

**[o.m3.p3] Structure Solution of the Highly Disordered Layer Silicate H-RUB-18** M. Borowski, H. Gies, B. Marler, *Ruhr-Universität Bochum*.

Keywords: layer silicate, structure solution from powder diffraction, MD-Simulation

H-RUB-18 is a layer silicate of the chemical formula  $Si_{32}O_{56}(OH)_{16}$ . At standard pressure two modifications of H-RUB-18 are known, a low temperature ( $\alpha$ -) form stable at temperatures  $T < \sim 343$  K and a high temperature ( $\beta$ -) form stable between  $\sim 343$  K and the decomposition temperature. One of H-RUB-18 remarkable properties is the intercalation of organic molecules<sup>1</sup>.

H-RUB-18 is obtained from the layer silicate Na-RUB-18 ( $Na_8[Si_{32}O_{64}(OH)_8] \times 32H_2O$ )<sup>2</sup> by ion exchange with diluted acids. From chemical analysis it is known that all  $Na^+$ -ions are replaced by protons. Karl-Fischer titration and thermal analysis proved that in contrast to most layer silicates H-RUB-18 contains no free hydrate.

The powder diffraction pattern of H-RUB-18 is of low resolution. Because of the poor periodic register of neighboring layers only a few broad reflections with anisotropic linewidths arise from the background. Therefore it is not possible to solve the structure only from the XRD-data. NMR studies give hints that the layerlike silicate anions of H-RUB-18 and Na-RUB-18<sup>2</sup> have the same topology. Moreover the NMR spectra indicated that the silicate units are fairly ordered at short range (i.e. within the layerlike unit).

Based on all experimental evidence an approximate model of the crystal structure was developed. This model served as starting conformation for MD-calculations, which have been performed with the program package *InsightII*<sup>3</sup> using a forcefield specifically developed and tested for this type of problem. Depending on the simulation conditions two reasonable structures were obtained, which are in good agreement with the powder pattern of the  $\alpha$ - or  $\beta$ - form of H-RUB-18. These structures are used as start values for the Rietveld refinement by TOPAS<sup>4</sup>. Since the simulation also showed different possible sites for the atoms, the resulting stacking faults become obvious and offer the possibility of a reasonable refinement using split positions for certain atoms.

**[o.m3.p4] Werner Clathrates: A New Polymeric Inclusion Compound.** M.L. Kilkenny, L.R. Nassimbeni., *Dept. of Chemistry, University of Cape Town, Rondebosch, 7701, Cape Town, South Africa.*

Keywords: Werner clathrate, inclusion compound, kinetics.

The x-ray crystal structure of  $Ni(NCS)_2(3-NH_2-C_5H_4N)_3 \cdot C_2H_5OH$  (**1**) has been solved. The coordination geometry around the Ni(II) centre is pseudooctahedral.

Compound **1** crystallises in polymeric chains, in which the amino group of one 3-aminopyridine ligand coordinates to an adjacent Ni centre, with the result that one substituted pyridine ligand acts as a bidentate ligand, whereas the other two are monodentate.

The thermal behaviour of compound **1** has been studied using Thermogravimetric Analysis and Differential Scanning Calorimetry, while the kinetics of guest release have been studied using Isothermal Techniques.

[1] K.Kosuge, P.S.Singh *Chem.Mater.* 12 (2000) 421-427

[2] S.Vortmann, J.Rius, S.Siegmann, H.Gies *J.Phys.Chem. B* 101 (1997) 1292-1297

[3] BIOSYM/MSI, InsightII Molecular Modeling System, User Guide, San Diego (1996)

[4] Bruker AXS, TOPAS V2.0: General Profile Analysis Software for Powder Diffraction Data. - User Manual, Karlsruhe (2000)