

P09.03.23*Acta Cryst.* (2008). A64, C481**Crystal phase analysis of by-products from NaBH₄ production via high-low pressure process by XRD**

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Sodium borohydride (NaBH₄) is a safe and practical way of storing hydrogen due to its high hydrogen capacity (10.6% wt). NaBH₄ is synthesized from different boron minerals by the thermal-chemical reactions. Constitution of by-product is little demand in NaBH₄ 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 NaBH₄ 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 α 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, Na₂SiO₃ (PDF Number: 00-016-0818), Na₂SiO₃.5H₂O (PDF Number: 00-003-0433), Na₂SiO₃.6H₂O (PDF Number: 00-018-1246) and Na₈Si(Si₆O₁₈) (PDF Number: 01-088-1229) were defined for each by-products HPP_{BP} and LPP_{BP} 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^{1,2}, David Havlicek¹, Jaroslav Cihelka³¹Faculty of Science, Charles University in Prague, Department of Inorganic Chemistry, Hlavova 8, Praha 2, 128 43, Czech Republic,²Institute of Physics of the ASCR, Cukrovarnicka 10, 162 00 Praha 6, Czech Republic, ³J. 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 H₃PO₄ 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)H₂PO₄ 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*₂₁/*c* and phosphate units are interconnected to two-dimensional network. Another compound (NEAH)₂HPO₄ . 2H₂O 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)₃H₂PO₄HPO₄ . 4.5 H₂O is an interesting example of such structure and belongs to *C*2. Very short hydrogen bond between HPO₄²⁻ and H₂PO₄ 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-Mijangos¹, Cordero-Borboa Adolfo²¹Universidad de Sonora, Departamento de Investigacion en Fisica, P O Box 5-88, Hermosillo, Sonora, 83190, Mexico, ²Instituto 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 Debbabi¹, Fatma Bannani^{1,2}, Rene Thouvenot²¹University of Monastir, ENIM, Monastir, Monastir, 5019, Tunisia,²CIM2, 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

desirable to attach organic functionalities covalently to the surface of polyoxoanions. As part of a broad program centered on the functionalization of polyoxometalates, we have been interested in the derivatisation of Lindqvist type polyoxoanions with organosilyl moieties. The condensed polyoxometalate $(n\text{Bu}_4\text{N})_4[(\text{TaW}_5\text{O}_{18})_2\text{O}]$ which is synthesized by reacting $[\text{TaW}_5\text{O}_{19}]^{3-}$ with BuSnCl_3 , crystallises in the orthorhombic system, space group $\text{Pbn}\bar{b}$ with lattice parameters $a = 15.7981(14)$, $b = 17.939(3)$, $c = 35.216(6)\text{\AA}$, $V = 9980\text{\AA}^3$ and $Z = 4$. The crystallographic study of $(n\text{Bu}_4\text{N})_4[(\text{TaW}_5\text{O}_{18})_2\text{O}]$ shows that the dimer is composed from two polyoxoanions fragments linked by linear Ta-O-Ta bridge. Such a linkage readily reacts with organosilyl (Lewis electrophilic reagents), such as $\text{RR}'_2\text{SiOH}$ ($\text{R} = \text{R}' = \text{Et}$, $i\text{Pr}$, OtBu , Ph ; $\text{R} = \text{tBu}$, $\text{R}' = \text{Me}$) to yield monomeric plenary Lindqvist derivatives $(n\text{Bu}_4\text{N})_2[\text{W}_5\text{O}_{18}\text{Ta}(\text{O})\text{SiR}'\text{R}_2]$. These derivatives are characterized in the solid state by IR and in solution by multinuclear NMR (^{13}C , ^{29}Si , ^{183}W). The crystallographic study of $(n\text{Bu}_4\text{N})_2[(\text{W}_5\text{O}_{18}\text{Ta}(\text{O})\text{SiPh}_3)]$ indicates that $\{\text{SiPh}_3\}^+$ is grafted on the surface of the polyanion through the terminal O-Ta oxygen atom.

Keywords: polyoxometalates, X-ray structure, NMR spectroscopy

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Octamolybdates - promising materials for industry and medicine

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Molybdates are interesting and perspective candidates for various applications in electronics and medicine (cancer therapy) [1], catalysis and environment protection. Polymolybdates are a numerous group of compounds and despite of enormous work done so far, synthesis of selected types of polymolybdates is still a challenging and demanding task, requiring experience, intuition and vast experimental work. Recently a group of 5 new beta-octamolybdates was obtained in our lab. We solved the crystal structures and investigated selected properties of: 1/ Ammonium tris(triethylammonium) octamolybdate, ($a, b, c, \alpha, \beta, \gamma$, SG) 25.230(5), 10.859(2), 19.033(3), 121.92(1), $C 2/c$ 2/ Tetrakis(trimethylammonium) octamolybdate dihydrate; 10.433(3), 10.486(2), 10.708(4), 102.40(2), 101.78(3), 118.35(3), $P-1$ 3/ Tetrakis(naphthalen-1-aminium) octamolybdate tetra(1-naphthylamine); 15.561(5), 18.969(8), 7.54(3), 100.80(3), 103.04(3), 73.42(3), $P-1$ 4/ Bis(1-amino-1-phenyleneammonium) bis(2-methylbenzimidazolium) octamolybdate; 8.541(5), 10.293(5), 13.018(5), 80.03(5), 83.74(5), 75.12(5), $P-1$ 5/ Tetrakis(2,6-dimethylanilinium) octamolybdate: 11.878(3), 10.533(3), 11.586(2), 101.12(2), 120.26(1), 75.46(2), $P-1$ Compounds 1 - 2 were obtained in hydrothermal conditions while 3-5 from hot mixtures of H_2MoO_4 , amine and H_2O . Most of these compounds crystallise in SG $P-1$, in compound 3 protonated and neutral amines are present, in 4 unexpected 2-methylbenzimidazolium cation was obtained. Based on the results of crystal structure determination of polymolybdates, some rules concerning the crystal engineering of isopolymolybdates, will be presented. Supported by ICDD and Polish MEiN grant 1T09A 07730

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Keywords: molybdenum VI oxycompounds, materials science and engineering, single-crystal X-ray analysis

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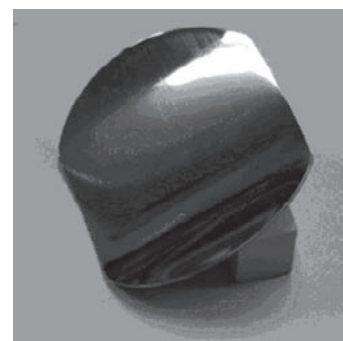
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Strongly and accurately shaped Ge crystal for non-scanning X-ray fluorescence spectrometer

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Plastic deformation technique of Si and Ge single crystals, which enables us to obtain the various forms, makes impact upon the field of X-ray spectroscopy, because Si and Ge are commonly used as analyzing crystals for x rays. Recently, we developed a deformation technique for obtaining strongly and accurately shaped Si or Ge wafers of high crystal quality, although covalently bonded Si or Ge crystals have long been believed to be not deformable to various shapes. The use of the deformed wafer made it possible to produce fine-focused x rays. In the present study, we prepared a cylindrical Ge wafer with a radius of curvature of 50 mm (Fig.), and acquired fluorescent x rays simultaneously from 4 elements by combining the cylindrical Ge wafer with a position-sensitive detector. The energy resolution of the x-ray fluorescence spectrum was as good as that obtained using a flat single crystal, and its gain was over 100. The demonstration of the simultaneous acquisition of high-resolution x-ray fluorescence spectra indicated various possibilities of x-ray spectrometry, such as one-shot x-ray spectroscopy and highly efficient wave-dispersive x-ray spectrometers.



Keywords: Ge wafer, X-ray spectrometer, plastic deformation

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Determination of thermal treatment effect of plating sludge by phase identification: XRD technique

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Thermal treatment was used to recover the precious metals or stabilize solid industrial wastes such as metal plating sludge which includes Zinc (Zn), Chromium (Cr), Copper (Cu), Nickel (Ni), iron etc. compounds. After thermal treatment of the samples, metal compounds were converted to metal oxides and then they were leached with suitable reagents to recover the precious metals. Moreover treatment temperature is very important in order to determine the optimum conditions. Therefore, metal plating sludge's composition changes due to operating process parameters. In addition to examined metal plating sludge's crystal structure which were shown differences when compare with the others. These