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Structural Study of the Lanthanide Phosphonate Family $[\text{H}_3\text{N}(\text{CH}_2)_4\text{NH}_3]\text{Ln}[\text{hedpH}][\text{hedpH}_2]$ Eva Fernández-Zapico^a, Laura Rocés^{a*}, Santiago García-Granda^a, José R. García^b, Fengyi Liu^c, João Rocha^c
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Keywords: structural characterization, phosphonates, hydrothermal synthesis

Metal phosphonate chemistry has attracted much attention due to the potential applications of these materials in ion-exchange, catalysis, and in sensor devices. Often, metal phosphonates exhibit polymeric structures consisting of chains, layers, or three-dimensional networks, but monomeric structures are also known. In particular, the tunable organic units in diphosphonate $[\text{R}(\text{PO}_3)_2]^{4-}$ allow the construction of metal phosphonate materials with new architectures. One type of these phosphonates is based on 1-hydroxyethane-1,1-diphosphonic acid $[\text{hedpH}_4, \text{H}_2\text{O}_3\text{PC}(\text{OH})(\text{CH}_3)\text{PO}_3\text{H}_2]$, which has been widely used as a strong chelating agent in the preparation of functional metal diphosphonates and found to bind strongly to numerous metal ions. We have recently reported the hydrothermal synthesis and crystal structure of several members in the new family of 1D lanthanide phosphonates $[\text{H}_3\text{N}(\text{CH}_2)_4\text{NH}_3]\text{Ln}[\text{hedpH}][\text{hedpH}_2]$, where Ln is a trivalent lanthanide metal. The crystal structure of these compound, which crystallize in space group P-1, is built up from one-dimensional covalent lanthanum phosphonate cross-linked chains and $[\text{NH}_3(\text{CH}_2)_4\text{NH}_3]^{2+}$ cations. Herein, we report the structural determination by synchrotron X-ray powder diffraction of all the members in this family (Ln: La, Tb, Yb, Sm, Nd, Pr, Gd, Eu, Er). The complete structural study of these compounds and the description of the intricate hydrogen bonding network which holds the chains together will be presented here.

[1] Rocés, L., García-Granda, S., García, J.R., Liu, F., Carlos, L.D. *Acta Cryst.* 2006. A62, s268.

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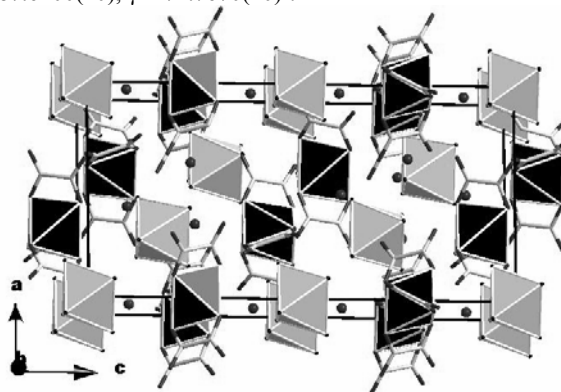
Synthesis and Investigation of Double Complex Salts $[\text{Ir}(\text{NH}_3)_6][\text{M}(\text{C}_2\text{O}_4)_3] \cdot n\text{H}_2\text{O}$ (M = Fe, Co, Cr) Evgeny Filatov^a, Kirill Yuesenko^a, Evgeniya Vikulova^b, Irida Baidina^a, Yuriy Shubin^a, ^aNikolaev Institute of Inorganic Chemistry, Novosibirsk, Russia. ^bFaculty of Natural Science, Novosibirsk State University, Russia. E-mail: decan@che.nsk.su

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Preparation of novel double complex salts (DCS) simultaneously comprising a platinum group metal and a forth raw transition metal is highly topical fro these compounds can serve as precursors of diverse bimetallic materials. Application of such systems can be exemplified by production of ultrafine particles of metal solid solutions or intermetallides deposited on various supports with the purpose of development of high-performance catalysts [1] having decreased content of noble metals.

Several DCS involving hexaammine cations and tris-oxalate anions are reported in the literature [2, 3]. However, only one study was devoted to the compound combining these ions [3]. The choice of the $[\text{Ir}(\text{NH}_3)_6]^{3+}$ cation was determined by the lack of data on structural features and thermal properties of such compounds.

Compounds $[\text{Ir}(\text{NH}_3)_6][\text{M}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ (M = Fe, Cr) are isostructural and crystallize in a trigonal space group P-3c1 with unit cell parameters $a = 12.6128(7)$, $c = 20.8480(22)$ Å and $a = 12.608(7)$, $b = 21.180(14)$ Å, respectively. The compound $[\text{Ir}(\text{NH}_3)_6][\text{Co}(\text{C}_2\text{O}_4)_3]$ does not contain water of crystallization and belongs to a triclinic space group P1 with parameters $a = 7.5630(2)$, $b = 9.6046(3)$, $c = 11.8116(4)$ Å, $\alpha = 84.7950(10)$ Å, $\beta = 87.6100(10)$, $\gamma = 71.7670(10)^\circ$.



Thermal decomposition of the title compounds has been studied. Final products of thermal decomposition are single-phase cubic closed-packed metal solid solutions $\text{Ir}_{0.5}\text{Fe}_{0.5}$ ($a = 3.745(4)$ Å) and $\text{Ir}_{0.5}\text{Co}_{0.5}$ ($a = 3.710(3)$ Å). Thermolysis of $[\text{Ir}(\text{NH}_3)_6][\text{Cr}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ affords a solid solution $\text{Ir}_x\text{Cr}_{1-x}$ ($x \sim 0.5$, $a = 2.690(2)$, $b = 4.291(4)$ Å) and a small amount of chromium oxide..

[1] Korenev S.V., Venediktov A.B., Shubin Yu.V., Gromilov S.A., and Yusenko K.V., *Russ. J. Struct. Chem.*, 2003, 44, 58.

[2] Bolshakova L.D, Lapkin V.V., *Russ. J. Inorg. Chem.*, 1997, 42, №9, 1497.

[3] Pecheniuk S.I., Semushina A.V., Kadyrova G.I., *Coord. Chem.*, 2005, 31, №12, 912.

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The Structures and Kinetics of Lithium Magnesium Alanates Hilde Grove, Ole Martin Løvvik, Bjørn C. Hauback, *Physics Department, Institute for Energy Technology, Kjeller, Norway.* E-mail: hilde.grove@ife.no

Keywords: Hydride structure, neutron powder diffraction, X-ray powder diffraction.

Safe storage and transportation are one of the limiting factors for a wide use of hydrogen as an energy carrier. Solid compounds like metal hydrides contribute a promising alternative for traditional hydrogen storage. The 4 wt % hydrogen capacity for NaAlH_4 containing Ti-catalyst, showed about 10 years ago [1] the potential for reversible hydrogen storage in complex hydrides. In the search for novel materials for hydrogen storage the mixed alanate $\text{LiMg}(\text{AlD}_4)_3$ has been considered [2-3]. LiMgAlD_6 was synthesized by using the ball milling technique. $3\text{LiAlD}_4 + \text{MgCl}_2 \rightarrow 2\text{LiCl} + \text{LiMg}(\text{AlD}_4)_3$. The product was recrystallized in diethyl ether/toluene to increase the crystallinity and to remove LiCl. The sample