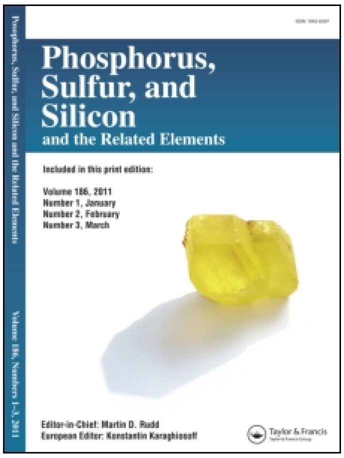

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Synthesis, Metallation and Complexes of Annulated 1H-1,3-Azaphospholes

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Synthesis, Metallation and Complexes of Annulated 1H-1,3-Azaphospholes

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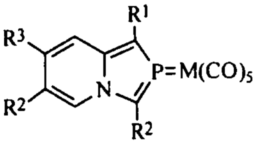
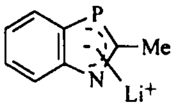
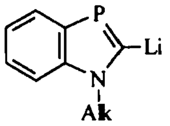
We studied the complex chemical behavior of neutral and anionic annulated 1H-1,3-azaphospholes. 2-Phosphaindolizines, synthesized from pyridinium salts and PCl_3 / Et_3N, reacted with $(CO)_5M(THF)$ (M = Cr, Mo, W) yielding $(\eta^1$-phosphaindolizine$)M(CO)_5$ complexes. Comparison of ^{31}P-, ^{1}H and ^{13}C-NMR data of free ligands and complexes allows to characterize donor and acceptor properties of this ligand type. According to ^{13}C coordination shifts, π-back donation has a rather local effect. An X-ray structure analysis of a $LCr(CO)_5$ complex and of the respective ligand L shows a more effective delocalization with nearly equal P-C bond lengths in the complex-bound ligand.







Attempts to make $(\eta^5$-phosphaindolizine$)M(CO)_3$ complexes by reaction with norbornadiene-$Cr(CO)_4$, mes$W(CO)_3$ etc. failed. Instead, a variety of σ complexes $LM(CO)_5$, *cis*-$L_2Cr(CO)_4$, *fac*-$L_3M(CO)_3$ (M = Mo, W) and *mer*-$L_3Mo(CO)_3$, were formed. A convenient 3 step synthesis of 1H-1,3-benzazaphospholes is presented, including a new type of $LiAlH_4$-mediated reductive ring closure of 2-hydroxyarylphosphonic acid esters. Metallation of 1,3-benzazaphospholes and NMR-spectra are studied.

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