o.m12.p22.la Crystal structure of palladium 2-methylthio-8-mercaptoquinolinate $\left\{\mathrm{Pd}_{3}\left[\mathrm{C}_{9} \mathbf{H}_{5}\left(\mathbf{S C H}_{2}\right) \mathrm{NS}\right]_{3}\right\}$. E. Silina1, Yu. Bankovsky1, V. Belsky2, A. Stash2, L. Pech1, J. Lejejs1. Institute of Inorganic Chemistry of the $e$ 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$\left\{\mathrm{Pd}_{3}\left[\mathrm{C}_{9} \mathrm{H}_{5}\left(\mathrm{SCH}_{2}\right) \mathrm{NS}\right]_{3}\right\}$ was established on the basis of Xray diffraction analysis.

The structure is built of trimeric $\left.\mathrm{Pd}_{3}\left[\mathrm{C}_{9} \mathrm{H}_{5}\left(\mathrm{SCH}_{2}\right) \mathrm{NS}\right]_{3}\right\}$ units. The thridentatic ( $\mathrm{S}, \mathrm{N}, \mathrm{C}$ ) function of the ligand - 2-methylthio-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) $\AA$ ) and $\operatorname{Pd-N}(1.971(18)-2.015(15) \AA$ ). 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 PdC (2.03(2)-2.05(2) $\AA$ ). Each S atom bridges two palladium atoms inside the molecular units. The bond $\mathrm{Pd}-\mathrm{S}($ 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) ${ }^{\circ}$; NPdC 86.2(9) - 87.4(8) ${ }^{\circ}$; interligand angles S(chelate)PdS (bridge) 92.7(2) - 96.0(2) ${ }^{\circ}$; S(bridge)PdC 92.9(7) - 95.9(6) ${ }^{\circ}$; S(chelate)PdC 170.4(6) - 171.1(7) ${ }^{\circ}$ and NPdS (bridge) 170.6(5) - 174.6(5) ${ }^{\circ}$. The three crystallographically nonequivalent palladium atoms are in distorted cis-square $(2 \mathrm{~S}+\mathrm{N}+\mathrm{C})$ coordination. The central part of the complex is formed by nonplanar six-membered heterocycle $3 \mathrm{Pd}+3 \mathrm{~S}$ 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 $\left\{\mathrm{Pd}_{3}\left[\mathrm{C}_{9} \mathrm{H}_{5}\left(\mathrm{SCH}_{2}\right) \mathrm{NS}\right]_{3}\right\}$ differs essentially from that of palladium 2-methy-8mercaptoquinolinate $\operatorname{Pd}\left[\mathrm{C}_{9} \mathrm{H}_{5}\left(\mathrm{CH}_{3}\right) \mathrm{NS}\right]_{2}{ }^{1}$ in which a distorted cis-square surrounding $(2 \mathrm{~S}+2 \mathrm{~N})$ is characteristic of the palladium atom.

Crystal data for $\left\{\mathrm{Pd}_{3}\left[\mathrm{C}_{9} \mathrm{H}_{5}\left(\mathrm{SCH}_{2}\right) \mathrm{NS}\right]_{3}\right\}$ : triclinic, $\mathrm{a}=$ 8.256(2); $b=14.706(3) ; c=15.638(3) \AA ; \quad \alpha=115.29(3)$; $\beta=96.76(3) ; \gamma=96.35(3)^{\circ} ; \mathrm{V}=1677.2(6) \AA^{3} ; \mathrm{Z}=2 ; \mathrm{D}_{\mathrm{c}}=1.840$ $\mathrm{g} / \mathrm{cm}^{3}$; sp.gr. P-1; CAD-4; $\lambda \mathrm{MoK} \alpha ; \mathrm{R}=0.0593$.

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#### Abstract

o.m13.p23.1a Tripotassium trichromium (III) tetraarsenate $\mathrm{K}_{3} \mathrm{Cr}_{3}\left(\mathrm{AsO}_{4}\right)_{4}$ 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 $\mathrm{K}_{3} \mathrm{Cr}_{3}\left(\mathrm{AsO}_{4}\right)_{4}$ crystallises in the Cmca ( $\mathrm{n}^{\circ} 64$ ) space group with $\quad \mathrm{a}=10.671(1) \AA, \quad \mathrm{b}=20.911(5) \AA, \quad \mathrm{c}=6.500(3) \AA$, $\mathrm{V}=1450.4(8) \AA^{3}, \mathrm{Z}=4, \mathrm{R}\left(\mathrm{F}^{2}\right)=0.0501$ and $\operatorname{Rw}\left(\mathrm{F}^{2}\right)=0.1529$ for 924 reflections with $\mathrm{F}^{2}>2 \sigma\left(\mathrm{~F}^{2}\right)$. The structure consists of $\mathrm{CrO}_{6}$ octahedra and $\mathrm{AsO}_{4}$ tetrahedra sharing corners and edges to form a tow-dimensional framework. Mixed Cr-OAs bridges link these polyhedron together. The $\mathrm{K}^{+}$cations are located in the inter layer space.


[^0]:    [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.

