by H−N−H hydrogen bridges which further stiffen inorganic chains. Cations are connected with polyanions via ionic interactions directed by hydrogen bonds i.e. ionic hydrogen bonds.

Neighbouring, electroneutral chains are linked into layers of secondary structure by N−H⋯O hydrogen bonds. They are formed between cations and terminal oxygen ligands in one boundary mode or between cations and bridging oxygen ligands in the other extreme. One of our structures (salt **5**) exhibits intermediate manner of interlayer connection with both bridging and terminal oxygen ligands involved in the formation of secondary structure.

The layers are connected into 3D, tertiary structure by a variety of linkers ranging from N−H⋯O hydrogen bonds in salt **1**, a −CH2−CH2− moiety in salt **2**, C−H⋯π and van der Waals interactions in salt **3**, O⋯H_2O⋯HOH⋯O bridges in salt **4** O−H⋯O bridges in salt **5**. This shows that the modification of organic moiety in cation leads to significant changes in the tertiary structure of investigated salts. Final 3D molecular organisation is a result of fine interplay between interactions constituting secondary and tertiary structure.

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Keywords: ammonium arsenite, supramolecular structure, weak interactions

MS.67.4

Complex topologies as a result of simple relationships between 2D and 3D actinide borates

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Recently, we have found a very easy and productive method of actinide borate crystallization [1]. This method allowed us to synthesize more than 40 new actinide (Th, U, Np, Pu) phases, and to characterize them using methods like X-ray diffraction, UV-vis and MAS-NMR spectroscopy. With the only exception of Th borate [2], the structures of all the phases are based on complex actinide-borate sheets linked by BO_3 and/or BO_4 into 2D layered structures or 3D frameworks. The topologies of the oxoborate networks which incorporate the actinide atoms are quite diverse and complex. We have observed seven different topologies of 2D oxo-borate nets. Some of these nets are enantiomorphic and, thus, have different "left" or "right" configuration. Various stacking modes of such nets result in a significant number of structure types [3].

The structural complexity increases even more by insertion of BO_3 or BO_4 groups into the inter-sheets space. This kind of oxo-borate sheets modification can generate single or double layers, and 3D frameworks via poly-condensation of BO_2OH fragments [4]. The double layers can have between one and three BO_3 triangles within an inter-sheet space, and a total thickness of up to 2 nm [5, 6].

About 70% of actinide borates are non-centrosymmetric, because of the structure complexity of the layers and their possible different orientation. The lack of inversion allows for non-linear optical properties which, in fact, were shown to exist by measuring of SHG signals.

The layers can be functionalized via substitution of B-OH by B-F groups. This changes the structures dramatically as the substitution of terminal OH groups by F prevents the formation of hydrogen bond,

and thus the possibility of single layer condensation into double layers or 3D frameworks.

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Keywords: layered borates, framework, actinides

MS.67.5

Temperature and moisture dependent studies on kanemite (NaSi$_2$O$_4$(OH)*3H$_2$O)

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Kanemite is a naturally occurring hydrated layer sodium silicate that was first discovered in evaporites at Kanem, Lake Chad. Apart from being a mineralogical curiosity, synthetic kanemite has been studied frequently due to its feasible applications as absorbent, catalyst support and precursor for the preparation of mesoporous or microporous materials.

Concurrently, two groups reported the orthorhombic crystal structure of kanemite (space group type *Pbcn*, a=4.946, b=20.510, c=7.277Å) [1,2]. The compound is based on negatively charged corrugated [$Si_2O_4(OH)$]-sheets which contain six-membered tetrahedral rings. Sodium cations are coordinated by six water molecules in form of distorted octahedra. They are sandwiched between the layers. The cationic and anionic sub-structures are linked by hydrogen bonding.

Our investigations on the thermal behaviour and moisture sorption of kanemite indicate a stepwise release of structural water with increasing temperature and a partial dehydration below 15% relative humidity (RH).

The moisture sorption isotherm at 25°C was started at 43% RH where the sample contains about 40% water. On lowering the relative humidity in 5% steps to 15% RH the water content is constant but decreases towards dryer conditions to about 20% water. In the sorption cycle the water content increases in a distinct step between 35 and 50% RH and then increases almost exponentially. The highest RH value (98% RH) was obtained by storing the material over saturated potassium sulphate solution in a semimicro hygrostate, where the sample takes up more than 12 mol water. In the desorption cycle, the water content drops down quickly at 90% RH and releases about 2 mol water between 90 and 50% RH. The original water content is completely retained after the desorption/sorption cycle.

In a N_2-atmosphere, thermogravimetric analysis shows a first dehydration step between 25°C and 110°C in which two water molecules per formula unit are expelled from the kanemite structure. With increasing the relative humidity this partially dehydrated material transforms back into kanemite. The remaining amount of one mol of structural water is released between 110°C and 250°C.

In-situ high temperature X-ray powder diffraction of kanemite

in the range between 15 and 250°C reveals the occurrence of at least two partially dehydrated phases. From the evaluation of the collected patterns it can be concluded that the principal crystallographic differences between these two materials and the kanemite structure are due to a pronounced decrease of the lattice parameter perpendicular to the silicate layers (direction [010]).

An ab-initio structure determination by simulated annealing of the first dehydration product of kanemite with nominal composition $NaSi_2O_4(OH)*H_2O$ was successful. Whereas the silicate layers of the kanemite structure are retained almost unmodified, pronounced changes can be observed in the interlayer sheet containing the Na cations. The previously unknown phase adopts space group *Pbcn* as well. Lattice parameters at ambient conditions are as follows: a=4.888, b=15.332, c=7.203Å.

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Keywords: hydration, transition, silicate

MS.68.1

Data reduction of area detector measurements

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Time of flight Laue neutron diffraction a powerful tool to sample reciprocal space in a highly effective manner by collecting scattering data of a wide wavelength band simultaneously. Efficiency is further improved by highly pixelated large area detector coverage with excellent time resolution. This produces a wealth of data with every sample setting. Consequently large files of raw data need to be handled for data visualization, raw data corrections for incident spectrum variations, detector efficiency, background, sample effects and contributions. Furthermore, the software needs to be easy to use by new and experienced users alike. An increasingly important part of data collection is a strategy software that allows to tailor the measurement time and conditions to the sample symmetry and resolution requirements.

Examples of new and improved software developed and implemented at the TOPAZ single crystal diffractometer at the Oak Ridge National Laboratory will be presented. CrystalPlan helps define the measurement strategy and ISAW EV analyzes live neutron event data on the fly.

Keywords: data reduction, tof neutron single crystal diffraction, area detectors

MS.68.2

Applications of area detectors to texture measurements

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The use of 2-dimensional detectors in Quantitative Texture Analysis (QTA) has been pointed out long ago mainly in order to reduce acquisition times, both at x-ray and neutron diffraction instruments [1], [2]. Indeed, the necessity to acquire several pole figures to correctly refine the orientation distribution functions makes indispensable their simultaneous acquisitions. Since typically 1000 pole figure points have to be measured, using point detectors creates very often acquisition times over several days, a dramatic drawback particularly at scarce neutron beam times. Historically, the development and use of linear, then curved position sensitive (CPS) detectors helped in reducing the acquisition to several hours [3], even at neutron steady state reactors [4]. Furthermore, it opened the way to the treatment of the whole pattern simultaneously including QTA information and more, a procedure nowadays called Combined Analysis [5]. CCD cameras and image plate systems further offer fast QTA analysis with no loss in the capability of full-profiling the patterns. Curvature of image plate detectors can be operated to create cylindrical solid angles for x-ray instruments, and at neutron lines, shaping parts of cylinders with individual detector plates or wires has been recently developed. For all these 2D-detectors, the aim to reduce the number of sample orientations to be measured has been at least partly achieved.

We will illustrate the main scheme used to construct pole figures and calculate ODFs from area patterns, giving some exemples of the use of Combined Analysis, which includes the determination of structure, phase and microstructure also. One of the last developments allowed by the use of Curved Area PSDs is the determination of Magnetic Quantitative Texture Analysis [6], which we will illustrate also.

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Keywords: texture analysis, area detectors

MS.68.3

Modern trends in area detectors for single-crystal neutron diffraction

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Neutron single-crystal diffraction is the tool of choice to determine the accurate positions of hydrogen atoms in solids and the arrangements of magnetic moments. In some cases, hydrogen positions can be inferred from the positions of the other atoms, but if hydrogen bonding occurs, this is not reliable. Since neutron fluxes are typically low compared to those available at synchrotron X-ray facilities, there has been a major effort to maximize the exploitation of the available scattering data. Much emphasis has been placed in particular on the provision of large area detector systems. The D19 diffractometer at the ILL which has been equipped with a 120º x 32º multiwire proportional counter since 2007 provides greatly improved data quality for crystallographic work in structural chemistry, physics and the biosciences.

Another slightly less precise single-crystal diffraction method is the Laue method where the crystal is illuminated by a broad spectrum of wavelengths, and a large area of neutron-sensitive image plates (VIVALDI at the ILL, KOALA at ANSTO) is used to measure