

[o.m3.o3] 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 (α -) form stable at temperatures $T < 343$ K and a high temperature (β -) form stable between ~ 343 K and the decomposition temperature. One of H-RUB-18 remarkable properties is the intercalation of organic molecules¹.

H-RUB-18 is obtained from the layer silicate Na-RUB-18 ($Na_8[Si_{32}O_{64}(OH)_8] \times 32H_2O$)² 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-18² 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*³ 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 α - or β - form of H-RUB-18. These structures are used as start values for the Rietveld refinement by TOPAS⁴. 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.o4] Structures of the K^+ and NH_4^+ Forms of Linde J. R.W. Broach^a and R.M. Kirchner^b, (a) *UOP LLC, Des Plaines, IL 60017 USA* and (b) *Manhattan College, Chemistry Dept., Bronx, NY 10471 USA*.

Keywords: Rietveld refinement, zeolite topology, Linde J

Zeolite Linde J was described in a patent by Breck and Acara¹ in 1961. E. M. Flanigen saved and kindly provided powder samples used in this study. The structure was solved in 1993 after structure solution methods for powders became more powerful, but it was not reported because of space group ambiguities. Recent Rietveld refinements have resolved the ambiguities.

Kirchner solved the structure of the as-synthesized K^+ form of Linde J using direct-methods on powder diffraction data collected by J. J. Pluth at beam line X7a at NSLS. The space group of highest topological symmetry for the Linde J framework is $P4_12_12$. In the as-synthesized form, the K^+ cations distort the framework and lower the symmetry. Determination of the correct space group requires parallel Rietveld refinements in subgroups of $P4_12_12$.

Preliminary x-ray powder diffraction studies^{2,3} on the NH_4^+ exchanged form of Linde J indicated tetragonal symmetry consistent with space group $P4_12_12$. However, this space group is not appropriate for the description of the ammonium cations or water molecules contained in the zeolite cavities.

Linde J co-crystallizes with zeolite K-F.⁴ To account for K-F impurities, a second phase (zeolite K-F has the EDI topology⁵) was included in all Rietveld refinements of the K^+ and NH_4^+ forms of Linde J.

The structures of the K^+ and NH_4^+ forms will be described, and the problems of determining the most appropriate space groups will be discussed.

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