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This work is part of the ORION-project (Ordering of Ions in Minerals) within the EuroMinSci programme of the ESF which aims at the elucidation of ionic ordering by combining experimental investigations and theoretical calculations. Previous solid state NMR spectroscopic investigations of phlogopite, a trioctahedral 2:1 layer silicate, indicated a non-statistical distribution of cations and anions in the octahedral sheets: F prefers sites coordinated by three Mg, whereas OH prefers sites with Al as next-nearest-neighbours [1]. Further investigations were carried out on phlogopites with various Al-contents and synthesized at different temperatures. Cross-polarization (CP) $\{^1\text{H}\}$ ^{29}Si CPMAS NMR experiments were performed to clarify whether the degree of ordering in the octahedral sheets is related to that in the tetrahedral layers. These experiments clearly indicate a direct neighbourhood of aluminium in the tetrahedral sheets to aluminium in the octahedral sheets and, thus, a relationship between the ordering of ions in both sheets. Support for these conclusions comes also from atomistic simulations of ordering using the so-called "J formalism" in which total-energy calculations with interatomic potentials are used to generate a set of pair interaction parameters which are then employed in Monte Carlo (MC) simulations [2,3]. In phlogopite we have considered the OH-rich extreme and performed MC simulations for several overall concentrations of Al in the range $0 < x < 1$, finding significant segregation of the Al atoms with a strong spatial correlation between the Al-rich domains in the two layers.

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X-Ray Powder Diffraction of Synthetic Al-rich Phlogopites: Lattice Parameters, Polytypes, Stacking Faults, and Implication of Modulations.

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X-ray powder diffraction experiments of synthetic Al-rich phlogopites synthesized at 800 °C and 2 kbar were carried out. The grain size of the synthetic phlogopites ranges between particle diameters of 3 to 5 µm. The samples

have already been well characterized by solid state NMR spectroscopy, and thus, possible impurity phases have been assigned. As for all micas, the phlogopite structure can be described in terms of the OD-theory, where 2-d periodic layers can be stacked in different ways without changing the transition from one layer to the adjacent one [1]. One aim of the experiments was to study the change of lattice parameters with increasing F- and Al-content. Previous investigations have already shown that the c-lattice parameter decreases strongly with increasing F-content [2]. From combined powder XRD and TEM investigations we expect to get detailed information on the polytypes which were formed during synthesis and on stacking faults in the mica structure. Up to now, many natural phlogopite single-crystals and synthetic powder samples have been characterized using XRD techniques. The most common polytype is the one-layer monoclinic polytype (1M) with space group symmetry C2/m. The other two possible polytypes, 2M₁ (space group symmetry C2/c) and 3T (space group symmetry P3₁12) are far less abundant [3]. Our first refinements indicate that all phlogopite samples consist of polytype 2M₁. However, there may also be a mixture of polytype 2M₁ and polytype 1M, leading to inelastic X-ray scattering. In some powder patterns with better resolution, satellite reflections surrounding hkl-reflections occur, which have not yet been reported and imply the presence of a modulated structure. While solid-state NMR spectroscopic investigations offer an insight into the local structure of the nuclei, the powder XRD patterns refinements may give new information about the long-range order of the tetrahedral and octahedral sheets. Combined with the TEM investigations, the results will contribute to a better understanding of stacking faults in the mica structure. Furthermore, this information may clarify the presence and type of modulations in the structure.

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The structure and lattice parameters of the tantalum and tantalum oxides phases were determined by temperature X-ray diffraction under vacuum and low pressure carbone monoxide between 293 and 2278K. We show that the metallique phase structure is stabilized by inclusion of oxygen into the metal octahedral hole. We determined the crystalline characteristics at high temperatures for Ta and Ta₂O₅ phases.