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Structural variety in the high-pressure synthetic sheet-disilicate Phase-X, $\mathbf{K_{(2-x)}Mg_2Si_2O_7H_x}$

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The structure of the synthetic high-pressure sheet-disilicate Phase-X (PhX), a possible host of H_2O in the mantle, has been determined for a crystal synthesized at 16 GPa/1300 ˚C/23 hr. The composition of the sample obtained by electron microprobe is K1.61(8) Mg2.10(3) Si2.05(1) O7. The structures of four crystals of PhX were determined by single-crystal X-ray diffraction (XcaliburE) and had very similar diffraction characteristics and structural results; the structure of one the larger crystals is reported here. Reflection intensity statistics strongly indicate that PhX is centrosymmetric with space group $P6_3/mcm$. While it was possible to obtain low agreement indices for refinements in $P6_3cm$, there were strong correlations between atoms that are equivalent in $P6_3/mcm$, suggesting that the correct structure is centrosymmetric. Full anisotropic refinement in spacegroup $P6_3/mcm$ gave R1 = 0.036, wR2 = 0.079, GoF = 1.467. As with all previous studies of PhX, the H atom was not located. The K atom is displaced from the six-fold axis onto a site 0.3 Å away. Refinement of the K content of this site gave a value of 0.75(1), corresponding to 1.5 K apfu, in excellent agreement with the content derived from electron microprobe analysis. The PhX crystals studied here have compositions close to PhX50/Anhydrous-PhX50, which appears to be a recurring composition in several studies. Diffraction patterns of all four crystals, reconstructed from the full intensity data collection, consistently show the presence of a large hexagonal superstructure with dimensions $8a$(sub) $\times$ $8a$(sub) $\times$ c(sub), having Z = 128, compared with Z = 2 for the subcell: a= 40.378(2) Å, c= 13.1966(6) Å, V = 18633(1) Å^3. Complex arrays of superlattice reflexions occur in layers with l= $2n$, but are absent from l = $2n$ + 1 layers. We attempted to determine this superstructure, but the weakness of many superlattice reflections has not allowed a model to be obtained so far. It is speculated that this large supercell arises from the ordering of K atoms and vacancies within the interlayer, and that the centrosymmetric structure is characteristic of ordered intermediate compositions at or near PhX50/Anhydrous-PhX50. Identification of a new space group and recognition of a previously unobserved superstructure point to new possibilities for PhX and its derivatives that may bear significantly upon their stability at mantle conditions. An updated synopsis of the variety of the family of Phase-X structures, including their polytypism, will be presented.