

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  $\text{VO}_5$  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  $\text{H}_2\text{O}$  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

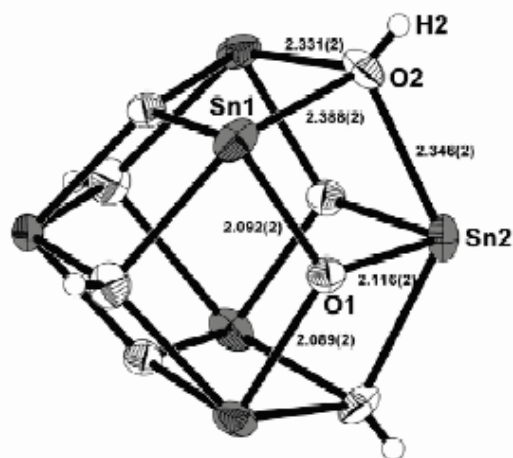
**Low temperature, single crystal X-ray data of synthetic hydromarchite,  $\text{Sn}_6\text{O}_4(\text{OH})_4$ .** Hans

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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,  $\text{SnO}$ , 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.



$\text{Sn}_6\text{O}_8\text{H}_4$ : tetragonal,  $P-42_1c$  (no. 114),  $a = 7.8809(2) \text{ \AA}$ ,  $c = 9.0595(4) \text{ \AA}$ ,  $V = 562.67(3) \text{ \AA}^3$ ,  $Z = 2$ ,  $d_{\text{cal}} = 4.983 \text{ g/cm}^3$ ,