

NMR and PXRD Analysis of the Structure of a New Hydrous Layer Silicate

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RUB-39 is a synthetic hydrous layer silicate with an unusual crystal structure. Upon heating neighboring layers condense to a framework silicate with empty zeolitic pores. The crystal structure RUB-39 (P 2/c, $a = 7.3312(1) \text{ \AA}$, $b = 10.7238(1) \text{ \AA}$, $c = 17.5135(1) \text{ \AA}$, $\beta = 115.6911(6)^\circ$) was solved from PXRD data and refined in its hydrated layered form with FULLPROF to final $\chi^2 = 3.1$. The diffraction analysis also allowed for a detailed analysis of the organic species used as structure directing agent (SDA) which is intercalated between silicate layers.

¹H solid state NMR experiments were used to study the role of the intercalated water and SDA-cation. The low field signal at 16.7 ppm indicates a strong hydrogen bond which is connecting neighboring silicate layers. This is unusual and might explain the topotactic condensation reaction upon heating. The spectrum also reveals that molecular water is intercalated in the interlayer space which is released only at temperatures above 170 °C.

PXRD and NMR studies following the further heating of the material to temperatures above 250°C showed the breakdown of the SDA and the condensation process of the silicate. Attempts to isolate the SDA-free layered material failed so far since concomitant to the release of the intercalated molecules the condensation to the framework silicate sets in.

Keywords: powder XRD, solid state NMR, hydrous layer silicate