

Table 1. Atomic coordinates and ADPs in $(K_{0.3}Rb_{0.8})\text{-}\beta\text{-ferrite}$.

(a) Model 1 ($R=0.0397$, $wR=0.1225$)

	site	occupancy	x	y	z	Uiso (Å)
O5	2c	1	1/3	2/3	1/4	0.021(2)
Rb1	6h	0.280(2)	0.6830(4)	0.3170(4)	1/4	0.0056(8)
K1	6h	0.099(9)	0.8824(15)	0.1176(15)	1/4	0.016(5)

(b) Model 2 ($R=0.0402$, $wR=0.1236$)

	site	occupancy	x	y	Z	Uiso (Å)
O5	2c	1	1/3	2/3	1/4	0.020(2)
Rb1	6h	0.263(4)	0.6829(4)	0.3171(4)	1/4	0.0055(8)
K1	6h	0.037(4)	0.6829(4)	0.3171(4)	1/4	0.0055(8)
Rb2	6h	0.015(8)	0.8826(15)	0.1174(15)	1/4	0.016(6)
K2	6h	0.065(8)	0.8826(15)	0.1174(15)	1/4	0.016(6)

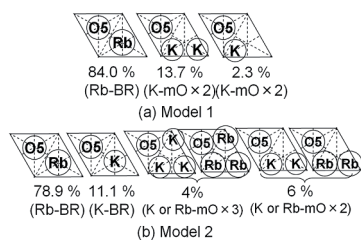


Fig.1 Distributions of K^+ and Rb^+ in $(K_{0.3}Rb_{0.8})\text{-}\beta\text{-ferrite}$.

[1] H. Watarai, K. Fujimoto, S. Ito, *ICC3 2010*, S1-P0245.

Keywords: structure refinement, mixed alkali beta-ferrite

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Structural analysis of a new complex containing tetrapropionatdirhodium units

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Rhodium dinuclear complexes with metal-metal bond with a variety of architectures, properties and different scientific approaches have been reported [1]. Although most of these compounds are based on discrete units, rhodium dimetallic species are currently used as building blocks to achieve an increasing number of coordination polymers with promising properties, mostly one-dimensional chains. However, many interesting arrangements based on dirhodium paddlewheel molecules can be obtained. In this abstract, we present a new dirhodium tetrapropionate with formula $\{K_2[Rh_2(O_2CET)_4(I)_2]\cdot H_2O\}$. In order to understand the structure of this compound a topological analysis has been carried out.

A good geometrical analysis of molecule-based structural types needs to answer, at least, two questions: the type of building blocks found in the structure, and the way in which these building blocks are connected. Sometimes, the resulting three-dimensional arrangement can be very complicated, and a conventional approach cannot clearly

explain the arrangement in the solid state. In these intricate structural types, it is often more illustrative to find the topological parameters that can describe the building blocks and explain their connectivity.

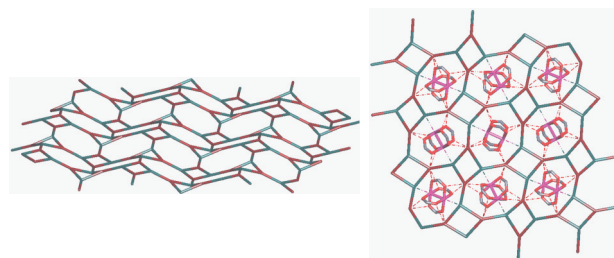


Figure 1. KI subnet (left) and simplified perpendicular view of a $\{K_2[Rh_2(O_2CET)_4(I)_2]\cdot H_2O\}_\infty$ layer (right).

Single crystal X-ray diffraction of $\{K_2[Rh_2(O_2CET)_4(I)_2]\cdot H_2O\}$ displays a complex arrangement of $[Rh_2(O_2CET)_4]$ paddlewheel molecules with the two axial positions occupied by two iodine atoms. However, the long Rh-I distance, which is higher than the sum of ionic radii, indicates the ionic nature of this interaction. The crystal structure can be described as a stacking of $\{K_2[Rh_2(O_2CET)_4(I)_2]\cdot H_2O\}_\infty$ layers parallel to the (-111) crystallographic plane. These layers consist of an undulated two-dimensional ionic network of KI, which can be topologically described as a 3-connected uninodal Shubnikov plane net (4.8^2). The dirhodium units are located inside the 8-member rings, displaying Rh-I and K-O ionic interactions with the inorganic framework. There are also weak hydrogen bonds between adjacent layers through the oxygen atoms from the water molecules bonded to the K^+ cations.

[1] F.A. Cotton, C.A. Murillo, R.A. Walton, *Multiple Bonds between Metal Atoms*, 2005.

Keywords: rhodium, coordination, crystallochemistry

MS67.P12

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Low temperature form of layer silicate RUB-15-LT, $[Si_{24}O_{48}(OH)_8][N(CH_3)_4]_8 \cdot 20H_2O$

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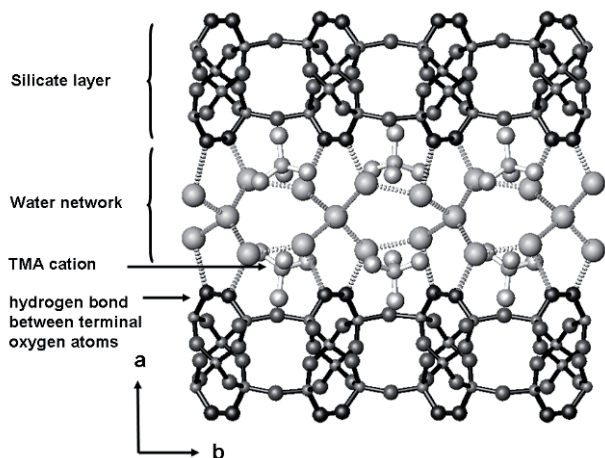
RUB-15 [1] is an interesting layer silicate with layers made up of 4- and 6-rings forming a puckered sheet of interconnected cups which point alternatively up and down. The cups can be described as one half of a sodalite cage with tetramethylammonium (TMA) cations intercalated between layers. Neighboring silicate layers are interconnected through additional hydrogen-bonded water molecules. This generates a 3-dim. bonding network consisting of layer like silicate ions and water molecules.

RUB-15 was synthesized at 130°C from a reaction mixture of SiO_2 / TMAOH / H_2O . Synchrotron powder diffraction data were collected between 295 and 150 K at HASYLAB, Hamburg to monitor the phase transition. For a structure analysis of RUB-15-LT a complete powder diffraction data set was collected at 150 K using a Bruker D8 diffractometer with $MoK\alpha$ radiation. To investigate hydrogen bonds 1H Solid-State MAS NMR spectra were recorded at RT on a Bruker ASX400 spectrometer.

RUB-15 shows a phase transition at ca. 200 K from ortho-rhombic to monoclinic symmetry. Upon cooling the unit cell volume shrinks from 2702.4 Å³ (295 K) to 2631.3 Å³ (150 K),

RUB-15-RT: $Ic2a$, $a_0 = 27.905 \text{ \AA}$, $b_0 = 8.408 \text{ \AA}$, $c_0 = 11.518 \text{ \AA}$,
 RUB-15-LT: $I2$, $a_0 = 27.4376 \text{ \AA}$, $b_0 = 11.4032 \text{ \AA}$, $c_0 = 8.4138 \text{ \AA}$,
 $\beta = 91.725^\circ$. The refinement converged to $R_{\text{Bragg}} = 4.1$, $\chi^2 = 2.4$.

The structures of RUB-15-RT and RUB-15-LT differ only slightly: The powder diagram of monoclinic RUB-15-LT still shows the reflection conditions for the (pseudo-) symmetry $Ic2a$.



However, for the low temperature form it is possible to analyze the bonding system between water molecules, siloxane groups and silanol groups in more detail. The analysis of this bonding network revealed strong intra-layer hydrogen bonds between neighboring oxygen atoms of the terminal SiOH/SiO-groups. The analysis allowed to distinguish between Si-OH and Si-O-groups of the silicate layer showing additional strong hydrogen bonds between the Si-O-groups and water molecules of the water network.

The ¹H NMR spectrum confirms the results of the X-ray structure analysis. It displays a broad signal at 3.4 ppm attributed to the protons of the TMA cation and of the water network, and a signal at 16.4 ppm corresponding to strong hydrogen bonds with an O...O distance of about 2.4 Å.

[1] U. Oberhagemann et al., *Angew. Chem. Intern. Ed.*, **1996**, *35*, 2869.

Keywords: layered, silicate, structure

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Large 2D-PSD for neutron single crystal structure analysis

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For structure analysis of complicated hydrogen-included materials, neutron single crystal structure analysis is very powerful tool. In addition, neutron single crystal experiment is indispensable for magnetic structure analysis for complicated magnetic system such like multiferroic materials. Usually, four-circle diffractometers are used to accumulate intensity data. The only difficulty is the lack of efficiency of the measuring time due to the low flux of neutrons in general. Usually it requires long beam time, and sometimes, it is impossible to carry out. In order to overcome such low efficiency on single crystal neutron experiments, we have developed a large two dimensional position sensitive neutron counter (2D-PSD), and applied to structure analysis.

The large curved 2D-PSD for neutrons we have developed has an active area size 937 x 520 mm². The radius of the detector is 535 mm,

and the two-theta resolution is 0.2 degree /CH (Fig. 1). Two detectors were fabricated and installed at KAERI-HANARO and JAEA-JRR3. As the test experiment, standard samples NaCl and Tb₃Fe₅O₁₂ (TbIG) are used. We could successfully performed structure analysis of TbIG with the following exposure conditions: the continuous rotation speed of the crystal is 0.4 degree/min, and 120 degree is covered, and exposure time is 5 hours. 962 Bragg points are covered including 181 absent Bragg reflections by symmetry. This is almost 10-20 times effective for consuming time compared with a conventional four-circle diffractometer. Not only the efficiency, we can survey unknown Bragg spots showing cell doubling at phase transition for instance. One of the 2D-PSD will be installed at the dedicated beam port at HANARO.

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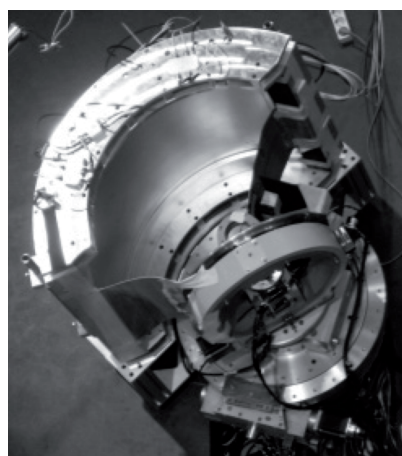


Fig 1. 2D-PSD

Keywords: 2D-PSD, neutron diffraction, structure analysis

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Data correction of a novel CMOS detector

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Area detectors typically have systematic measurement errors that must be calibrated and corrected before the data can be used for crystallography. The techniques for the correction of CCD data are well established. However, the newest generation of CMOS based detectors presents new challenges. In particular, CMOS detectors exhibit low level nonlinearities and line-to-line offset noise (so called reset noise). This fixed pattern variation must be characterized and requires compensation. Here we describe the characteristics of CMOS detectors, the origin of these systematic errors and the techniques that can be used to correct them. The algorithms presented have been implemented in a special purpose processor for real time correction of the data stream from a CMOS detector. For this purpose the detector was equipped with considerable FPGA processing power. This is essential in order to dynamically normalize data at the pixel level for minor variations in intensity responsiveness.

Keywords: detector, CMOS, software