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

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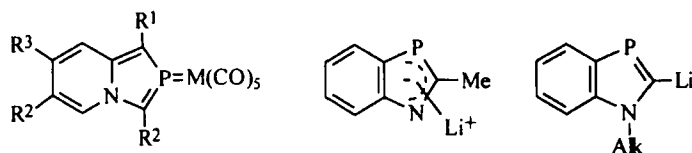
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Synthesis, Metallation and Complexes of Annulated 1H-1,3-Azaphospholes

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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 $(\text{CO})_5\text{M}(\text{THF})$ ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$) yielding $(\eta^1\text{-phosphaindolizine})\text{M}(\text{CO})_5$ complexes. Comparison of ^{31}P -, ^1H 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 $\text{LCr}(\text{CO})_5$ 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 $(\eta^5\text{-phosphaindolizine})\text{M}(\text{CO})_3$ complexes by reaction with norbornadiene- $\text{Cr}(\text{CO})_4$, $\text{mesW}(\text{CO})_3$ etc. failed. Instead, a variety of σ complexes $\text{LM}(\text{CO})_3$, $\text{cis-L}_2\text{Cr}(\text{CO})_4$, $\text{fac-L}_3\text{M}(\text{CO})_3$ ($\text{M} = \text{Mo}, \text{W}$) and $\text{mer-L}_3\text{Mo}(\text{CO})_3$ 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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