ESRF	**Experiment title:** Single crystal diffraction experiments on disordered kaolinite	**Experiment number:** CH233
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Report:

Kaolinite, is a common clay mineral that exhibits a wide range of disorder. The subject of this study were highly disordered kaolinite samples. The powder &action pattern of these kaolinites reveal hardly any features besides a typical asymmetric and broad diffuse scattering. Single crystals of a kaolinite from Georgia, USA, were mounted onto thin glass fibers. The crystal volumes ranged from 15 to less than 1 μm^3. This very successful1 mounting technique for micrometer sized single crystals uses a scanning electron microscope to prepare single crystals for diffraction experiments that could otherwise not be handled due to their exceptionally small size.

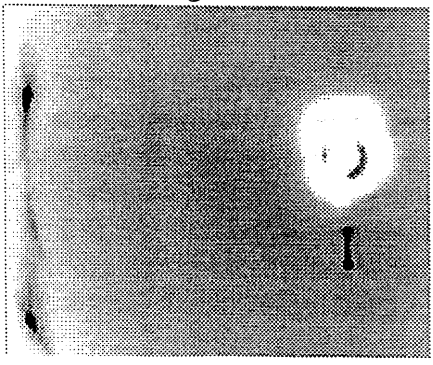
The data collections were carried out at the microfocus beamline ID1 3. This beamline is the only beamline suited for diffraction experiments on crystals of such small sizes. The diffraction pattern were recorded by the oscillation technique. Short ω scans of 3° were collected over a whole 360° range. The exposure times were set to 30 and 400 s

their intercalation was possible.

The data collections were carried out at the microfocus beamline ID 13. This beamline is the only beamline suited for diffraction experiments on crystals of such small sizes.
Several samples were screened to find a completely intercalated sample of good crystal quality. These checks revealed that all samples did not intercalate completely and that they decayed rapidly within some 10 minutes in the primary beam. Subsequent attempts to preform experiments at liquid nitrogen temperature with a cryogenic gas stream failed due to ice build up. The experimental background has to be reduced to the utmost minimum in order to record diffraction pattern form micrometer sized crystals. This requires a maxmimum free path of the X-ray through air of 7mm. For a successful1 repetition of the experiments we are currently investigating in collaboration with the beam line staff the options of microfocussing of a cryogenic gas stream.

Subsequent experiments on powders in the laboratory revealed that the kaolinite:DMSO intercalate decomposes already at temperatures as low as 70°, while previous reports suggested temperatures above 120°.

The X-ray induced thermal decomposition was followed during the current experiment by taking repeated exposures around the 001 reflection. We could show that at these low temperatures the diffusion of the DMSO is not a cooperative effect as it is at higher temperature. The final structure after thermal decpomposition above 160° resumes the original crystal quality. A lower temperatures still decomposes but a much more intensive diffuse scattering is observed in our single crystal diffraction pattern. Our preliminary interpretation is that many small islands of intercalated material remain in a matrix of collapsed kaolinite. This causes severe elastic deformation of the silicate sheet and in turn diffuse scattering.



The pattern shows the two maxima of the 001_{DMSO} and 001 with a broad abnd of diffuse scattering connecting them. The reflections to the left reveal diffuse scattering due to stacking faults as well.