ESRF	**Experiment title:** Structural characterization of P-, Si- and Ge-based solid state ionic conductors	**Experiment number**: 31-01-19
Beamline: BM21	**Date of experiment**: from: 16.03.2017 to: 22.03.2017	**Date of report**: 22.08.2017
Shifts: 9	**Local contact(s)**: Wouter Van Beek	*Received at ESRF:*

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Report:

The study focused on identification and structural characterization of new hydrogen-rich solid state ionic conductors prepared by mechanical alloying.

A series of compounds with a nominal composition $K_xBa_{3-x}H(D)_x(PO_4)_2$, obtained by mechanical milling in various atmospheres (argon, hydrogen/deuterium and air), was measured with a high-resolution synchrotron powder X-ray diffraction (SR-PXD) instrument at room temperature. During experiment powders were sealed in a boron-glass capillary (d = 0.3 mm).

Collected powder diffraction patterns showed formation of a trigonal $K_xBa_{3-x}H_x(PO_4)_2$ phase in all as-milled samples. Unfortunately, all powders also contained secondary phases, which abundance varied depending on the applied milling atmosphere. The lowest concentration of impurities was observed in the powder processed in argon.

All samples from the series were also calcinated at 1000 °C in air and measured by high-resolution SR-PXD. Obtained powder diffraction patterns showed phase transition of trigonal to orthorhombic $K_xBa_{3-x}H_x(PO_4)_2$ in all investigated powders (Figure 1 & 2). In most of them no residual phases were detected.

In order to evaluate presence of hydrogen atoms in the crystal structures of the formed phases selected samples, containing deuterium instead of hydrogen, were also measured by powder neutron diffraction (PND) and joint analysis of SR-PXD and PND data is in progress.

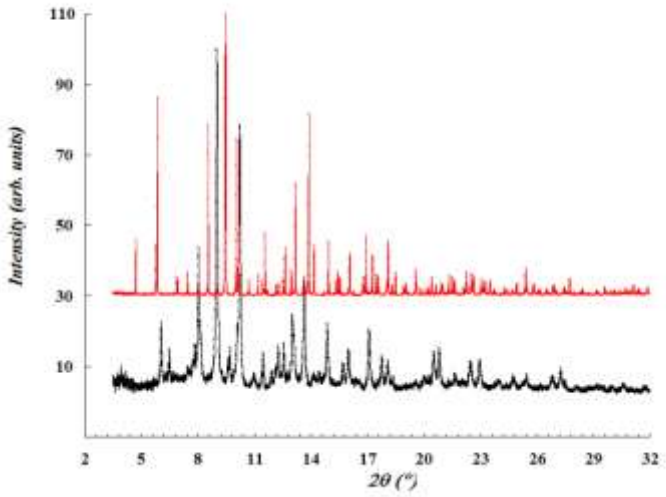

Figure 1. High resolution diffraction profiles (λ = 0.50218 Å) obtained for as-milled and calcinated $K_xBa_{3-x}H_x(PO_4)_2$.

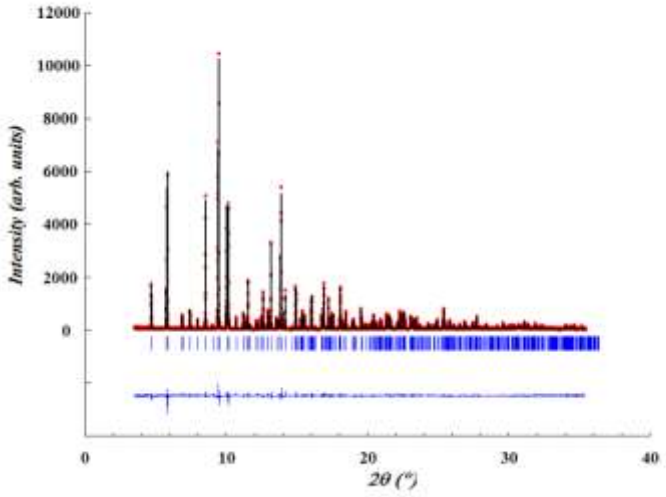

Figure 2. Observed (red), calculated (black) and difference (blue) high resolution diffraction profiles (λ = 0.50218 Å) obtained for calcinated $K_xBa_{3-x}H_x(PO_4)_2$ (space group: *Pnma*). Vertical bars indicate Bragg peaks positions of the crystalline $K_xBa_{3-x}H_x(PO_4)_2$ phases.