o.m12.p22.la Crystal structure of palladium 2-methylthio-8-mercaptoquinolinate {Pd₃[C₉H₅(SCH₂)NS]₃}. E. Silina1, Yu. Bankovsky1, V. Belsky2, A. Stash2, L. Pech1, J. Lejejs1. Institute of Inorganic Chemistry of the Riga Technical University, L.Karpov Institute of Physical Chemistry, Moscow, Russia.

Keywords: crystal structure, palladium complexes, 8-mercaptoquinolinate.

Palladium 2-methylthio-8-mercaptoquinolinate has been synthesized in the scope of systematic comparative structure investigations of chelates of 8-hydroxy-, 8-mercapto- and 8-hydroselenoquinolines with metals.

The complex was prepared by interaction of 2-methylthio-8-mercaptoquinoline with palladium chloride in aqueous ethanol medium. The chemical formula of palladium 2-methylthio-8-mercapto-quinolinate- $\{Pd_3[C_9H_5(SCH_2)NS]_3\}$ was established on the basis of X-ray diffraction analysis.

The structure is built of trimeric Pd₃[C₉H₅(SCH₂)NS]₃} units. The thridentatic (S.N.C) function of the ligand - 2methylthio-8-mercaptoquinoline - has been observed for the first time. Three ligands act as divalent anions in tridentate (S,N,C-) fashion between three palladium atoms forming neutral unit. Each palladium atom forms one 5membered metal-containing ring with one 2-methylthio-8mercaptoquinoline ligand via covalent bonds Pd-S (2.348(6)-2.384(7)Å) and Pd-N (1.971(18)-2.015(15)Å). Palladium atom has been established to substitute the hydrogen atom in the methylthio group, and the second 5member of metal-containing ring containing the same ligand is closed by formation of a strong covalent bond Pd-C (2.03(2)-2.05(2)Å). Each S atom bridges two palladium atoms inside the molecular units. The bond Pd-S(bridge) is always shorter than the Pd-S(chelate) bond. The ranges of the bond angle values: chelate angles SPdN 84.8(5) -85.1(5)°; NPdC 86.2(9) - 87.4(8)°; interligand angles S(chelate)PdS (bridge) 92.7(2) - 96.0(2)°; S(bridge)PdC 92.9(7) - 95.9(6)°; S(chelate)PdC 170.4(6) - 171.1(7)° and NPdS (bridge) 170.6(5) - 174.6(5)°. The three crystallographically nonequivalent palladium atoms are in distorted cis-square (2S+N+C) coordination. The central part of the complex is formed by nonplanar six-membered heterocycle 3Pd+3S consisting from the alternating palladium and mercapto group sulfur atoms. The values of the bond angles SPdS and PdSPd are in the range of 92.7(2) to $96.0(2)^{\circ}$ and of 92.0(2) to $105.9(2)^{\circ}$ correspondingly.

The structure of $\{Pd_3[C_9H_5(SCH_2)NS]_3\}$ differs essentially from that of palladium 2-methy-8-mercaptoquinolinate $Pd[C_9H_5(CH_3)NS]_2^{-1}$ in which a distorted *cis*-square surrounding (2S+2N) is characteristic of the palladium atom.

Crystal data for $\{Pd_3[C_9H_5(SCH_2)NS]_3\}$: triclinic, a= 8.256(2); b= 14.706(3); c= 15.638(3) Å; α = 115.29(3); β = 96.76(3); γ =96.35(3)°; V=1677.2(6)ų; Z=2; D_c= 1.840 g/cm³; sp.gr. P-1; CAD-4; λ MoK α ; R=0.0593.

o.m13.p23.la Tripotassium trichromium (III) tetraarsenate K₃Cr₃(AsO₄)₄ Synthesis and structural study. B. Bouzemi, H. Boughzala* & T. Jouini. Département de Chimie, Faculté des Sciences, Université de Tunis II Campus Universitaire 1060 Tunis. TUNISIA. habib.boughzala@ipein.rnu.tn Keywords: single crystal, arsenate, chromium.

The tripotassium trichromium (III) tetraarsenate $K_3Cr_3(AsO_4)_4$ crystallises in the Cmca (n°64) space group with a=10.671(1)Å, b=20.911(5)Å, c=6.500(3)Å, V=1450.4(8)ų, Z=4, R(F²)= 0.0501 and Rw(F²)=0.1529 for 924 reflections with F²>2 σ (F²). The structure consists of CrO_6 octahedra and AsO_4 tetrahedra sharing corners and edges to form a tow-dimensional framework. Mixed Cr-O-As bridges link these polyhedron together. The K $^+$ cations are located in the inter layer space.

^[1] Pech L., Bankovsky Yu., Kemme A., Silina E., Ashaks J. "Crystal and molecular structure of palladium 2-methyl-8-mercapto-quinolinate.", Latvian Journal of Chemistry, 1998, N 2: 93.