

investigation of the B_2O_3 -CuO- Li_2O ternary system". [3] N. A. Kuratieva, D. Mikhailova, H. Ehrenberg, "A new polymorph of $Cu_3B_2O_6$ ", *Acta Cryst. C* 65 (2009) 185-186. [4] Behm, H. (1982). *Acta Crystallogr. B* 38, 2781, "Pentadecacopper (II) Bisborate Hexaorthoborate Dioxide".

Keywords: Jahn-Teller distortion of Cu(II), layered structure, anisotropic thermal expansion

FA2-MS16-P42

Crystallographic Orientation Relationship between Bastnaesite, Fluocerite and Cerianite in a Single Crystal from the Pikes Peak Pegmatites. Mathis M. Müller, Hans-Joachim Kleebe. *GeoScience, Technical University Darmstadt, Germany.*

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A fluocerite (Ce,La) F_3 single crystal intergrown with bastnaesite (Ce,La)[CO $_3$]F from the Pikes Peak area in Colorado, USA was prepared for the investigation of the crystallographic orientation relationship between the two mineral phases via transmission electron microscopy. The epitaxial overgrowth of bastnaesite on fluocerite was confirmed by evaluating the electron diffraction patterns of the relevant mineral phases. During the examination, a third mineral phase, which was unexpected in this phase assemblage, was detected and identified as cerianite CeO $_2$ by EDS in conjunction with electron diffraction. Its orientation relationship to the main mineral phases can be described as a syntactical growth. The La-concentration of the bastnaesite changed with the distance from the cerianite interface, as confirmed by EDS. The reduction of La-content near the interface together with the respective crystallographic relationship yielded a model explaining the formation of the encountered mineral assemblage of bastnaesite, fluocerite and cerianite. The orientation matrix and the direction-plane relation for the three mineral phases was established.

Keywords: bastnaesite, orientation relationship, orientation matrix

FA2-MS16-P43

Ordering in intercalated Co atoms and electron density distributions of layered compounds Co_xTiS_2

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A structural study of layered compounds Co_xTiS_2 ($x=0.26, 0.43$, and 0.57) by X-ray diffraction analysis was performed to investigate both the ordered atomic arrangement and disordering behavior of intercalated Co atoms, and the nature of the chemical bond from the electron density distribution (EDD) obtained by the maximum entropy method. The $2a \times 2a \times 2c$ superstructure of the Co atoms is observed for $x=0.26$ and 0.57 , and disappears at 510 and 550K, respectively. On the other hand, $\sqrt{3}a \times \sqrt{3}a \times 2c$ superstructure of the Co atoms is observed for $x=0.43$, and disappears at 610 K. It is understood that the type of the transition is second-order-like. The overlapping of the EDD between Co and S atoms & Ti and S atoms are clearly seen, which are corresponding to the covalent bonding in the van der Waals gap layer and in the TiS_2 one. It is expected that nature of covalent bonding

between Co and S atoms causes decreasing of the interlayer distance.

Keywords: layered compounds, ordering of the Co atoms, EDD

FA2-MS16-P44

Structural trends in the $(emim)_m[A_n(Mo_8O_{26})]$ ($emim = 1$ -ethyl-3-methylimidazolium; $m=2,3$; $n=1,2$; $A=K, Rb, Cs$) group of compound. Anna Pakhomova, Sergey Krivovichev. *Saint-Petersburg State University, Russian Federation.*

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Polyoxometalates (POMs) have many properties that make them attractive for applications in medicine, biology, magnetism, materials science and catalysis. POMs are a kind of competent structure-tunable building block because they exhibit a wide variety of structural motifs of different sizes and topologies. As far as we know, all known octamolybdates has been synthesized by hydro- and solvothermal methods. To the best of our knowledge, ionothermal methods were not used, although they recently have shown high promise in the preparation of novel metal-organic frameworks.¹ We have used ionic liquid (emim)Br (emim=1-ethyl-3-methylimidazolium) as both solvent and template for the purpose of construction of novel organic-inorganic polymolybdates of alkali metals. The three novel octamolybdates (emim) $_3K(Mo_8O_{26})$ (**1**), (emim) $_3Rb(Mo_8O_{26})$ (**2**) and (emim) $_2Cs_2(Mo_8O_{26})$ (**3**) (emim = 1-ethyl-3-methylimidazolium) have been obtained in the ionothermal reactions. The structures of these compounds are described on the basis of octamolybdate anions $[\beta-Mo_8O_{26}]^{4-}$. In compounds **1** and **2**, $[\beta-Mo_8O_{26}]^{4-}$ units are linked by metal cations to form chain structures, while Cs cations in the compound **3** link to polyoxoanions to form layer structure.

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[1] Ranham E.R.; Morris R.E. *Acc. Chem. Res.* 2007, 40, 1005.

Keywords: ionothermal synthesis, alkali polyoxomolybdates, X-Ray crystal structure determination

FA2-MS16-P45

Comparison of HP data for X-ray diffraction in Beand diamond-backed DACs. Benedetta Periotto^a,

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High-pressure cells reduce significantly the accessible reciprocal space in single-crystal studies and, moreover, the parts of cells traversed by radiation produce as a rule strong background features. In order to increase the quality of measured data and accuracy of calculated structural parameters it is of primary importance to reduce these deleterious effects. In Diamond Anvil Cells (DAC) diamond anvils must be supported by a strong and X-ray transparent

material. For this, backing plates made of polycrystalline beryllium, which satisfies both criteria, have often been chosen. However, this metal presents several disadvantages. Besides its high toxicity and temperature sensitivity which limits its use to studies at moderate temperatures, it produces spotted and broad diffraction rings which influence large part of the background and can significantly impair measurement of weaker diffraction spots of investigated crystals. Miletich et al. [1] proposed diamond plates as an alternative anvil support. In the present study a comparison between high pressure structural data acquired using two DACs with the same construction (ETH-type) but equipped with diamond or beryllium backing plates, respectively, is presented in order to evaluate directly the possible improvements in data. A synthetic single crystal of $\text{NaInSi}_2\text{O}_6$ pyroxene was selected for the study and loaded consecutively into the two cells in equal orientation. Intensity data were collected using a Bruker-AXS four circle diffractometer equipped with a Smart1000 CCD area detector and a crystal of quartz was used as an internal pressure standard. The SMART [2] software was used for the data collection, SAINT+ [2] for the integration and reduction of data and ABSORB V.6.0 [3] for the absorption correction. The measurement strategy and the calculation procedures were equal in order to perform a reliable comparison. As already indicated by [1] the use of a low absorbing single crystal as a replacement of the commonly used polycrystalline beryllium as backing-plate material reduces the unwanted background from broad powder rings to only a few strong spots of a kind already present and originating from diamond anvils. We can prove a significant improvement in data quality as witnessed by better reliability factors in structure refinement and lower errors on the atomic coordinates, thermal parameters and average bond lengths. The resulting structural data are more reliable and able to reveal more subtle structural changes under pressure. The differences between the two structure refinements will be illustrated and discussed in detail.

[1] Miletich R., Allan D.R., Kuhs W.F., *Rev. Mineral. Geochem.* 2000, 41, 445. [2] Bruker-AXS products. [3] Angel R.J., *J. Appl. Crystallogr.* 2004, 37, 486.

Keywords: Diamond Anvil Cell, Single crystal X-ray diffraction, Be and diamond backing-plates

FA2-MS16-P46

Two novel ($\text{V}_{18}\text{O}_{42}$)-Isopolyoxovanadates. Sebastian Prinz, Georg Roth. *Institut für Kristallographie, RWTH Aachen, Germany.*

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Vanadium(IV) is found in a wide variety of different structures ranging from spin chains to square lattices to the cage-like polyoxovanadates. These materials often display interesting magnetic properties due to geometrical frustration of the spin-(1/2) centres. Dark brown single crystals were obtained from a mixture of caesium/rubidium hydroxide and vanadyl sulphate by hydrothermal synthesis at 240°C and autogenous pressure in Teflon-lined Parr autoclaves for three days. The furnace was then turned off and the autoclaves were left to cool down to room temperature. Novel Cs- and Rb-compounds with ($\text{V}_{18}\text{O}_{42}$)-cluster anions hosting single water molecules were obtained. The structure of these compounds was studied by single crystal X-ray diffraction on an Imaging Plate Diffraction System (IPDS II). It was found that both

compounds consist of anionic clusters of 18 VO_5 pyramids sharing edges and corners similar to those found by Müller and co-workers [1]. Cs and Rb ions compensate for the negative charge carried by the complex anions. Both compounds have similar crystal structures but contain less H_2O and crystallize in different space group symmetry than those characterized by Müller and co-workers. In our contribution we give a detailed description of the crystal structure and discuss the differences with the latter.

[1] Müller, A. et al., *Inorg. Chem.*, 1997, 36, 5239.

Keywords: polyoxovanadate, spin 1/2, hydrothermal

FA2-MS16-P47

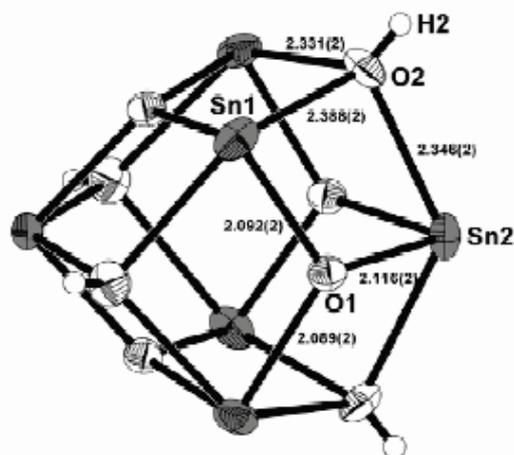
Low temperature, single crystal X-ray data of synthetic hydromarchite, $\text{Sn}_6\text{O}_4(\text{OH})_4$. Hans

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Aqueous solutions of tin(II) salts contain a lot of different mono- and polynuclear, cationic or anionic, oxo-hydroxo species depending on its salt and proton concentrations. From neutral solutions, colorless tin(II) monoxide, SnO , separates in form of a bad defined, voluminous, water insoluble precipitate, containing different amounts of water. A stoichiometric exact hydrate of composition, $\text{SnO} \cdot 1/3\text{H}_2\text{O}$, can only be prepared on certain conditions. Some older structure investigations [1],[2] reveal that this compound is a polynuclear molecular oxide hydroxide with formula $\text{Sn}_6\text{O}_4(\text{OH})_4$. This compound is also known to be a so-called anthropogenic mineral because archeologists found it on many tin objects which were covered for long periods by water. In mineralogy this compound is named hydromarchite.

Because it is difficult to grow appreciate single crystals on this compound our knowledge about its structure are limited. By chance we found single crystals of the title compound which were of high quality and large enough for X-ray diffraction experiments, in a more than ten years old sample of $^n\text{BuSnH}_3$ in toluene.



$\text{Sn}_6\text{O}_4\text{H}_4$: tetragonal, $P-42_1c$ (no. 114), $a = 7.8809(2) \text{ \AA}$, $c = 9.0595(4) \text{ \AA}$, $V = 562.67(3) \text{ \AA}^3$, $Z = 2$, $d_{\text{cal}} = 4.983 \text{ g/cm}^3$,