

four Bailey's groups (or OD subfamilies) termed A, B, C, and D, according to shifts and rotations of consecutive layers:  $+a_i/3$  shifts for group A,  $+a_i/3$  shifts and 180 degrees rotation for group B,  $+b/3$  or zero shift for group C,  $+b/3$  or zero shifts and 180 deg. rotation for group D, where  $a_i$  and  $b$  correspond to the edges of hexagonal and orthohexagonal cells, respectively. The ordered polytypes are represented by regular sequences of shifts and/or rotations allowed by the stacking rule of the respective subfamily. More or less disordered crystals are common. Recently, crystal structures of polytypes of cronstedtite 3T, 1T, and 2H2, representing groups A, C, and D, respectively, were refined. No polytype in the group B has been found to date. Subfamilies and polytypes can be identified by following methods: (1) Single-crystal X-ray diffraction (XRD) (2) Selected area electron diffraction (SAED) (3) Electron back scattering diffraction (EBSD) (4) High resolution electron transmission microscopy (HRTEM) Refined structures, X-ray diffraction pattern, HRTEM, and SAED images of several polytypes are presented.

Keywords: layered silicates, HRTEM, single-crystal X-ray diffraction

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#### New NaAlSiO<sub>4</sub> polymorphs: Monoclinic and orthorhombic trinepheline

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Recently, a new structural variety of nepheline with composition Na<sub>7.85</sub>Al<sub>7.85</sub>Si<sub>8.15</sub>O<sub>32</sub> was obtained by single crystal growth experiments using a cryolite flux. It crystallizes in space group *P*112<sub>1</sub>,  $a = 9.9897(6)$  Å,  $b = 9.9622(6)$  Å,  $c = 24.979(2)$  Å,  $\gamma = 119.788(4)^\circ$ ,  $Z = 3$ . The crystal structure of monoclinic trinepheline can be explained as a stacking sequence of a conventional nepheline unit cell and one additional layer built exclusively of elliptical rings, as observed in the structure of hexagonal trinepheline. When heated to 373(5) K, monoclinic trinepheline transformed to trinepheline of hexagonal symmetry (space group *P*6<sub>1</sub>). Upon further heating at 473(5) K the crystal structure corresponds to that of conventional nepheline. Furthermore, we have determined the structure of an orthorhombic polymorph of NaAlSiO<sub>4</sub> (*P*na2<sub>1</sub>,  $Z = 36$ ), which was hydrothermally synthesized and described by Klaska in 1974 (Thesis, University of Hamburg). The lattice parameters are related to the *P*6<sub>3</sub> ones through the relationship  $a(\text{ortho}) \approx \sqrt{3}/2 * a(\text{hex}) \approx 8.66$  Å;  $b(\text{ortho}) \approx 1.5 * a(\text{hex}) \approx 14.94$  Å;  $c(\text{ortho}) \approx c(\text{hex}) \approx 25.14$  Å. The crystals were affected by twinning via the sixfold axis. This polymorph is built of the elliptical rings and the resulting layers are comparable to the ones in hexagonal trinepheline. In hexagonal trinepheline, however, the neighbouring layers are rotated with respect to each other. In the orthorhombic modification, the layers are stacked on top of each other in identical orientations. We will also present some results of an *ab-initio* study on Na nepheline (known for showing Na-solid state conductivity) performed within the DFT formalism as implemented in the SIESTA code.

Keywords: single-crystal growth, single-crystal structure analysis, *ab-initio* calculations

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#### Hydrogen positions in beryllate minerals and materials by combined X-ray and neutron diffraction

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For mineral systems information on their structure, including the localisation of light atoms such as hydrogen and beryllium, is of considerable importance as it allows a better understanding of the material behaviour under natural conditions including its paragenesis, phase stability, compressibility/water content and thermal expansion. Many important natural minerals and their analogues are not simple silicates or aluminosilicates, but also incorporate other framework forming species such as the beryllate tetrahedron, BeO<sub>4</sub>. The role of the hydrogen containing species such as H<sub>2</sub>O and OH is also central to determining the structure formed and distribution of non-framework species in the material. We have found that the optimised crystallographic method of determining the full crystal structures of these minerals, including accurate hydrogen positions and framework distributions of Si, Al and Be, is a combined single crystal X-ray - powder neutron diffraction method. Powder neutron diffraction data are collected on the hydrogenous natural material at 120 K and the data analysis simultaneously fits this and the SXD (single-crystal X-ray) data collected at the same temperature. In the presented study the following minerals have been investigated and detailed structures will be presented: 1) Eudidymite and Epididymite. Differences in these compositionally identical minerals, NaHBeSi<sub>3</sub>O<sub>8</sub>, have been determined and results from the orientation of an extra framework hydroxide anion. 2) Leifite Na<sub>2</sub>(Si,Al,Be)<sub>7</sub>(O,OH,F)<sub>14</sub>. 3) Sorensenite Na<sub>4</sub>SnBe<sub>2</sub>Si<sub>6</sub>O<sub>18</sub> 2 H<sub>2</sub>O. 4) Nabesite Na<sub>2</sub>BeSi<sub>4</sub>O<sub>10</sub> 4 H<sub>2</sub>O. 5) Semenovite (Na,Ca)<sub>9</sub>(Ce,La)<sub>2</sub>(Fe<sup>2+</sup>,Mn)(Si,Be)<sub>20</sub>(O,OH,F)<sub>48</sub>.

Keywords: combined X-ray - neutron diffraction, beryllates, hydrogen positions

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#### Structural features of the M-site vacancies and possible hydrogen positions in hydrous forsterite

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The result of the structural refinement [1] of synthetic Fe-bearing hydrous forsterite synthesized at 13.5 GPa and 1400°C [2] revealed that the cation vacancies at the octahedral M sites predominantly occur at the M2 site, in contrast to the case [3][4] of Fe-free hydrous forsterite synthesized at 13.5 GPa and 1300°C [5] revealed that the cation vacancies at the octahedral M sites predominantly occur at the M1 site. In the case of Fe-free hydrous forsterite, the structural strain from the ideal size is larger in M1 site than in M2 site. Two H atoms may replace the Mg atom predominantly at the M1 site to reduce the structural strain, giving the vacancy at the M1 site. In the case of Fe-bearing hydrous forsterite, the Fe atoms occupy both M1 and M2 sites, leading the combinations of Mg-Mg, Fe-Fe, Mg-Fe and Fe-Mg for the occupation of M1-M2 sites. Among these, the combination of Fe-Mg for M1-M2 sites may have maximum structural strain from