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Carbene complexes: XXII. Preparation, properties and structures of the N,N-functionalized bis(amino)carbenemolybdenum(O) carbonyls $[\text{Mo}(\text{CO})_4 \{$

$\text{CN}(\text{CH}_2\text{CH}:\text{CH}_2)(\text{CH}_2)_2\text{N}\}(\text{CH}_2\text{CH}:\text{CH}_2)\}]]$ and 

$\text{Mo}(\text{CO})_3 \{ \text{CN}[(\text{CH}_2)_3\text{PPh}_2] (\text{CH}_2)_2\text{N} \{ (\text{CH}_2)_3\text{PPh}_2 \}_n \} \star$

Dedicated to Professor G.P. Chiusoli, a respected colleague, in recognition of his many significant contributions to organometallic chemistry, on the occasion of his 70th birthday.



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Abstract

Treatment of 1,2-bis(γ -diphenylphosphinopropylamino)ethane [obtained from 1,2-bis(allylamino)ethane and 2PPh₂H and CH(NMe₂)(OMe)₂] yielded the electron-rich enetetramine  $\text{CN}(\text{R})(\text{CH}_2)_2\text{NR}]_2 [\text{R} \square \text{CH}_2)_3\text{PPh}_2]$ [abbreviated as $\text{L}_2^{(\text{CH}_2)_3\text{PPh}_2}$]. Although L_2^{All} , the analogue in which $\text{R} \square \text{CH}_2\text{CH}:\text{CH}_2$, is unknown, the carbenemolybdenum title compounds $[\text{Mo}(\text{CO})_4(\text{L}^{\text{All}})]$ (**3**) and  $\text{Mo}(\text{CO})_3 \{ \text{L}^{(\text{CH}_2)_3\text{PPh}_2} \}_n$ (**4**) have been prepared from $[\text{Mo}(\text{CO})_6]$: for **3** from CH(NMe₂)(OMe)₂ + [All(H)N(CH₂)₂]₂ and for **4** from $\text{L}_2^{(\text{CH}_2)_3\text{PPh}_2}$. In **3** only one of the R groups behaves in a ligating mode. L^{All} being a chelating ligand, **3** is thus a rare example of a metal complex having both carbene- and η^2 -alkene-ligation and the first for molybdenum. The presumed polymeric structure of **4** is believed to arise from one of the P^{III} sites behaving in a chelating fashion with the other in a bridging mode. Treatment of **3** with ¹³C¹⁸O stereoselectively gave the isotopomer in which a CO *trans* to a CO was displaced, the *trans*

effect thus being parallel to the *trans* influence (X-ray data); a coproduct was *cis*-[Mo(CO)₄(¹³CO)(L^{All})]. Similarly, *cis*-[Mo(CO)₄(L^{All})(PEt₃)] was the sole product of reaction between **3** and PEt₃, which upon irradiation yielded *fac*- together with *mer*-[Mo(CO)₃(L^{All})(PEt₃)]. Thermolysis of **3** afforded AllC: N(CH₂)₂NAll as the only organic product. The X-ray structure of **3** showed that the mutually *trans* Mo□CO bonds are significantly longer [av. 2.037(5) Å] than Mo□CO *trans* to carbene [1.984(4) Å] or alkene [1.975(5) Å], with Mo□C_{carb} at 2.229(4) Å.

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