

**MS13-2-20 Thermodiffraction study of $(\text{NH}_4)_{0.5}\text{Co}_{1.25}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot(\text{H}_2\text{O})_{0.5}$ with CZP framework topology
#MS13-2-20**

W. Mogodi ¹, D. Billing ², M. Fernandes ², A. Fitch ³

¹Department of Chemistry, University of Cape Town - Cape Town (South Africa), ²School of Chemistry, University of the Witwatersrand - Johannesburg (South Africa), ³European Synchrotron Radiation Facility (ESRF) - Grenoble (France)

Abstract

Four CZP (chiral zincophosphate) zeolite topology compounds with the general formula $M^I M^{II}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot y\text{H}_2\text{O}$ ($M^I = \text{Na}, \text{NH}_4$ and $M^{II} = \text{Mn}, \text{Co}$, $y = 0.5, 1$) have been prepared under mild hydrothermal conditions (at 180 °C). Such microporous compounds with aesthetically interesting crystal structures [1] can have interests in fields such as catalysis, storage, separation and ion-exchange. One compound of this family, $(\text{NH}_4)_{0.5}\text{Co}_{1.25}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot(\text{H}_2\text{O})_{0.5}$, has been studied by variable temperature high resolution powder X-ray diffraction experiments carried out from 298 to 1073 K. Complete Rietveld refinements were achieved by combining stereochemical restraints of the powder diffraction data. At room temperature, this compound crystallizes in the $P6_5$ (No. 170) space group with $Z = 4$ belonging to the hexagonal system. The unit cell parameters obtained were: $a = 9.4330(2) \text{ \AA}$, $c = 15.5203(2) \text{ \AA}$, $V = 1196.01(5) \text{ \AA}^3$. The crystal structure consists of a helical anionic framework, $\infty[\text{BP}_2\text{O}_8]^{3-}$, composed of corner sharing BO_4 and PO_4 tetrahedra. Water and ammonia molecules are found within the helical channels running along the $[001]$ direction. This compound undergoes a series of dehydration, de-ammoniation (analysis augmented by thermogravimetric experiments) and finally diminished sharp Bragg peaks indicating loss of long-range order. Total scattering analysis [2] was applied for the first time coupled to the above conventional structural refinement approach to further unravel this gaseous dissociation and temperature induced amorphization of the rigid host structure. Preliminary X-ray PDF indicates that the local coordination environment found in the low temperature crystalline phases also persists in the amorphous phase.

References

- [1] Kniep, R., Will, H.G., Boy, I. and Röhr, C. *Angewandte Chemie International Edition*, 1997, 36, 1013.
[2] Egami, T. and Billinge, S.J. Elsevier, 2003.

Diffraction behaviour of helical borophosphates

