

Dinuclear complexes of copper(I) with crown ether-containing N-thiophosphorylated bis-thioureas and 2,2'-bipyridine or 1,10-phenanthroline: Synthesis, characterization, and picrate extraction properties

Babashkina M., Shakirova E., Safin D., Sokolov F., Klein A., Szyrwił Ł., Kubiak M., Kozłowski H., Krivolapov D.

Kazan Federal University, 420008, Kremlevskaya 18, Kazan, Russia

Abstract

Reaction of O,O'-diisopropylthiophosphoric acid isothiocyanate (iPrO) $2P(S)NCS$ with 1,10-diaza-18-crown-6, 1,7-diaza-18-crown-6, or 1,7-diaza-15-crown-5 leads to the N-thiophosphorylated bis-thioureas N,N'-bis[C(S)NHP(S)(OiPr) $_2$]-1,10-diaza-18-crown-6 (H $_2$ LI), N,N'-bis[C(S)NHP(S)(OiPr) $_2$]-1,7-diaza-18-crown-6 (H $_2$ LII) and N,N'-bis[C(S)NHP(S)(OiPr) $_2$]-1,7-diaza-15-crown-5 (H $_2$ LIII). Reaction of the potassium salts of H $_2$ LI-III with a mixture of CuI and 2,2'-bipyridine (bpy) or 1,10-phenanthroline (phen) in aqueous EtOH/CH $_2$ Cl $_2$ leads to the dinuclear complexes [Cu $_2$ (bpy) $_2$ LI-III] and [Cu $_2$ (phen) $_2$ LI-III]. The structures of these compounds were investigated by 1H , $^{31}P\{^1H\}$ NMR spectroscopy, and elemental analysis. The crystal structures of H $_2$ LI and [Cu $_2$ (phen) $_2$ LI] were determined by single-crystal X-ray diffraction. Extraction capacities of the obtained compounds in comparison to the related compounds 1,10-diaza-18-crown-6, N,N'-bis[C(=CMe $_2$)CH $_2$ P(O)(OiPr) $_2$]-1,10-diaza-18-crown-6, N,N'-bis[C(S)NHP(O)(OiPr) $_2$]-1,10-diaza-18-crown-6 towards the picrate salts LiPic, NaPic, KPic, and NH $_4$ Pic were also studied. Copyright © 2010 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.

<http://dx.doi.org/10.1002/zaac.201000263>

Keywords

1-10-Phenanthroline, 2-2'-Bipyridine, Copper, N-Thiophosphoryl bis-thiourea, X-ray diffraction