

**P09.03.23***Acta Cryst.* (2008). A64, C481**Crystal phase analysis of by-products from NaBH<sub>4</sub> production via high-low pressure process by XRD**

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Sodium borohydride (NaBH<sub>4</sub>) is a safe and practical way of storing hydrogen due to its high hydrogen capacity (10.6% wt). NaBH<sub>4</sub> is synthesized from different boron minerals by the thermal-chemical reactions. Constitution of by-product is little demand in NaBH<sub>4</sub> production process, because of requirements of its reuse or disposal issues. It is important to know the mineralogical properties of by-product for its disposal or reusing. In our study we investigated of characterization of by-products which were obtained from NaBH<sub>4</sub> production is based on the conversion reaction of borosilicate glass under high and low hydrogen pressure process (HPP-LPP) by XRD analysis. The XRD analysis was carried out at an ambient temperature by using Philips Panalytical X'Pert-Pro diffractometer in a range of diffraction angle from 10° to 90° with CuK $\alpha$  radiation  $\lambda=0.15418$  nm at operating parameters of 40 mA and 45 kV with step size 0.02° and speed of 1° /min. According to the X-ray powder diffraction data, four crystal phases, Na<sub>2</sub>SiO<sub>3</sub> (PDF Number: 00-016-0818), Na<sub>2</sub>SiO<sub>3</sub>.5H<sub>2</sub>O (PDF Number: 00-003-0433), Na<sub>2</sub>SiO<sub>3</sub>.6H<sub>2</sub>O (PDF Number: 00-018-1246) and Na<sub>8</sub>Si(Si<sub>6</sub>O<sub>18</sub>) (PDF Number: 01-088-1229) were defined for each by-products HPP<sub>BP</sub> and LPP<sub>BP</sub> obtained via HPP and LPP, respectively. By comparison XRD results of by-products were obtained under high and low hydrogen pressure processes, there are same crystallographic properties have been detected.

Keywords: X-ray diffractometers, crystal phases, inorganic compounds

**P09.03.24***Acta Cryst.* (2008). A64, C481**Acid phosphates of 1-(1-naphthyl)ethylamine - Importance of symmetry relation between enantiomers**Ondrej Kaman<sup>1,2</sup>, David Havlicek<sup>1</sup>, Jaroslav Cihelka<sup>3</sup><sup>1</sup>Faculty of Science, Charles University in Prague, Department of Inorganic Chemistry, Hlavova 8, Praha 2, 128 43, Czech Republic,<sup>2</sup>Institute of Physics of the ASCR, Cukrovarnicka 10, 162 00 Praha 6, Czech Republic, <sup>3</sup>J. Heyrovsky Institute of Physical Chemistry of the ASCR, Dolejskova 2155/3, 182 23 Praha 8, Czech Republic, E-mail : kaman@fzu.cz

The molecule of 1-(1-naphthyl)ethylamine (NEA) with asymmetric carbon linked to large aryl group is used as chiral agent in NMR studies, as a chiral selector in separations and for derivatization in enantioselective catalysis. It is able to form a wide variety of acid phosphates comprising interesting networks of hydrogen bonds. These compounds arise from water - alcohol solutions of NEA and H<sub>3</sub>PO<sub>4</sub> and are affected by symmetry relations between enantiomers present in the racemate. Salts prepared only from one enantiomer exhibit different and unique structures. We have prepared two different forms of ( $\pm$ )-(NEAH)H<sub>2</sub>PO<sub>4</sub> with distinct networks of hydrogen bonds. The triclinic dihydrogenphosphate crystallized from mixture with stoichiometric excess of the acid whilst monoclinic form was discovered by crystallization with stoichiometric excess of the base. The triclinic form is described by *P*-1 and its phosphate anions

form chains of hydrogen bonds. The monoclinic structure belongs to *P*<sub>2</sub><sub>1</sub>/*c* and phosphate units are interconnected to two-dimensional network. Another compound (NEAH)<sub>2</sub>HPO<sub>4</sub> . 2H<sub>2</sub>O described by *C*2/*c* comprises complicated network of hydrogen bonds. The salts of optically pure (*S*)-NEA cannot crystallize in space groups involving reflexion or inversion operations of symmetry. The (*S*)-(NEAH)<sub>3</sub>H<sub>2</sub>PO<sub>4</sub>HPO<sub>4</sub> . 4.5 H<sub>2</sub>O is an interesting example of such structure and belongs to *C*2. Very short hydrogen bond between HPO<sub>4</sub><sup>2-</sup> and H<sub>2</sub>PO<sub>4</sub> represents remarkable feature in comparison to structures described above. Sets of crystalline phases obtained from solutions with different ratio of acid and base (separately for racemic and optically pure) were studied using XRD and some structures were determined from single crystal data.

Keywords: acid phosphates, short hydrogen bonds, chirality

**P09.03.25***Acta Cryst.* (2008). A64, C481**A comparative study of two multiphasic alkali halide crystals: Quinary vs. exenary**Ricardo Rodriguez-Mijangos<sup>1</sup>, Cordero-Borboa Adolfo<sup>2</sup><sup>1</sup>Universidad de Sonora, Departamento de Investigacion en Fisica, P O Box 5-88, Hermosillo, Sonora, 83190, Mexico, <sup>2</sup>Instituto de Fisica, Universidad Nacional Autonoma de Mexico, P O Box 20-364, Mexico D F 01000, Mexico, E-mail: mijangos@cajeme.cifus.uson.mx

A comparative study of two mixed crystals grown by a Czochralsky technique is realized: a quinary and a exenary. The first one is made by a melt of five salts: KBr, RbCl, RbBr, KI and RbI, mixed in equal molar composition and the second one made with the same salts, but adding KCl, being the six salts in equal molar composition. The X ray diffractometry technique, determine for the quinary three phases with NaCl structure type: a single, a binary and a ternary, each one with a lattice constant well defined, in the exenary determine two phases, also with NaCl type: a binary and a quaternary, each one with a lattice constant well defined. By using equations of mass balance is obtained for each phase the concentrations of the components in molar fraction in the quinary and exenary crystals. Is remarkable the KCl play role simplifying the phase number from three to two when increase the components number from five to six. A qualitative analysis is done explain this phenomena.

Keywords: inorganic materials, growth crystal, composition and structure of materials and alloys

**P09.03.26***Acta Cryst.* (2008). A64, C481-482**From dimeric tantalopentatungstate to monomeric organosilyl Lindqvist type polyoxometalates**Mongi Debbabi<sup>1</sup>, Fatma Bannani<sup>1,2</sup>, Rene Thouvenot<sup>2</sup><sup>1</sup>University of Monastir, ENIM, Monastir, Monastir, 5019, Tunisia,<sup>2</sup>CIM2, case courrier 42 University Pierre et Marie Curie, 4 place Jussieu, Paris cedex 05, E-mail: dmongi2@yahoo.it

Polyoxometalates (POMs) are a well-known class of inorganic metal-oxygen clusters with an unmatched structural variety combined with a multitude of properties. The search for novel POMs is predominantly driven by exciting catalytic, medicinal, material science and bioscience applications. However, the mechanism of action of most polyoxoanions is not selective towards a specific target. In order to improve selectivity it appears highly