

## LETTERS TO THE EDITOR

# Trinuclear Rhodium $\mu_3$ -Oxoacetate Complexes with Water and $\beta$ -Picoline

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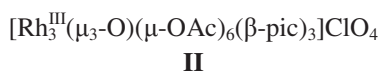
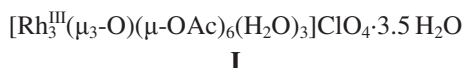
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The information on the synthesis and structure of rhodium(III) oxygen-bridged carboxylate complexes is extremely poor [1–3]. We have synthesized and characterized two new trinuclear rhodium  $\mu_3$ -oxoacetate complexes **I** and **II**.



According to the X-ray diffraction analysis, complexes **I** and **II** contain a  $\text{Rh}_3(\mu_3\text{-O})$  framework which represents an almost regular triangle of rhodium atoms. These rhodium atoms are bound pairwise by acetate bridging groups. The octahedral coordination of rhodium atoms is supplemented with donor atoms of water or  $\beta$ -picoline molecules. The  $\text{Rh-O}(\mu_3\text{-O})$  distances and, consequently,  $\text{Rh} \cdots \text{Rh}$  distances increase in going from  $\text{L} = \text{H}_2\text{O}$  to  $\beta$ -pic, and the average  $\text{Rh-N}$  distance ( $\text{L} = \beta\text{-pic}$ ) is shorter than the averaged  $\text{Rh-O}$  distances ( $\text{L} = \text{H}_2\text{O}$ ).

Complex **I** crystallizes in a monoclinic system, space group  $C2/c$ ;  $a$  26.23,  $b$  14.068,  $c$  22.05 Å;  $b$  130.50°,  $Z$  8,  $R$  5.83%.

Complex **I** has an extensive system of intermolecular hydrogen bonds, which allows it to be considered as a supramolecular complex compound.

Complex **II** crystallizes in a monoclinic system, space group  $P2_1/c$ ;  $a$  26.023,  $b$  7.994,  $c$  20.990 Å;  $b$  105.65°,  $Z$  4,  $R$  3.69%. Unlike **I**, structure **II** contains no hydrogen bonds and supramolecular constructions.

**( $\mu_3$ -Oxo)hexakis( $\mu$ -acetato)triaquatrirhodium(III) perchlorate 3.5-hydrate (I).** A 0.2-g sample of rhodium(II) acetate  $\text{Rh}_2(\mu\text{-OAc})_4(\text{H}_2\text{O})$  was dissolved in 20 ml of glacial acetic acid. The solution was ozonized at 90–100°C within 3 h (an OGVK-02K ozonizer, consumption of an oxygen–ozone mixture 25 l h<sup>−1</sup>, ozone concentration 40–45 mg l<sup>−1</sup>). The ozonation product was dissolved in a minimum of water, 1 ml of 60%  $\text{HClO}_4$  was added, and the mixture was evaporated in air at 20°C until brown crystals formed. The crystals were filtered off and dried in a vacuum. Complex **I** is readily soluble in water, ethanol, methanol, and acetone. Found, %: C 15.9; H 3.6; Cl 4.3; Rh 34.5.  $\text{C}_{12}\text{H}_{31}\text{ClO}_{23.5}\text{Rh}_3$ . Calculated, %: C 16.1; H 3.5; Cl 4.0; Rh 34.5.

**( $\mu_3$ -Oxo)hexakis( $\mu$ -acetato)tri( $\beta$ -picolino)tri rhodium(III) perchlorate (II).** The ozonation product of rhodium(II) acetate, obtained by the procedure described above, was dissolved in 15 ml of methanol.  $\beta$ -Picoline, 0.1 ml, and 40 mg of  $\text{NaClO}_4$  were added to the mixture, after which it was evaporated in air at 20°C until needle-like reddish brown crystals formed. The compound is readily soluble in dichloromethane and chloroform and moderately soluble in acetone and methanol. Found, %: C 34.1; H 3.7; Cl 3.6; N 4.1; Rh 29.5.  $\text{C}_{30}\text{H}_{39}\text{ClN}_3\text{O}_{17}\text{Rh}_3$ . Calculated, %: C 34.1; H 3.7; Cl 3.4; N 4.0; Rh 29.2.

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3. Glowiak, T., Kubiak, M., and Szymanska-Buzar, T., *Acta Crystallogr., Sect. B*, 1977, vol. 33, p. 1732.