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at 800°C) was established by the Lacorre et al [1]. There is no such a compound in the Nd₂O₃ - MoO₃ system, but there is one with the fluorite-like structure near the Nd₂MoO₆ [2], [3], [4]. Composition and crystal structure of the Nd₂MoO₆ were studied before on polycrystals [4] as well as similar structure of Pr₂MoO₆ [5]. It was found that the Pr₂MoO₆ crystals have a fluorite-like superstructure in Pn-3n sp. gr. There are Pr₁ at 12e position, Pr₂ at 8c, Mo at 16f, and two kinds of oxygen atoms O₁ at 4b and O₂ at 16f. Overestimated thermal parameters are given in [5] for the Pr₂ (B(eq=1.32(16) Å²) and Mo (B(eq=0.98(7) Å²) atoms, while B₅=0.7(4) Å² for the O₂ atom.

Single-crystal structure refinement of Nd₅Mo₃O₁₆ and study of the physical properties have been performed. Dark-violet cubic Nd₅Mo₃O₁₆ single crystals were grown by spontaneous flux crystallization. The conductivity of the compound is hypothetically anion and reaches approximately 10⁻⁵ S/cm at 800°C.

The structure of Nd₅Mo₃O₁₆ single crystal was studied by the X-ray techniques (XCalibur S CCD-diffractometer, T=293 K). The unit cell was found to be cubic with a=11.028(1) Å, which relates to that of fluorite CaF₂ as a=2a. The structure was solved using the Pn-3n symmetry group. It was found that the Nd1 and Mo atoms are shifted from their positions on the 4-fold axis with the formation of 4 positions (0.25 occupation) around it. The Nd2 atoms are not disordered; they are located on the 3-fold axis and are surrounded by oxygens with 6 distances around it. The Nd2 atoms are not disordered; they are located on the 3-fold axis and are surrounded by oxygens with 6 distances around it. The Nd2 atoms are not disordered; they are located on the 3-fold axis and are surrounded by oxygens with 6 distances around it.

Inclusion of Mo atoms into Nd₅Mo₃O₁₆ structure, which are surrounded by O₂ atoms (Mo-O₂ distances from 1.695 to 1.892 Å) lead to the strong distortion of the Nd1 – polyhedron and to the deviation of the structure framework from the fluorite-like. Some shift of the Nd1 and Mo atoms from the axis appears as a result, which was established in the present work (R=3.6%, R(eq=3.27%, S=1.99, B=0.24(1) Å² for the Pr2 atom).

This study was supported in part by the RFBR Grant No. 11-03-00243 and the Council on Grants of the President of the Russian Federation for the Support of Leading Scientific Schools (project no. NSh 4034.2010.5).

**Keywords:** Nd₅Mo₃O₁₆, crystal structure, oxide-ion conductivity

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**MS81.P34**

**Synthesis and structure of new vanadium(V) complex based on isonicotinic hydrazide**

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Although the biological significance of vanadium was recognized long ago, the coordination chemistry of this element continues to attract the attention of researchers, not least because of the discovery of vanadium as a component of several enzymes, such as some peroxidases, as models for bromoperoxidase, nitrogenases and other biological systems.

The crystalline coordination compound VO₂L₂H₂O has been obtained as the result of interaction of 2,4-pentanedione isonicotinic hydrazone (HL) with VO₂SO₃H₂O. This compound is soluble in chloroform, dimethylformamide, less soluble in EtOH and MeOH.

The product VO₂L₂H₂O is characterized using IR spectroscopy, elemental analysis and X-ray method.

The disappearance of the band at 1624 cm⁻¹ (ν(C=O)) and the absence of oscillations bands of N-H, as well as the appearance of absorbance in the regions 2700-2500 and 2250-2000 cm⁻¹ attributed to vibrations of =C=N=H in the spectrum of VO₂L₂H₂O as compared to that of the uncoordinated ligand, indicates upon the coordination L in enolic form with proton migration (from NH) to the heterocyclic nitrogen. The presence of a narrow band of moderate intensity at 1636 cm⁻¹ confirms the formation =C=N=H= . The new band at 1520 cm⁻¹ may be attributed to the oscillations of coordinated ν(C=O)ₙₑₓₑ.

The crystallization water molecules are associated (ν(OH)=3215 cm⁻¹), forming three hydrogen bonds. The oscillations ν(N=O) are manifested at 908 cm⁻¹ and is the most intensive band in the spectrum.

According to the x-ray single crystal study, the isolated compound VO₂L₂H₂O forms a molecular structure composed by neutral [VO₂L₂] complexes and solvate water molecules in 1:2 ration. Vanadium atom adopts a NO₃ trigonal-bipiramidal environment, being coordinated by two oxygen atoms and one tridentate Schiff-base ligand L.

**Acknowledgements:** The authors gratefully acknowledge the financial support of SCSTD of ASM (Grant for young scientists 10.819.05.04. F)

**Keywords:** coordination compounds, vanadium, Schiff-base

**MS81.P35**

**X-Ray studies in a manganese lawsonate coordination polymer**

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Lawsonite (2-hydroxy-1,4-naphthoquinone) is a natural spin carrier in metal complexes molecules. The redox properties of quinones and their complexes have been explored for applications such as the development of new drugs for cancer [1] and switches for molecular electronics [2]. Here, we describe the synthesis and the characterization of [Mn(C₅H₄O₂)₃]ₙ by infrared and ultraviolet spectroscopy, thermogravimetric analysis and single-crystal X-ray diffraction. X-ray data shows the formation of a coordination polymer with the manganese atom in a center of a fully distorted octahedron (C2/c). The carbonyl and the phenoxy oxygens (O1 and O2, respectively) chelate to the manganese, and the other carbonyl oxygen (O3) bridges to another metal center forming a 2D arrangement. Temperature dependent X-ray studies (225K and 110K) shows a significantly variation in bond distances around the metal center, which indicates change in the bond length.
character. A similar behavior reported early for an analogue complex with lapachol suggest an electron transfer process between the metal center and the naphtoquinonate [3].

**MS81.P36**


**Synthesis and investigation of double complex salts involving [AuHal₄]⁻ anions**

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Double complex salts (DCS) are promising single-source precursors for preparation of ultrafine metal systems. The composition of a DCS can be represented with the general formula \([ML₄]([M′L′]₈)_n\), where \(M, M′\) — a transition metal, \(L, L′\) — a neutral or charged ligand. Thermal decomposition of such complexes, occurring at relatively low temperatures, allows targeted preparation of metallic systems with composition strictly pre-determined by the stoichiometry of the original DCS [1].

DCS’s \([M(NH₃)₄]H[AuCl₄][CH₃NH₃]_n\) \((\text{Hal} = \text{Cl, Br})\) and \([M(NH₃)₄]H[AuBr₇]_n\) \((M = \text{Ir, Rh}; n = 0-1)\) have been synthesized and characterized with a number of physical chemical techniques. Crystal structures have been determined for all compounds. Single crystal X-ray diffraction analysis was carried out on a Bruker X8 APEX CCD-based diffractometer with MoKα-radiation at 150 K. The structures were solved by standard direct methods and refined anisotropically using the SHELX-97 program package [2]. All hydrogen atoms were located geometrically.

\([M(NH₃)₄]H[AuCl₄][CH₃NH₃]_n\) are monoclinic, sp. gr. C2/m; \([M(NH₃)₄]H[AuCl₄][NO]_n\) are orthorhombic, sp. gr. Pnma. \([M(NH₃)₄]H[AuBr₇]_n\) crystallizes in the triclinic crystal system, sp. gr. P-1.

It was shown that DCS’s containing \([AuCl₄]⁻\) anions can be obtained with high yield (60-80%) with stoichiometry of \(M{:}Au (1:1)\) and DCS’s containing \([AuBr₇]⁻\) with stoichiometry \(M{:}Au (1:2)\). Compounds with \([AuBr₇]⁻\) have structures with stacks of complex anions along \(a\) axis (figure shows packing for \([\text{Rh(NH₃)}₄]\)[AuBr₇] \(\cdot\) \(2\) \(\text{H₂O}\) with \(\text{Au...Au}\) distances 3.966(1) – 5.816(1) Å.

The thermal decomposition of the DCS’s in different atmospheres has been examined by thermal analysis. The mechanism of the process has been suggested on the basis of the data of thermal analysis, powder X-ray diffraction and IR spectroscopy. It has been demonstrated that ultrafine powders of solid solutions Ir-Au and Rh-Au can be prepared on thermal decomposition of the synthesized compounds. Phase composition and particle sizes of the bimetallic products have been found to be essentially determined by the process conditions: atmosphere, temperature, heating rate.

The work has been supported by RFBR Grant 11-03-00668-a, Presidium SB RAS interdisciplinary project No 112 and State contract № P960 of Federal target program «Scientific, Research And Teaching Specialists In Russia» 2009–2013.

**MS81.P37**


**Assembly a new water-insoluble strontium metal-organic framework and its luminescent studies**

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The research in the field of metal-organic frameworks (MOFs) has grown exponentially in the last decade due to potential applications of MOFs such as gas storage, catalysis, magnetism, luminescence, and gas separation. Recently, we have reported a new magnesium complex with BTEC which can be easily prepared by direct-mixing method starting from the acid, metal hydroxide, and water. It is desired to apply such a kind of environmentally friendly and efficient chemical processes to the syntheses of diverse chemical compounds, such as metal-organic frameworks or metal coordination polymers. The compound \([\text{Sr}_2(BTEC)(\text{H}_2\text{O})_2]_2\cdot\text{H}_2\text{O} (1)\) \((\text{H}_2\text{BTEC} = \text{benzene-1,2,4,5-tetraacarboxylic acid})\) is a new strontium metal-organic framework which is insoluble in water and emits strong luminescence at around 437 nm after dehydration. Single-crystal X-ray diffraction shows that the structure of compound 1 possesses an extended 3D framework. Each \(\text{SrO}_₆\) polyhedron shares its three edges with other three \(\text{SrO}_₆\) polyhedra forming thus an inorganic layer lying in the bc plane. The layers are pillared by the BTEC ligands and form a 3D metal-organic framework.

**Keywords:** strontium complex, metal-organic framework, luminescent

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**Keywords:** coordination polymer, lawsonite, manganese complex


**Keywords:** crystal structure analysis, gold compounds, thermal decomposition.