

## FA2-MS16-P50

**Crystal Structures of  $[M^A(NH_3)Cl]M^BO_4$  ( $M^A = Co, Rh, Ir$ ;  $M^B = Mo, W$ ).** Elena Shusharina<sup>a,b</sup>, Svetlana Khramenko<sup>b</sup>, Pavel Plusnin<sup>b</sup>, Sergey Gromilov<sup>b</sup>.

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Double complex salts can be used as precursors for the preparation of bimetallic alloys of high-melting metals upon temperatures less than 600 °C. The crystals of the title compounds have been synthesized by mixing together the aqueous solutions of complex salts containing  $[M^A(NH_3)Cl]^{2+}$  ( $M^A = Co, Rh, Ir$ ) cations and  $MoO_4^{2-}$  or  $WO_4^{2-}$  anions. Single crystal X-ray diffraction analysis was carried out on a Bruker X8 APEX CCD-based diffractometer with  $MoK\alpha$ -radiation at 150 K. All involved complex salts are isostructural and crystallize in the orthorhombic space group  $Pnma$  ( $Z = 4$ ). The compounds have the island crystal structures with the isolated ions. Thermal decomposition in the reducing atmosphere of the double complex salts occurs at temperatures 100–600° K. The XRD analysis of the thermolysis products was performed on an ARL X'TRA diffractometer with  $CuK\alpha$ -radiation and shows the solid solutions formation of title metals with particle sizes less than 50 nm. The final products phase composition is shown to depend on phase diagrams of the respective bimetallic systems. Owing to the fact that those complex salts are isostructural it is possible to prepare the solid solutions and the triple and quadruple alloys after their decomposition.

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

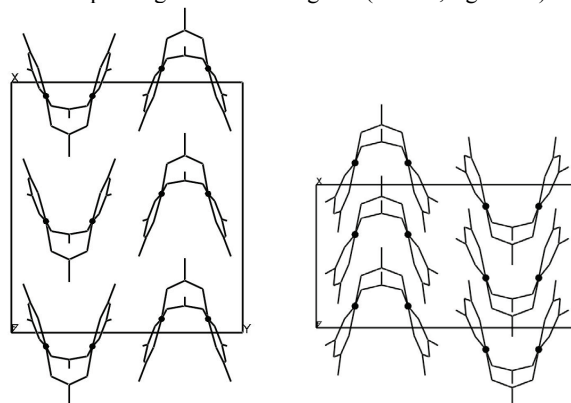
## FA2-MS16-P51

**Two modifications of  $Pd_2(\mu-CH_3COO)_2(CH(CH_3CO)_2)_2$**  Elena Shusharina<sup>a,b</sup>, Svetlana Khramenko<sup>b</sup>, Sergey Gromilov<sup>a,b</sup>. <sup>a</sup>Novosibirsk State University, Russia. <sup>b</sup>Nikolaev Institute of Inorganic Chemistry, Novosibirsk, Russia.

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The reaction of  $Pd_3(CH_3COO)_3NO_2$  with acetylacetone (1:3) dissolved in chloroform gives the  $Pd_2(\mu-CH_3COO)_2(CH(CH_3CO)_2)_2$  solution. Upon crystallization of the solution with a small amount of acetic acid the mixture of 3 crystalline phases was formed, two modifications of  $Pd_2(\mu-CH_3COO)_2(CH(CH_3CO)_2)_2$  and a small impurity of  $Pd(CH(CH_3CO)_2)_2$ . X-ray data were collected on a Bruker X8APEX automated diffractometer ( $MoK\alpha$ -radiation, graphite monochromator, CCD-detector) at 150(2) K and at the ambient temperature. Crystallographic data for modification I, T= 150(2) K:  $a = 15.6585(5)$ ,  $b = 14.4689(4)$ ,  $c = 8.0876(3)$  Å, space group  $Pnma$ ,  $V = 1832.3(1)$  Å<sup>3</sup>,  $Z = 4$ ,  $d_{calc} = 1.918$  g/cm<sup>3</sup>; for modification II, T= 150(2) K:  $a = 8.0285(3)$ ,  $b = 14.4785(5)$ ,  $c = 15.2092(5)$  Å, space group  $Pnma$ ,  $V = 1767.9(1)$  Å<sup>3</sup>,  $Z = 4$ ,  $d_{calc} = 1.988$  g/cm<sup>3</sup>. The structures were solved by standard direct methods and refined in an anisotropic (isotropic for H atoms) approximation. All calculations were performed using the SHELX-97 program

package [1]. The H atoms were located experimentally. The Pd...Pd distances are 2.9040(2) (I) and 2.9316(3) Å (II). The molecule packing is shown in Figures (left – I, right – II).



No significant changes in the crystal structures at the ambient temperature have been observed. Series of four unit cell measurements (through 50 K from 147 to 297 K) have shown no phase transitions.

[1] Sheldrick, G.M., *Acta Crystallogr.* 2008, A64, 112.

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

## FA2-MS16-P52

**First Example of Crystal Structure Containing  $[RhNH_3(NO_2)_5]^{2-}$  Anion.** Elena Shusharina<sup>a,b</sup>, Aleksandra Rybinskaya<sup>a,b</sup>, Pavel Plusnin<sup>a,b</sup>, Sergey Korenev<sup>a,b</sup>, Sergey Gromilov<sup>a,b</sup>. <sup>a</sup>Novosibirsk State University, Russia. <sup>b</sup>Nikolaev Institute of Inorganic Chemistry, Novosibirsk, Russia.

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The crystal structure of complex salts containing  $[Rh(NO_2)_6]^{3-}$  and  $[Rh(NO_2)_4(NH_3)_2]^{2-}$  anions can be found in literature. The current work covers the crystal structure of a double complex salt containing the  $[RhNH_3(NO_2)_5]^{2-}$  anion. The synthesis of the  $[Pd(NH_3)_4][RhNH_3(NO_2)_5]$  salt can be summarized in the following scheme:  $Na_3[RhCl_6] \rightarrow (NH_4)_2[RhNH_3Cl_5] \rightarrow K_2[RhNH_3Cl_5] \rightarrow K_2[RhNH_3(NO_2)_5] \rightarrow [Pd(NH_3)_4][RhNH_3(NO_2)_5]$ . The crystal structure of the latter compound has been determined by X-ray diffraction. The data from a transparent crystal of the compound was collected on a Bruker X8APEX automated diffractometer ( $MoK\alpha$ -radiation, graphite monochromator, CCD-detector) in the  $\theta$  range of 2.83° to 30.52° at 150(2) K. Crystallographic data for H15N10O10PdRh:  $a = 7.6458(5)$ ,  $b = 9.8813(6)$ ,  $c = 9.5788(7)$  Å,  $\beta = 109.469(2)^\circ$ , space group  $P2_1/m$ ,  $V = 682.30(8)$  Å<sup>3</sup>,  $Z = 2$ ,  $d_{calc} = 2.553$  g/cm<sup>3</sup>. The structure was solved by standard direct methods and refined anisotropically using the SHELX-97 program package [1]. All hydrogen atoms were located geometrically. The Rh-N( $NO_2$ ) distances range from 2.020(4) to 2.060(3) Å, which are similar to that in  $Na(NH_4)_2[Rh(NO_2)_6]$  (2.051(2) Å). The Rh-N( $NH_3$ ) distance is 2.074(4) Å. The shortest intermolecular distances  $N(NH_3)...O(NO_2)$  are shown in Figure.