattering measurements and is well developed and suited to the determination of spin waves from magnetic ions with large spins. However to date there has been limited success in applying spin wave theory to lower symmetry crystal structures; such structures are commonly occurring in molecule based magnetic systems whereby the packing of the organic ligands often favours lower symmetry space groups. Further the determination of powder averaged spectra has previously been difficult. Recently spin wave calculation software has become readily available; one such package SpinW [3] readily address the problems of low symmetry and powder averaging. A rotation matrix strategy is implemented so that it is suitable for incommensurate structures. In this contribution we will demonstrate the power of these techniques using data obtained on the cold neutron time-of-flight spectrometer PELICAN for the distorted kagomé lattice  $Mn_3(1,2,4-(O_2C)_3C_6H_3)_2$ . [4] Our interest in the distorted kagomé lattice compound stems from the observation of a combination of magnetic ordering and glassy behaviour in a distorted frustrated network. Our inelastic neutron scattering and spin wave theory results allow us to characterise all of the exchange interactions in the distorted lattice and relate these to the degree of frustration in the lattice.

[1] Bloch, *F. Zeitschrift für Phys.* **1930** 61, 206,[2] Slater, J.C. *Phys. Rev.* **1930**, 35, 509[3] Toth, S. Lake, B. *J.Phys. Cond. Mat.* ,**2015**, 27, 166002[4] Mole, R.A, Greene, S. Henry, P.F., Humphrey S.M., Rule, K.C., Unruh, T. Weldon G.F., Yu, D. Stride J.A., Wood, P.T., *Submitted to Inorg Chem.*

## **Recent upgrades to ANSTO's Thermal Triple Axis Spectrometer 'TAIPAN'**

**K. Rule**<sup>1</sup>, **S. Olsen**<sup>1</sup>, S. Danilkin<sup>1</sup>, F. Darmann<sup>1</sup>, T. Oste<sup>1</sup>, D. Bartlett<sup>1</sup>, T. Kafes<sup>1</sup>, A. Mcgregor<sup>1</sup>, A. Stampfl<sup>1</sup>, T. Ersez<sup>2</sup>, A. Orgrin<sup>3</sup>.

<sup>1</sup> *The Australian Centre for Neutron Scattering, ANSTO, Lucas Heights 2234.*

<sup>2</sup> *Nuclear Analysis Section, ANSTO, Lucas Heights 2234.*

<sup>3</sup> *Engineering and Capitol Projects Division, ANSTO, Lucas Heights 2234.*

The thermal triple axis spectrometer, TAIPAN, has been operational since 2010 and employs a graphite monochromator to access neutron energy transfers up to 80meV. A recent string of projects have greatly increased the capabilities of this instrument. Initially the wall shared with the cold TAS, SIKA, was modified to allow access to larger regions of Q-Energy space for TAIPAN. A more recent upgrade project has involved developing a new sapphire filter translation stage mechanism and a new Cu-200 double-focussing monochromators. The copper monochromator has been installed back to back with the previous highly ordered PG-monochromator and extends the accessible energy range of the neutrons from 70meV to 180meV.

## **PELICAN: Examples from the first two years of operation**

**R. A. Mole**<sup>1</sup>, G.N. Iles<sup>1</sup> and D. H. Yu<sup>1</sup>

<sup>1</sup> Australian Nuclear Science and Technology Organisation, Locked Bag 2001, Kirrawee DC, NSW 2225, Australia

The cold neutron time-of-flight spectrometer PELICAN has been in user operation since October 2014. Pelican is well suited to the study of low energy excitations and offers a diverse range of user experiments ranging from condensed matter physics to cell biology. PELICAN also includes the option of XYZ polarisation analysis using a supermirror polariser and a wide angle <sup>3</sup>He cell.

In this contribution we will give an overview of the technical details of the instrument, along with examples of experiments using recently commissioned instrument configurations and sample environment that have been brought into service. This includes high temperature scattering up to 1600 °C, in-situ gas and vapour delivery, single crystal spectroscopy and utilisation of harmonic wavelengths.

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## Magnetic Proximity Effect in YBCO/STO/LCMO Multilayers

O. Paull<sup>a,b</sup>, G. Causer<sup>a,b</sup>, A. Pan<sup>a</sup> and F. Klose<sup>b</sup>

<sup>a</sup> University of Wollongong

<sup>b</sup> Australian Nuclear Science and Technology Organisation (ANSTO)

Tailoring of the electronic properties of complex oxide heterostructures, thin films, and superlattices with atomically sharp interfaces is at the frontline of materials research at present [1, 2, 3]. Interfaces exhibit novel states that are not possible in bulk materials as a result of broken symmetry, induced strains, and modified exchange interactions.

In this work we examined the interaction between a superconducting  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  thin film layer and a ferromagnetic  $\text{La}_{2/3}\text{Ca}_{1/3}\text{MnO}_3$  layer using polarised neutron reflectometry.

The interaction at the interface between YBCO/LCMO multilayers has resulted in a variety of observed phenomena such as induced ferromagnetic moments in YBCO layers that are antiparallel to neighbouring LCMO layers, and a strong reduction in magnetization in LCMO near the interface. Prajapat et al. investigated the YBCO/LCMO interface using  $\text{SrTiO}_3$  (STO) as an intermediate insulating layer, and reported that the magnetic depletion (MD) in LCMO near the interface is dependent on the critical temperature of YBCO and the thickness of the LCMO layer, indicating that the origin of the MD in LCMO is due to Cooper pairs tunneling through the STO interlayer across the interface [5]. Our work has verified the observation of the MD layer in LCMO near the interface as claimed in the past report, and additionally studied the effect of a magnetic field on this layer. Polarised neutron reflectometry measurements performed at ANSTO on the PLATYPUS reflectometer indicate that the effect of applying fields at 0.03 T and 1 T is to cause a small restoration of magnetization in this depleted layer. This result supports the mechanism proposed by Prajapat et al. of superconductivity-induced MD from tunnelling through STO.

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[3] Fedoseev, S. A., Pan, A. V. et al., *ACS Nano* 7, 286-293 (2013)

[4] Hoppler, J. et al., *Phys. Rev. B* 82, 174439 (2010)

[5] Prajapat et al., *Nanoscale* 8, 10188-10197 (2016)



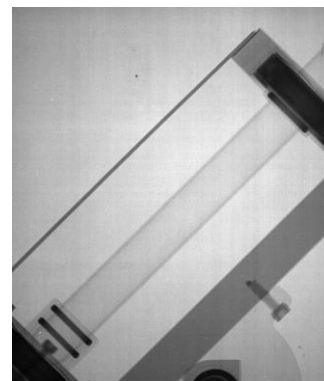
## **SANS and Neutron Imaging of Shear Stressed Single-Phase Systems for Otherwise Immiscible Liquids**

**J.T.E. Solheim**<sup>a,b</sup>, and C. L. Raston<sup>a,b</sup>

<sup>a</sup> School of Chemical and Physical Sciences, Flinders University, Adelaide, Australia.

<sup>b</sup> Flinders Centre of NanoScale Science and Technology, Flinders University, Adelaide, Australia.

Immiscible solvent systems are often necessary or desirable for a range of processes, from the synthesis and functionalization of carbon materials, to the execution of organic reactions with toxic metal catalysts whilst limiting metal contamination. Despite the advantages of immiscible solvent systems, the inability to form a homogenous mixture with conventional systems limits their viability. Here, we report the use of SANS and Neutron Imaging to show the formation of a single phase from typically immiscible liquids within the high-shearing environments present in a rapidly rotating tube. The microfluidic platform that allows this is the Vortex Fluidic Device (VFD) (inset, as seen under Neutron Imaging), which enables the formation of unusual carbon materials and accelerates chemical reactions [1,2]. SANS was used to investigate the homogeneity of a water/toluene mixture within the VFD, with results indicating the absence of particles in the 1 nm to 100 nm range. Neutron Imaging was subsequently used to investigate the time scale upon which this mixing becomes uniform, establishing that such mixing occurs on the order of seconds. These results provide useful insight into how fluid behaves in a high-shear environment, and further the understanding of the mechanism behind some of the remarkable capabilities of the VFD.



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**Investigations into the controllable change of Curie temperature in silver doped lanthanum manganite nanoparticles.**

**M. Westlake**<sup>a</sup>, M. Lerch<sup>a</sup>, K. Konstantinov<sup>a</sup>, K. C. Rule<sup>b</sup>, D Yu<sup>b</sup>, A Pan<sup>a</sup>, D. Cardillo<sup>a</sup>, J. Horvat<sup>a</sup>, and M. Tehei<sup>a</sup>

<sup>a</sup> School of Physics, University of Wollongong, New South Wales 2500, Australia.

<sup>b</sup> Bragg Institute, ANSTO, New South Wales 2234, Australia.

Our team is focused on research into the design, production, characterisation and implementation of optimized nanostructured particles for principally the diagnosis (as CT and MRI contrast agents) and treatment of cancer (using radiation, oncothermia and hyperthermia modalities). One magnetic nanoparticle of current interest is Lanthanum Manganite (LaMnO<sub>3</sub>) and its silver doped counterpart (La<sub>1-x</sub>Ag<sub>x</sub>MnO<sub>3</sub>). The high effective atomic number and magnetic moment of LaMnO<sub>3</sub> [1] makes this material appropriate for the basis of an MRI and CT contrast agent and enhancing radiation therapies. In addition La<sub>1-x</sub>Ag<sub>x</sub>MnO<sub>3</sub> is also considered as a good candidate for hyperthermia cancer therapy [2].

For the characterization of our samples we used XRD, PPMS, SEM, EDS and SEM. We observed that the Curie temperature increased with the increase of the silver doping concentration in the nanoparticles. This has led to an investigation into the mechanism behind this change. The spin-phonon interaction was considered to represent one potential mechanism and Time of Flight measurements were conducted on PELICAN at ANSTO. A lack of phonon evolution was seen within the temperature range of 1.5K - 300K. In order to access phonon density of states over a broader range of energies, we are then planning to use the Beryllium filter on TAIPAN and scan through a wide energy range while measuring scattered neutron over a vastly increased solid angle

The following poster will focus on the characterizations of our samples, our first experiments on PELICAN and our future planned experiments in the aim to better understand the mechanism that provokes the change of Curie temperature upon silver doping.

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## Determination of the Crystal Field Levels in $TmV_2Al_{20}$

**R. White**<sup>a</sup>, W.D. Hutchison<sup>a</sup>, G.N. Iles<sup>b</sup>, R.A. Mole<sup>b</sup>, J.M. Cadogan<sup>a</sup> and K. Nishimura<sup>c</sup>

<sup>a</sup> School of Physical, Environmental and Mathematical Sciences, The University of New South Wales, Canberra, ACT, 2600, Australia

<sup>b</sup> Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, Kirrawee DC, NSW, 2232, Australia

<sup>c</sup> Graduate School of Science and Engineering, University of Toyama, Toyama, 930-8555, Japan

There has been increasing interest in compounds of the  $RM_2Al_{20}$ -type (R = lanthanide, M = transition metal) in recent years due to the unique physical and magnetic properties many have been shown to display at low temperatures. Recent work carried out on  $PrV_2Al_{20}$  and  $PrTi_2Al_{20}$  has revealed a number of interesting phenomena, including a quadrupolar Kondo effect [1, 2] and superconductivity [3, 4] brought about by the cubic symmetry of the  $Pr^{3+}$  site inducing a non-magnetic ground state in the ion. As a hole analogue of the  $PrV_2Al_{20}$  compound,  $TmV_2Al_{20}$  has been investigated to see whether it too displays such phenomena at low temperatures. Crystal field calculations based on specific heat and magnetisation have been carried out previously [5] with parameters  $W = 0.5$  K and  $x = -0.6$  determined based on the Lea, Leask and Wolf formalism [6]. These results have been further refined to  $W = 0.42(1)$  K and  $x = -0.63(1)$  using inelastic neutron scattering data obtained from the PELICAN time-of-flight spectrometer located at the OPAL reactor, Lucas Heights.

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## **Low resolution structural studies of Munc18c complexed with a Syntaxin-4/T4-Lysozyme Fusion**

**Andrew E. Whitten**<sup>a,b</sup>, Asma Rehman<sup>b</sup>, Shu-Hong Hu<sup>b</sup>, Zakir Tnimov<sup>b</sup>, Michelle P. Christie<sup>b</sup>, Gordon J. King<sup>b</sup>, Russell J. Jarrott<sup>b</sup>, Suzanne Norwood<sup>b</sup>, Kirill Alexandrov<sup>b</sup>, Brett M. Collins<sup>b</sup>, Jennifer L. Martin<sup>b,c</sup>

<sup>a</sup> Australian Centre for Neutron Science, ANSTO, Lucas Heights NSW 2234

<sup>b</sup> Institute for Molecular Bioscience, University of Queensland, St Lucia QLD 4070.

<sup>c</sup> Eskitis Institute, Griffith University, Nathan QLD 4111.

Soluble N-ethylmaleimide-sensitive factor attachment protein receptors (SNARE) act at every intracellular trafficking pathway. Cognate *v*-SNAREs (e.g. VAMP) and *t*-SNAREs (Syntaxin (Sx) and SNAP) form a high affinity SNARE ternary complex (Sx-SNAP-VAMP) that brings the membranes together, triggering fusion. Syntaxins consist of a SNARE motif, and a three-helix bundle. In an open conformation, the SNARE motif is free to form the SNARE ternary complex (stimulating fusion), but in the closed conformation fusion is inhibited. Sec1p/Munc18 (SM) proteins bind to Sx, regulating SNARE mediated fusion [1], but their exact role is not well understood [2-4].

In the cell, Sx is bound to the membrane, and it is possible that this tethering may influence the manner in which it interacts with other proteins. As a means of investigating structural changes arising due to tethering, here, we investigate how the addition of a C-terminal T4-Lysozyme (soluble) fusion to Sx4 modulates its interaction with Munc18c. Preliminary low-resolution models of the Munc18c-Sx4T4 complex optimized against small-angle scattering data will be presented.

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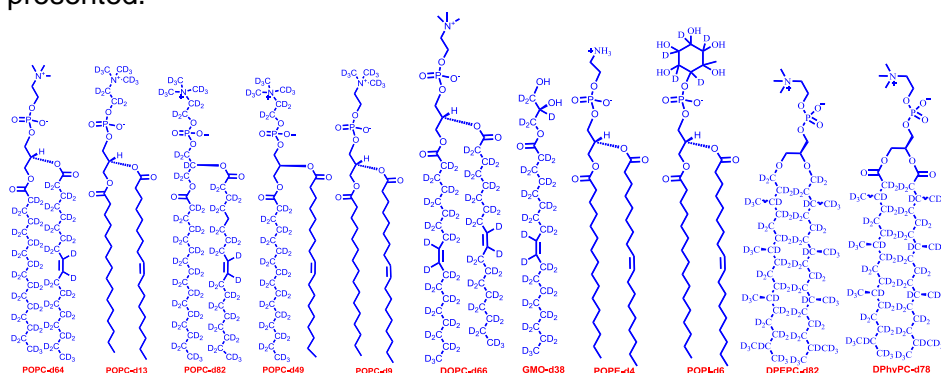
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## Synthesis of perdeuterated and selectively deuterated phospholipids and lipids for neutron applications

**Nageshwar R. Yepuri**, Tamim A. Darwish, Anwen Krause-Heuer, Anna Leung, Marina Cagnes and Peter J. Holden

The National Deuteration Facility, Bragg Institute, Australian Nuclear Science and Technology Organisation, Lucas Heights, New South Wales 2232, Australia.

The National Deuteration Facility (NDF) is focused on the provision of deuterated molecules which extends the options for contrast in neutron scattering to encompass not only solvent but molecular deuteration. Over the past few years the NDF has expanded its synthesis capability from simple deuterated fatty acids to complex deuterated molecules including lipids and phospholipids. We are now able to produce head or tail deuterated lipids including phospholipids based on oleic acid with a range of head groups (Fig. 1). These include perdeuterated 1,2-oleoyl-*sn*-glycero-3-phosphocholine (POPC), selectively deuterated POPC, branched chain (phytanic) phospholipids 1,2-diphytanoyl-*sn*-glycero-3-phosphocholine (DPhyPC), and perdeuterated mono oleoyl glycerol and phytanoyl monoethanolamide.[1] These lipids have been extensively used in constructing biologically more relevant model membranes and lipidic matrices for investigations using neutron studies. These include structural and dynamical studies of biomimetic membranes and the encapsulation of biomolecules in lipid-based bicontinuous cubic phases for drug-delivery, membrane protein crystallization, and biosensor applications. Details about design, synthesis and characterisation of these deuterated precursors and final compound will be presented.



**Figure 1:** Some of the NDF prepared phospholipids and lipids.

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## **Crystal growth and characterisation of a new J<sub>1</sub>-J<sub>2</sub> spin-chain material**

**Jack Zanardo**<sup>1</sup>, Kirrily C Rule<sup>1,2</sup>, Anwen Krause-Heuer<sup>3</sup>, Richard A Mole<sup>2</sup> and Michael Lerch<sup>1</sup>

<sup>1</sup> School of Physics, University of Wollongong, Wollongong NSW 2500 Australia

<sup>2</sup> Australian Centre for Neutron Scattering, ANSTO, Lucas Heights NSW Australia

<sup>3</sup> National Deuteration Facility, ANSTO, Lucas Heights NSW Australia

Recently a new one-dimensional (1D) quantum spin chain system has been synthesised: catena-dichloro(2-Cl-3Mpy)copper(II), [where 2-Cl-3Mpy=2-chloro-3-methylpyridine] [1]. Preliminary calculations and bulk magnetic property measurements indicate that this system does not undergo magnetic ordering down to 1.8K and is a prime candidate for investigating frustration in a J<sub>1</sub>/J<sub>2</sub> system (where the next nearest neighbour interactions, J<sub>2</sub>, are antiferromagnetic and the nearest neighbour interactions, J<sub>1</sub>, are ferromagnetic) [2]. Calculations predict 3 possible magnetic excitations below 6meV which may reveal the nature of the random static structural disorder predicted in this material. One method for directly observing the magnetic excitations is neutron scattering and measurements have been performed on the neutron Time of Flight spectrometer PELICAN at ANSTO [3]. To a first approximation, linear spin-wave theory has been used to model the expected neutron excitations for this J<sub>2</sub>/J<sub>1</sub> system using the Matlab package SpinW. The results of this project may provide valuable insight into the nature of magnetic frustration in materials.

To optimise the observed magnetic signal via the reduction of incoherent neutron scattering, this compound was deuterated at the National Deuteration Facility at ANSTO. In this presentation we will outline our deuterated growth procedure as well as the characterisation methods performed to understand the material further. This work forms the Honours thesis project of Jack Zanardo from University of Wollongong.

### References

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## **WOMBAT – High Intensity powder diffractometer at OPAL**

**A.J. Studer**, V.K. Peterson H.E. Maynard-Casely, J.R. Hester and C. Wang  
Australian Centre for Neutron Scattering, ANSTO.

Wombat is a high intensity neutron diffractometer located in the OPAL Neutron Guide Hall. It is primarily used as a high-speed powder diffractometer, but has also expanded into texture characterisation and single-crystal measurement, particularly diffuse scattering. The high performance comes from the combination of the best area detector ever constructed for neutron diffraction with the largest beam guide yet put into any research reactor and a correspondingly large crystal monochromator, all combine to provide an instrument which is unique in its capabilities within the Southern hemisphere.

Wombat has been used to explore a broad range of materials, including: novel hydrogen-storage materials, negative-thermal-expansion materials, methane-ice clathrates, piezoelectrics, high performance battery anodes and cathodes, high strength alloys, multiferroics, superconductors and novel magnetic materials.



**Quokka, the Pinhole Small Angle Neutron Scattering Instrument, at OPAL**

E. Gilbert, K. Wood, C. Garvey, J. Mata and C-M. Wu  
*Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, Australia.*

Quokka is a 40 metre small angle neutron scattering instrument installed on the cold source of the OPAL research reactor operated by ANSTO. The instrument is designed to enable measurements of scattering vectors over three orders of magnitude, from  $3 \times 10^{-4} \text{ \AA}^{-1} < q < 0.7 \text{ \AA}^{-1}$ . The instrument is able to perform polarized neutron experiments and a new high count rate detector will be commissioned on the instrument next year.

In addition to the standard 20-position automatic sample changer, a wide range of sample environment equipment is available for use on the beamline, some of which are unique worldwide. A rheometer, rapid heat/quench cell, dynamic light scattering, differential scanning calorimetry and a stop-flow mixing cell are all in use.

The capabilities and performance of Quokka will be presented.

**Scientific Highlights from Quokka, the Pinhole Small Angle Neutron Scattering Instrument, at OPAL**

E. Gilbert, K. Wood, C. Garvey, J. Mata and C-M. Wu  
*Australian Centre for Neutron Scattering, Australian Nuclear Science and Technology Organisation, Lucas Heights, NSW, Australia.*

Quokka is the 40 m pinhole SANS instrument at the OPAL reactor serving the growing needs of both domestic and international users [1]. In 2015, over 200 days of user experiments were run. Outputs from Quokka have been published that cover such diverse fields as magnetism, structural biology, mineralogy, polymers, food science and soft matter. We present here a selection of recent scientific highlights.

[1] [www.ansto.gov.au/ResearchHub/Bragg/Facilities/Instruments/Quokka/](http://www.ansto.gov.au/ResearchHub/Bragg/Facilities/Instruments/Quokka/)