

3-Hexyne Complexes of Molybdenum(II) and Tungsten(II). Crystal Structures of $[\text{WI}_2(\text{CO})(\text{PPh}_3)_2(\eta^2\text{-EtC}_2\text{Et})]$ and $[\text{WI}_2(\text{CO})\{\text{Ph}_2\text{P}(\text{CH}_2)_3\text{PPh}_2\}(\eta^2\text{-EtC}_2\text{Et})]$

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3-Hexyne Complexes, Molybdenum(II), Tungsten(II)

Treatment of $[\text{MI}_2(\text{CO})_3(\text{NCMe})_2]$ ($\text{M} = \text{Mo}, \text{W}$) with two equivalents of EtC_2Et in CH_2Cl_2 at 0°C yields the bis(3-hexyne) complexes $[\text{MI}_2(\text{CO})(\text{NCMe})(\eta^2\text{-EtC}_2\text{Et})_2]$ (**1** and **2**). Complexes **1** and **2** react with two equivalents of PPh_3 in CH_2Cl_2 to give the complexes $[\text{MI}_2(\text{CO})(\text{PPh}_3)_2(\eta^2\text{-EtC}_2\text{Et})]$ (**3** and **4**). The molecular structure of **4** ($\text{M} = \text{W}$) has been crystallographically determined and has a *pseudo*-octahedral geometry with the two PPh_3 ligands *trans* to each other with the two *cis*-iodo-ligands, which together with the carbonyl and 3-hexyne ligand are occupying the equatorial plane. Reaction of **1** and **2** with an equimolar amount of $\text{Ph}_2\text{P}(\text{CH}_2)_n\text{PPh}_2$ (for $\text{M} = \text{Mo}$, $n = 1$; for $\text{M} = \text{W}$, $n = 1$ to 6) in CH_2Cl_2 affords the mono(3-hexyne) complexes $[\text{MI}_2(\text{CO})\{\text{Ph}_2\text{P}(\text{CH}_2)_n\text{PPh}_2\}(\eta^2\text{-EtC}_2\text{Et})]$ (**5** - **11**). The tungsten complex $[\text{WI}_2(\text{CO})\{\text{Ph}_2\text{P}(\text{CH}_2)_3\text{PPh}_2\}(\eta^2\text{-EtC}_2\text{Et})]$ (**8**) has also been crystallographically characterised and has *cis*-phosphorus atoms in the equatorial plane with the carbonyl and one of the iodo-ligands, with the other iodo-ligand and the 3-hexyne occupying the axial sites.

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