

samples, both Cu⁺ and Cu²⁺ were detected, both incorporated in the glass matrix, the second one being responsible for the colour of the artefacts.

Keywords: archaeometry, glass, EXAFS

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Maya Blue: a Long Lasting Mystery Revealed

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The composition of Maya Blue, one of the most important pigments in antiquity, has puzzled scientists for decades. Recent studies using different analytical techniques including synchrotron and neutron powder diffraction, proved that the pigment is a combination of palygorskite (characterized by large channels), and the organic dye indigo.

Pigment preparation was repeated successfully and is easy (mix clay and dye and heat at ~100°C for a few hours). Chemical modelling showed indigo fitting the channels without impediment, thus it was assumed that indigo would fully or partially occupy the channels. However, energy calculations and thermal analyses showed indigo cannot penetrate into channels, since the water present does not leave the channels at synthesis temperature and strong H-bonds formed by the first molecule in the channels need to be broken.

A new concept of Maya Blue structure is presented, based on the fact that indigo only fill the grooves present at the crystal's surface. These grooves, which can be thought of as half channels split along cleavage planes, can equally well accommodate the dye molecule, forming one or two H-bonds explaining the stability and colour of the complex. A possible line of study to determine the provenance of Maya Blue is based on mutual abundance of the monoclinic and orthorhombic polymorph of palygorskite, determined via Rietveld refinement. . On the basis of the new theory all the facts previously hard to explain, become comprehensible.

Keywords: maya blue structure, palygorskite, synchrotron and neutron powder diffraction

MS59 APPLICATIONS OF SYNCHROTRON AND NEUTRON FACILITIES IN STRUCTURAL CHEMISTRY

Chairpersons: Paul Raithby, Trevor Forsyth

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Microcrystal X-ray Diffraction and MAS NMR Studies of Zeolites

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Zeolites are notoriously difficult to prepare as large single crystals. Until the last decade we have had to rely on powder diffraction as our major tool for determining their structure. With the advent of single crystal X-ray diffraction as a standard tool at synchrotron sources this limitation has been somewhat reduced. In this presentation I will discuss some of our recent work on studying the structure and properties of zeolites using the single crystal X-ray diffraction station at the Synchrotron Radiation Source, Daresbury, UK.

Not only have new structures been solved using this facility, but we have also shown how the details of previous powder X-ray diffraction work have sometimes been incorrect. In addition, the facility has also allowed us to crystallographically study the thermal properties of zeolites, such as negative thermal expansion, in more detail than was previously possible.

I will also explain how solid state MAS NMR studies can be used to improve the accuracy of our crystallographic models, and I will discuss the possibilities for solving structures from NMR data.

Keywords: zeolites, microcrystal diffraction, NMR

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Combined X-ray and Neutron Charge Density Studies on C-H activation catalysts

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The new single crystal diffractometer RESI (*RE*ciprocal Space Investigator) at the new high-flux neutron source FRM-II (www.frm2.tum.de) has been optimized for the demands of combined X-ray and neutron charge density studies (high resolution data at low backgrounds) by employing an X-ray and neutron sensitive imaging plate detector, a flexible κ -goniometer geometry and a focusing neutron guide for thermal neutrons. The new diffractometer concept will be outlined, and the advantage of combined X-ray and neutron charge density studies illustrated by first applications in the field of catalyzed C-H activation. We will demonstrate that so-called 'ligand-induced charge concentrations' in the valence shell of main group and transition metal complexes (*i*) can be identified by experimental charge density studies; and (*ii*) act as controlling parameters in C-H activation processes.[1,2] Systematic experimental studies of molecular charge distributions will thus lead to significant advances in the design and chemical control of catalysts for C-H activation and other processes, with central relevance to many reactions of academic and commercial importance.

[1] Scherer W., McGrady G.S., *Angew. Chem. Int. Ed.*, 2004, **43**, 1782. [2] Scherer W., Sirsch P., Shorokhov D., Tafipolsky M., McGrady G.S., Gullo E., *Chem. Eur. J.*, 2003, **9**, 6057.

Keywords: electron density, neutron diffraction, agostic interactions

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Structure and Dynamics of Co-ordination Compounds by Neutron Scattering

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The importance of transition metal hydrido-complexes, both classical and non-classical (i.e. compounds containing the M-H or M-H₂ moieties) in catalytic reactions, in particular hydrogenations, is well known, and much work has been carried out in recent years to obtain a detailed understanding of the nature and reactivity of the M-H and M-H₂ moieties.

Single crystal neutron diffraction, at low temperature, has been instrumental in establishing the correct co-ordination geometry in non-classical hydrides. It has been established that the H-H separation can cover a wide range of distances, from ~ 0.8 Å (strong H-H bond) to ~ 1.4 Å (weak H-H bond), corresponding to various degree of the H-H activation, thus neatly spanning the oxidative addition pathway and giving support to the mechanistic studies.

However, it is worth noting that a complete description of the reactivity in hydrido-compounds can only be achieved by combining the diffraction results with the knowledge of their dynamics that can be obtained from neutron incoherent inelastic scattering (INS) experiments.

I will discuss some recent structural results on transition metal poly-hydrides and show how, by combining neutron diffraction, INS and DFT calculations, the H₂ dynamics can be explained.

Keywords: transition metal hydrides, molecular hydrogen complexes, neutron scattering

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Location of a 4-coordinate H Atom via Neutron Diffraction

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