

a transition at 493 - 497 K from an ordered, antiferroelectric to a disordered paraelectric phase with a monoclinic structure, spacegroup $A/2a$ [1]. This is accompanied by a pronounced change in the shape and intensity of diffuse scattering and has been modeled with correlated shifts of Ti, Ca and O [1,2]. Previous characterization of this diffuse scattering probed a very limited portion in reciprocal space using synchrotron X-rays [1]. Here, we report preliminary results of a comprehensive neutron single crystal study done at room temperature. A 3D volume in reciprocal space has been measured using the SXD neutron diffractometer at ISIS and several sections have been extracted. Diffuse scattering is characterized by broad bands out to 20\AA^{-1} . We use a Monte Carlo simulation approach to characterise this with the average structure as input in order to provide a starting point for a characterization of the diffuse scattering above the phase transition.

[1] Malcherek T., Paulmann C., Chiara Domeneghetti M., Bismayer U. *J. Appl. Cryst.* 34, 108, 2001. [2] Ghose S., Ito Y., Hatch D.M., *Phys. Chem. Miner.* 17, 591, 1991.

Keywords: neutron diffuse scattering; single crystal diffraction; minerals

FA2-MS01-P18

Synthesis and Crystal Structure of SrFe[BP₂O₈(OH)₂] and CaCo(H₂O)[BP₂O₈(OH)] · H₂O. Prashanth Wilfred Menezes^a, Stefan Hoffmann^a, Yurii Prots^a, Rüdiger Kniep^a. ^aMax-Planck-Institut für Chemische Physik fester Stoffe, Nöthnitzer Str. 40, 01187 Dresden, Germany.

E-mail: menezes@cpfs.mpg.de

Single crystals of SrFe[BP₂O₈(OH)₂] and CaCo(H₂O)[BP₂O₈(OH)]·H₂O were synthesized under mild hydrothermal conditions and investigated by single crystal X-ray diffraction [1-2]. The crystal structure of SrFe[BP₂O₈(OH)₂] (triclinic, space group $P\bar{1}$ (No. 2), $a = 6.6704(12)\text{ \AA}$, $b = 6.6927(13)\text{ \AA}$, $c = 9.3891(19)\text{ \AA}$, $\alpha = 109.829(5)^\circ$, $\beta = 102.068(6)^\circ$, $\gamma = 103.151(3)^\circ$, $V = 364.74(12)\text{ \AA}^3$, $Z = 2$) contains isolated borophosphate oligomers, [BP₂O₈(OH)₂]⁵⁻ (unbranched tetrahedral triples, Figure 1(a)), which are interconnected by Fe^{III}O₄(OH)₂ coordination octahedra. The resulting framework is characterized by elliptical channels running along [011]. Strontium takes positions inside the channels. SrFe[BP₂O₈(OH)₂] represents the first example in the structural chemistry of borophosphates where the charge of the anionic partial structure is balanced by a divalent and a trivalent cation ($M^{\text{II}}M^{\text{III}}$). The crystal structure of CaCo(H₂O)[BP₂O₈(OH)]·H₂O (triclinic, space group $P\bar{1}$ (No. 2), $a = 6.5793(3)\text{ \AA}$, $b = 7.8320(1)\text{ \AA}$, $c = 8.8172(1)\text{ \AA}$, $\alpha = 68.785(7)^\circ$, $\beta = 82.719(10)^\circ$, $\gamma = 73.985(9)^\circ$, $V = 406.930(19)\text{ \AA}^3$, $Z = 2$) contains layers stacked along [011]. The layers are built from isolated borophosphate oligomers, [B₂P₄O₁₆(OH)₂]⁸⁻ (loop branched tetrahedral hexamers, Figure 1(b)), which are interconnected by dimers of CoO₃O_{2/2}(H₂O) coordination octahedra. Calcium ions and crystal water are located at the borders of the layers, close to intra-layer cavities. CaCo(H₂O)[BP₂O₈(OH)]·H₂O is the

first example in borophosphate crystal chemistry where dimers of metal octahedra together with borophosphate oligomers form a layered arrangement.

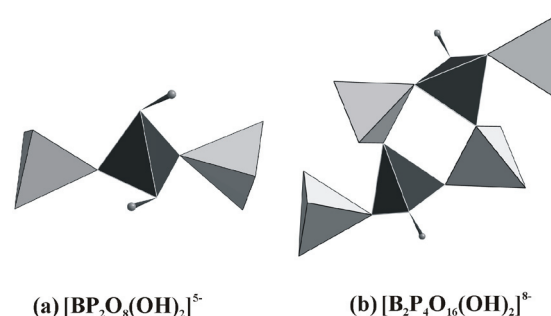


Figure 1. Anionic partial structure of (a) SrFe[BP₂O₈(OH)₂] and (b) CaCo(H₂O)[BP₂O₈(OH)]·H₂O (grey tetrahedra = BO₄; light grey tetrahedra = PO₄; grey spheres = protons).

[1] Menezes P.W., Hoffmann S., Prots Yu., Schnelle W., Kniep R., Z. *Anorg. Allg. Chem.*, 2009 (in press). [2] Menezes P.W., Hoffmann S., Prots Yu., Kniep R., Z. *Anorg. Allg. Chem.*, 2009 (in press).

Keywords: borophosphates; hydrothermal growth; single crystals

FA2-MS01-P19

Solid Solution Series in Transition Metal Borophosphates. Falk Gruchow^a, Stefan Hoffmann^a, Rüdiger Kniep^a. ^aMax-Planck-Institut für Chemische Physik fester Stoffe, Nöthnitzer Str. 40, 01187 Dresden, Germany.

E-Mail: gruchow@cpfs.mpg.de

A series of transition metal borophosphates with the general formula Mn_xCo_{1-x}[BPO₄(OH)₂] ($x = 0, 0.1, 0.2, \dots, 1$) was prepared under mild hydrothermal conditions. The reaction products were obtained in high yields as single phase materials according to powder X-ray diffraction data. The compounds $M^{\text{II}}[\text{BPO}_4(\text{OH})_2]$ ($M^{\text{II}} = \text{Co}, \text{Mn}$) [1] are isotopic and crystallize in the chiral space groups $P3_121$ (No. 152) or $P3_221$ (No. 154), respectively. Their crystal structures are characterized by edge-sharing helical $M^{\text{II}}\text{O}_6$ -octahedral chains wound around 3_1 or 3_2 screw axes (running along [001]). The chains are interconnected by borophosphate single chains of alternating BO₂(OH)₂ and PO₄ tetrahedra running perpendicular to [001], resulting in a three-dimensional framework structure. As a first example in the interesting group of chiral borophosphates a complete miscibility of Co- and Mn-based phases has been proven experimentally. A linear correlation between the nominal manganese content x and the lattice parameters (or the cell volume V) was found. Further studies regarding morphological aspects, magnetic properties and possible ordering effects of the metal cations are in progress.