Upon complex thermolysis in hydrogen the nanoalloy PD$_0.5$Rh$_0.5$ is formed ($\beta$-m-3m space group, $\alpha = 3.847$ Å). The thermolysis final temperature is 400 ºC, the average particle size is 20 nm. The work has been supported by RFBR Grant 08-03-00603, Presidium SB RAS interdisciplinary project No 112 and State contract № P960 of Federal target program «Scientific, Research And Teaching Specialists In Russia» 2009–2013.


**Keywords:** crystal structure analysis, X-ray phase determination.

**FA2-MS16-P53**

High resolution X-ray diffraction experiments for selected minerals. Marcin Stachowicz*, Maura Malinskaa, Jan Parafiniukb, Krzysztof Woźniakb.

aFaculty of Chemistry, University of Warsaw. bFaculty of Geology, University of Warsaw.

E-mail: marcin.stachowicz1@gmail.com

Jarosite is used as a by-product of the metal-processing industry as well as a common supergene mineral in ore deposits, and is associated with acid-mine waste. Jarosite is thought to exist on Mars, and its presence suggests that water existed on Mars in the past [1]. Celestine is the most abundant strontium mineral and is the principal commercial source of strontium [2].

The high resolution X-ray diffraction experiments have been carried out for series of natural minerals. Natojarosite NaFe$_3$(SO$_4$)$_3$(OH)$_3$ was mined in Greece and celestine SrSO$_4$ in Poland. These compounds crystallize in the trigonal R-3m and orthorhombic Pnma space groups, respectively. The multipole model within the Hansen-Coppens formalism [3] is applied for crystals containing disorder and heavy atoms. In American Mineralogist Crystal Structure Database are four structures of Celestine [2,4-6] all ordered, however fitting of the multipole models of electron density leads to identification of small degree of disorder. Several multipole model refinement strategies have been applied and compared to find the most suitable one. Topological analysis of electron densities estimated from multipole models gives opportunity for better understanding intermolecular interactions and bonding properties in these minerals.


**Keywords:** NASICON-type structure, Li-insertion, cation mixed occupancy.

**FA2-MS16-P54**

Structural investigations of Li$_3$Ti(MoO$_4$)$_3$. A. Thomas*, D. Mikhailova*, H. Ehrenberg*

*Institute for Complex Materials, IFW Dresden, Germany, Institute für Materialwissenschaft, Technische Universität Darmstadt, Germany.

E-mail: a.thomas@ifw-dresden.de

Complex molybdates with 3d transition metals crystallizing in a NASICON-type structure are promising materials for Li-storage during Li-insertion and deinsertion. Recently, a new molybdate with V(III), Li$_3$V(MoO$_4$)$_3$, was prepared and characterized. It was shown that Li-ions can be reversibly intercalated and deinserted with formation of Li$_3$V(MoO$_4$)$_3$ and Li$_2$V(MoO$_4$)$_3$ [1]. In the related system Li-Ti-Mo-O, only the composition Li$_3$Ti$_3$Mo$_3$(MoO$_4$)$_6$ with Ti(IV) and the same crystal structure type is known [2]. There are no examples in the literature for the coexistence of molybdenum(IV) and titanium(III) in one phase. Every stable product which contains titanium and molybdenum was synthesized from titanium(IV) and molybdenum(IV) composites, for example (Ti$_x$Mo$_{1-x}$)$_5$O$_{14}$ [3] and TiMo$_2$O$_8$ [4]. If titanium(III) and molybdenum(IV) were used as educts, molybdenum(IV) was reduced to metal and titanium(III) was oxidized to titanium(IV) [5].

A new phase with the composition Li$_3$Ti(MoO$_4$)$_3$ was synthesized by solid state reaction. It crystallizes in the space group $Pnma$ and is isostructural to Li$_3$V(MoO$_4$)$_3$. Similar electrochemical properties as for Li$_3$V(MoO$_4$)$_3$ are expected [1]. The material shows the same plateau at ca. 1.8 V during galvanostatic cycling against a Li-anode, which indicates the same two-phase mechanism during first Li-insertion. The similarity of Li’ and Ti’$^{3+}$ radii for octahedral oxygen coordination leads to mixed cation occupancy in the structure, which can be varied by different synthesis conditions. A difference in the lattice parameters for two samples, prepared at different temperatures, between $a = 5.0586(0)$ Å, $b = 10.4833(1) Å$ and $c = 17.5653(2) Å$ to $a = 5.0715(1) Å$, $b = 10.5096(2) Å$ and $c = 17.6050(3) Å$ confirms this assumption. In comparison the lattice parameters of Li$_3$Ti$_3$Mo$_3$(MoO$_4$)$_6$ are: $a = 5.0467(12)$, $b = 10.454(3)$ and $c = 17.5384(2)$ [2].


**Keywords:** NASICON-type structure, Li-insertion, cation mixed occupancy.

**FA2-MS16-P55**

Synthesis and structural studies of ammonium-cobalt-nickel phosphates, NH$_4$[Co$_{1-x}$Ni$_x$PO$_4$]$\cdot$H$_2$O.

Laura Torre-Fernández, Camino Trobajo, José R. García, Santiago García-Granda. Departamentos de Química Física y Analítica y Química Orgánica e Inorgánica, Universidad de Oviedo, Spain.

E-mail: torrelaura@uniovi.es