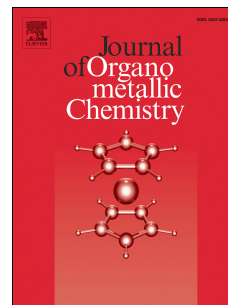


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Syntheses, characterization and reactivity of dinuclear ruthenium-nickel complexes with hexane-2,5-dione bis(thiosemicarbazonato) ligands

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Abstract

Treatment of hexane-2,5-dione bis(thiosemicarbazones) ($[\text{CH}_3\text{-C}\{\text{=N-NH-C(=S)-NHR}\}\text{-CH}_2]_2$, R = H, L^1H_2 ; CH_3 , $\text{L}^2\text{H}_2\text{-Me}$; CH_2CH_3 , $\text{L}^3\text{H}_2\text{-Et}$; C_6H_5 , $\text{L}^4\text{H}_2\text{-Ph}$) with nickel(II) acetate hydrate in refluxing ethanol gave a series of $\text{Ni}^{\text{II}}\text{N}_2\text{S}_2$ metalloligands $[\text{Ni}(\text{L-R})]$ for the generation of hetero-bimetallic complexes. The reaction of equal mole each of $[\text{Ni}(\text{L}^1)]$, $[\text{Ni}(\text{L}^2\text{-Me})]$, $[\text{Ni}(\text{L}^3\text{-Et})]$, or $[\text{Ni}(\text{L}^4\text{-Ph})]$ with $[\text{RuCl}_2(\text{dmsO})_4]$ (dmsO = dimethyl sulfoxide) at reflux resulted in isolation of neutral dinuclear ruthenium-nickel complexes $[\text{RuCl}_2\{\text{Ni}(\text{L}^1)\}(\text{dmsO})_2]$ (**1**), $[\text{RuCl}_2\{\text{Ni}(\text{L}^2\text{-Me})\}(\text{dmsO})_2]$ (**2**), $[\text{RuCl}_2\{\text{Ni}(\text{L}^3\text{-Et})\}(\text{dmsO})_2]$ (**3**), and $[\text{RuCl}_2\{\text{Ni}(\text{L}^4\text{-Ph})\}(\text{dmsO})_2]$ (**4**). Interaction of $[\text{Ni}(\text{L-R})]$ with $[\text{CpRu}(\text{PPh}_3)_2\text{Cl}]$ (Cp^- = cyclopentadienyl) at room temperature led to formation of cationic dinuclear organoruthenium-nickel complexes $[\text{CpRu}\{\text{Ni}(\text{L}^1)\}(\text{PPh}_3)]\text{Cl}$ (**5**), $[\text{CpRu}\{\text{Ni}(\text{L}^2\text{-Me})\}(\text{PPh}_3)]\text{Cl}$ (**6**), $[\text{CpRu}\{\text{Ni}(\text{L}^3\text{-Et})\}(\text{PPh}_3)]\text{Cl}$ (**7**), and $[\text{CpRu}\{\text{Ni}(\text{L}^4\text{-Ph})\}(\text{PPh}_3)]\text{Cl}$ (**8**). New bimetallic ruthenium-nickel complexes **1–8** have been characterized spectroscopically, of which molecular structures of three complexes $[\text{RuCl}_2\{\text{Ni}(\text{L}^2\text{-Me})\}(\text{dmsO})_2]\cdot\text{CH}_2\text{Cl}_2$ (**2**· CH_2Cl_2), $[\text{CpRu}\{\text{Ni}(\text{L}^2\text{-Me})\}(\text{PPh}_3)]\text{Cl}\cdot\text{EtOH}$ (**6**· EtOH), and $[\text{CpRu}\{\text{Ni}(\text{L}^3\text{-Et})\}(\text{PPh}_3)]\text{Cl}$ (**7**· H_2O) have been established by single-crystal X-ray crystallography. Their catalytic activities for the acetalation of benzaldehyde in the presence of molecular H_2 have been also investigated in this paper.

Keywords: $\{\text{Ni}(\mu\text{-S})_2\text{Ru}\}$ -type complex; Bis(thiosemicarbazones); Metalloligand; X-Ray crystal structure; Catalytic activity

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