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S.S. Shapovalov, O.G. Tikhonova, A.V. Kolos, A.A. Pasynskii, I.V. Skabitsky, G.L. Denisov, V.A. Grinberg

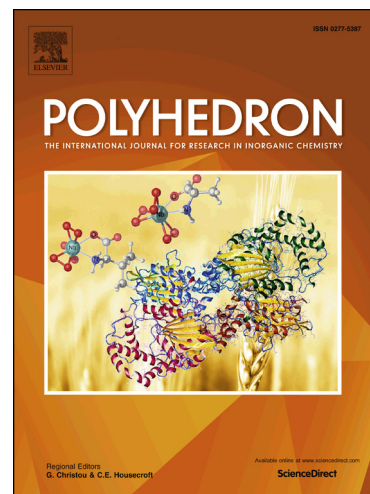
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Synthesis, structure and electrochemical properties of 1-ferrocenyl-3-cymantrenyl-containing β -diketone, pyrazole and mixed-metal chelated complex of *p*-cymene-ruthenium-iodide.

S.S.Shapovalov¹, O.G. Tikhonova¹, Kolos A.V.¹, A.A. Pasynskii¹, I.V.Skabitsky¹, G. L. Denisov², V.A.Grinberg³

1.Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Leninskii pr. 31, Moscow, 119991, Russia

2. A.N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, Vavilova st., 28, Moscow, 119991, Russia

3.Frumkin Institute of Physical Chemistry and Electrochemistry, Russian Academy of Sciences, Leninskii pr. 31, Moscow, 119991, Russia

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organometallic complex; electrochemistry; diketone; pyrazole

Abstract

Claisen condensation of acetylferrocene ($\eta^5\text{-C}_5\text{H}_5\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{COCH}_3)$) and methyl ether of cymantrenecarboxylic acid ($(\text{CO})_3\text{Mn}(\eta^5\text{-C}_5\text{H}_4\text{COOMe})$) results in β -diketone ($\eta^5\text{-C}_5\text{H}_5\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{-C(O)CH}_2\text{C(O)-}(\eta^5\text{-C}_5\text{H}_4)\text{Mn(CO)}_3$) (**I**) which reacts with hydrazine to form a heterometallic pyrazole ($\eta^5\text{-C}_5\text{H}_5\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{-N}_2\text{C}_3\text{H}_3\text{-}(\eta^5\text{-C}_5\text{H}_4)\text{Mn(CO)}_3$) (**II**) or reacts with diiodo(*p*-cymene)ruthenium(II) dimer giving chelate β -ketoenolate complexes of ruthenium, (*p*-cymene) $\text{RuI}[(\eta^5\text{-C}_5\text{H}_5\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{-C(O)CHC(O)-}(\eta^5\text{-C}_5\text{H}_4)\text{Mn(CO)}_3)]$ (**III**). The structures of complexes **I** - **III** were determined by single crystal X-ray diffraction. Electrochemical properties were studied too.

1. Introduction

β -Diketones are used as chelate ligands in homo- or heterometallic complexes of near all transition metals, *p*-block metals, and lanthanides. Such complexes are interesting as sources of volatile precursors for chemical vapor deposition (CVD) [1]. The most utilized method of β -diketone producing is a Claisen cross-condensation of enolate with the carboxylic acid ester. Preparation of β -diketones having various metal-containing substituents in a β -diketone group allows combining different metal atoms in the complexes which can later be used as precursors of catalysts or inorganic materials.

For monoferrocene-containing β -diketones known since 1958 [2, 3] a lot of chelate complexes of general formula $\text{M}(\text{ferrocenyl-}\beta\text{-diketonate})_n$ ($\text{M} = \text{VO, Mn, Co, Ni, Cu, Zn, } n = 2$; $\text{M} = \text{Al, Cr, Mn, Fe, } n = 3$) have been prepared [4]. It was noted the possibility of Fe-C bond cleavage of ferrocenylacetylacetone via photolysis in the presence of pyridine [5]. The complexes of ytterbium and lutetium $[\text{Ln}_4(\mu_3\text{-OH})_4((\eta^5\text{-C}_5\text{H}_5\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{-C(O)CHC(O)-Ph})_8)]$ have unusual cubane structure [6]. The bis-chelate ferrocenyl- β -diketonate complexes of Zr and Hf phthalocyaninate are also known [7]. The Claisen condensation has also used for the synthesis of ketoenols containing metal atoms in both side chains, in particular, of bis-ferrocenyl-ketoenol prepared by the reaction of acetylferrocene with methylferrocenylcarboxylate [8]. Further reaction with halide complexes of the transition metal M (for example, palladium, rhodium, iridium, ruthenium) produced chelate complexes $[(\eta^5\text{-C}_5\text{H}_5\text{Fe}(\eta^5\text{-C}_5\text{H}_4\text{-C(O)CHC(O)-}(\eta^5\text{-C}_5\text{H}_4)\text{Fe}(\eta^5\text{-C}_5\text{H}_5))] \text{ML}$

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