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Nucleophilic attack by isocyanides, phosphines and cyclohexenesulphide on the α-carbon of "side-on" bonded μ-$\sigma:\eta^2$-(4e)-vinylidenes; formation of thioketene and thioaldehyde dimolybdenum complexes

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Abstract

Reaction of $[Mo_2\{\mu\text{-}\sigma:\eta^2\text{-(4e)-}C=CR_1R_2\}(CO)_4(\eta\text{-}C_5H_5)_2]$ ($R_1 = R_2 = H$ or Me) with ^tBuNC, PMe_3 or $P(OMe)_3$ leads, surprisingly, to attack on the C_α or carbenoid carbon of the vinylidene moiety. A similar reaction with cyclohexenesulphide results in transfer of a sulphur atom to C_α and formation of a dinuclear thioketene complex, which *via* successive protonation and addition of H^- gives a μ-thioacetaldehyde complex.

Vinylidene is an important ligand in transition metal chemistry [1], interest in it being stimulated by the suggestion that such species are present on the surface of Fischer–Tropsch catalysts. Recently [2] we have developed a rational and convenient synthesis of an unusual group of dinuclear complexes carrying the first examples of "side-on" bonded vinylidenes, that is systems with μ-$\sigma:\eta^2$-(4e)-$C{=}CR_1R_2$ ligands. The reactivity of these species is clearly of interest, and in initial studies [3] we have established that protonation occurs selectively on the α or carbenoid carbon. It was obviously important to also examine the site of nucleophilic attack, and we report here that isocyanides, phosphines, phosphites and the sulphur-atom donor cyclohexenesulphide all attack the α-carbon of μ-$\sigma:\eta^2$-(4e)-vinylidenes.

In the expectation of substituting a terminal carbonyl ligand we treated the complex $[Mo_2\{\mu\text{-}\sigma:\eta^2\text{-(4e)-}C=CH_2\}(CO)_4(\eta\text{-}C_5H_5)_2]$ [1] (Scheme 1) with ^tBuNC. In toluene as solvent at room temperature there was a rapid change in colour from purple to red, and (73% yield) a red crystalline 1:1 adduct III, was formed, the

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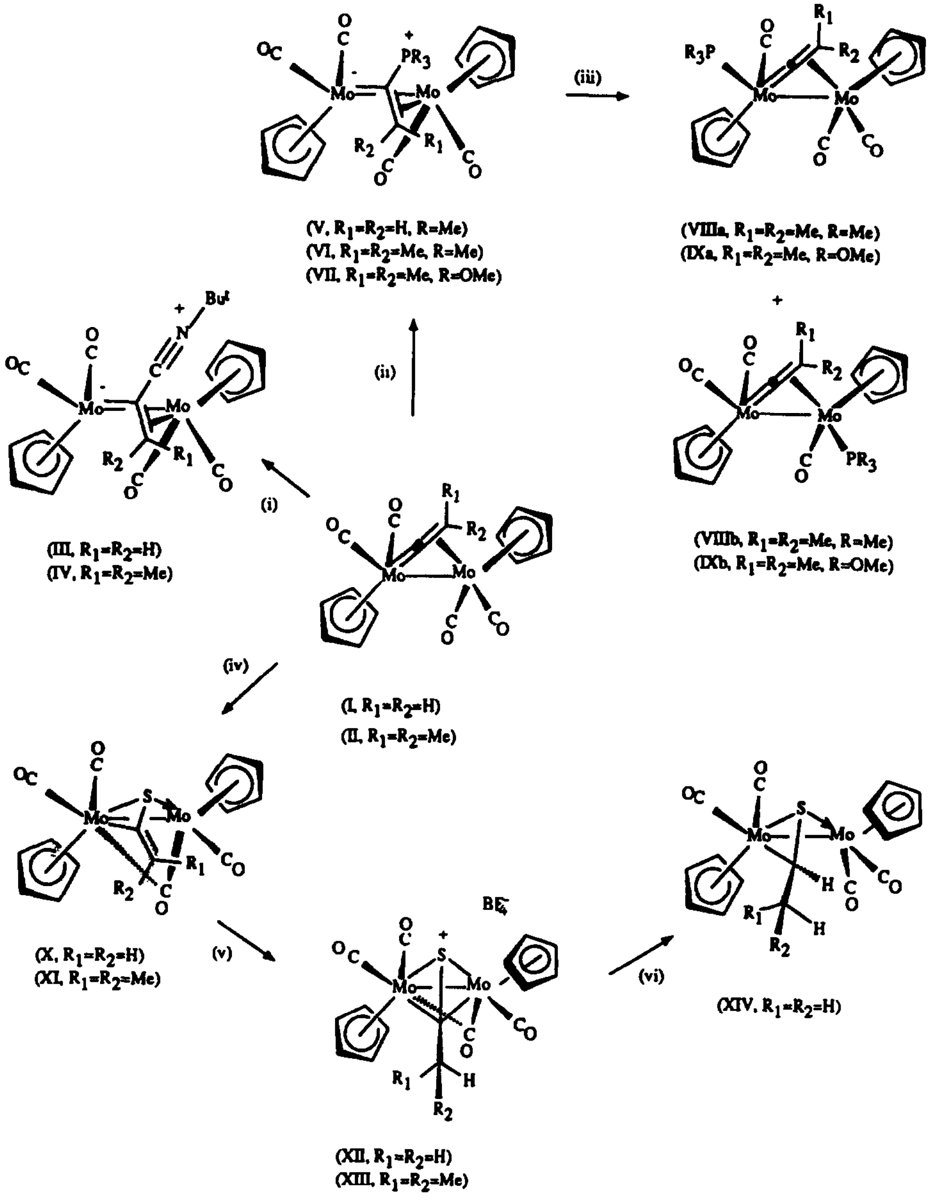

Scheme 1. (i) + ^{t}BuNC/toluene/room temperature; (ii) + PMe_3 or $P(OMe)_3$/toluene/room temperature; (iii) UV/THF/room temperature; (iv) cyclohexenesulphide/toluene/room temperature; (v) HBF_4Et_2O/CH_2Cl_2/ − 78°C; (vi) Li[$BHEt_3$]/THF, − 78°C.

adduct was soluble in polar solvents and insoluble in non-polar solvents. A similar reaction occurred between II and ^{t}BuNC to give IV. Inspection of the spectroscopic data [4*] for III and IV suggested, however, that the expected substitution

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* Reference number with asterisk indicates a note in the list of references.

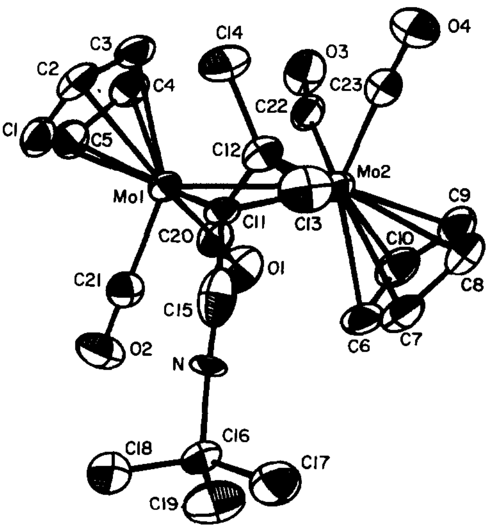

Fig. 1. Molecular structure of IV. All hydrogen atoms have been omitted for clarity. Selected bond lengths (Å) and angles (deg): Mo(1)–Mo(2) 3.0661(5), Mo(1)–C(11) 2.189(3), Mo(2)–C(11) 2.193(3), Mo(2)–C(12) 2.356(4), C(11)–C(12) 1.455(5), C(11)–C(15) 1.399(5), N–C(15) 1.153(4); N–C(15)–C(11) 176.3(4), N–C(16)–C(17) 107.4(3), C(14)–C(12)–C(13) 111.2(3).

reaction had not occurred. The low field ^{13}C resonance due to the C_α or carbenoid carbon of a μ-σ : η^2-(4e)-vinylidene was absent, and in addition the spectra of both adducts exhibited *four* carbon resonances assignable to the contact carbons of terminally bonded CO ligands. In order to establish the nature of this reaction a single crystal X-ray diffraction study [5*] of IV was undertaken, and this revealed the structure shown in Fig. 1.

The ^{t}BuNC has attacked the α-carbon of the vinylidene moiety to form a dinuclear complex, in which each molybdenum atom is coordinated to a η^5-C_5H_5 and two terminal carbonyl ligands, the cyclopentadienyl ligands being orientated mutually *trans*, as in the parent complex II. The Mo–Mo single bond [3.0661(4) Å] is bridged by a μ-vinyl group [Mo(1)–C(11) 2.189(3); Mo(2)–C(11) 2.193(3), Mo(2)–C(12) 2.356(4) Å] formed from the $C=CMe_2$ fragment and ^{t}BuNC, the three core atoms of the isocynanide adopting an almost linear [C(15)–N–C(16) 177.3(4)°] geometry in the adduct. The positive charge assumed to be present on the nitrogen atom is compensated for by a negative charge on Mo(1), the presence of which is indicated by the observation that the Mo–C bond lengths of the terminal carbonyl ligands coordinated to Mo(1) are shorter than the corresponding Mo(2)–C distances.

Similar nucleophilic addition reactions took place between the vinylidenes I and II, and trimethylphosphine or trimethylphosphite. Reaction occurred rapidly at room temperature (toluene) to give excellent yields (85%) of the zwitterionic 1 : 1 adducts V–VII [4*]. Again single crystal X-ray crystallography on V confirmed [5*] (see Fig. 2) that trimethylphosphine selectively attacks C_α of the vinylidene fragment to form a complex with very similar structural features to those exhibited by the isocycanide adduct IV.

These observations are particularly interesting in the context of previous studies of nucleophilic attack on co-ordinated vinylidenes, in which it was found that

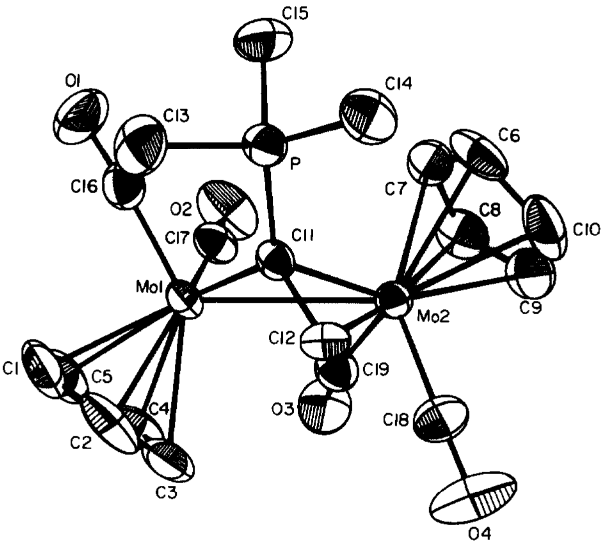

Fig 2. Molecular structure of V. All hydrogen atoms have been omitted for clarity. Selected bond lengths (Å) and angles (deg): Mo(1)–Mo(2) 3.0499(5), Mo(1)–C(11) 2.168(4), Mo(2)–C(11) 2.182(4), Mo(2)–C(12) 2.297(4), C(11)–C(12) 1.449(5), C(11)–P 1.759(4); Mo(1)–C(11)–C(12) 117.0(3), Mo(1)–C(11)–P 124.4(2).

mononuclear Mn [6] and Fe [7] complexes also react with phosphines on the α-carbon, whereas the trinuclear complex [FeCo$_2${μ-σ,σ : η^2-(4e)-C=CH$_2$}(CO)$_9$] reacts under kinetic control with PMe$_3$ at the β-carbon [8].

In the case of the trinuclear FeCo$_2$ system it was also reported [8] that on thermolysis the PMe$_3$ ligand migrated from the β-carbon on to one of the cobalt centres. In contrast, the dinuclear adducts (V–VII) were found to be thermally stable, but on UV irradiation a molybdenum to carbon double bond was re-established and the phosphorus ligand migrated on to either of the two molybdenum centres to give the purple crystalline complexes VIIIa/VIIIb [4*] (3 : 1) and IXa/IXb [4*] (3 : 1) with loss of carbon monoxide. The intramolecular nature of this reaction was indicated by the observation that irradiation of a THF solution of VII containing an excess of PMe$_3$ resulted in the formation of only IXa and IXb, *i.e.* the trimethylphosphite adducts.

These findings suggested that it may also be possible to carry out atom transfer reactions with dinuclear "side-on" bonded vinylidenes by treating them with suitable nucleophiles. This possibility was confirmed when it was found that both I and II reacted with cyclohexene sulphide. A sulphur atom is transferred to the α-carbon, to give the thioketene complexes X and XI [4*], which were isolated in good yield (60%) as dark-red and black crystalline materials, respectively.

Although mononuclear thioketene complexes have been synthesised previously, they were formed either directly from thioketenes carrying bulky substituents [9], or in a few cases by addition of S$_8$ to a rhodium-vinylidene [10]. Little is known about dinuclear systems, and in particular their reaction chemistry is unexplored. Addition of HBF$_4$Et$_2$O to either X or XI resulted in a rapid reaction at −78°C and formation of the orange crystalline μ-thioacylium cations XII and XIII [4*].

We suggest that these complexes are isostructural with the oxygen analogues $[Mo_2(\mu\text{-}MeCO)(CO)_4(\eta\text{-}C_5H_5)_2][BF_4]$ prepared by Beck and coworkers and characterised by X-ray crystallography [11]. These Mo_2 μ-thioacylium cations are potential sources of μ-thioaldehyde complexes, and this was confirmed by the regio-specific delivery of H^- to carbon and formation (70%) of the stable black crystalline μ-thioacetaldehyde complex XIV [4*]. In principle a range of μ-thioaldehyde and μ-thioketone complexes should be accessible by this approach. Previously such complexes have been prepared by reaction of $[W_2(\mu\text{-}S)(CO)_6(\eta\text{-}C_5H_5)_2]$ with CH_2N_2 [12] or by treatment of $[Mo_2(CO)_4(\eta\text{-}C_5H_5)_2]$ with thioketones carrying bulky substituents [13]. Obviously, our new methodology has far greater potential scope.

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4 Selected spectroscopic data for III: ^{1}H NMR (CD_2Cl_2). δ 5.12 (s, 5H, C_5H_5), 5.02 (s, 5H, C_5H_5), 3.71 [d, 1H, =CH'H, *J*(HH) 1.12 Hz], 1.63 (s, 9H, CMe_3), 1 42 ppm [d, 1H, =CH'H, *J*(HH) 1.10 Hz] ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 252.00 (CO), 242.25 (CO), 235.47 (CO), 233 08 (CO), 122.00 (^{t}BuN*C*CCH$_2$), 94.16 (C_5H_5), 92.95 (C_5H_5), 78.33 (^{t}BuNC*C*CH$_2$), 59.60 (*C*Me$_3$), 56.94 (^{t}BuNCC*C*H$_2$), 30.94 ppm (C*Me*$_3$). IR: ν(NC) (CH_2Cl_2) 2197, ν(CO) 1920 w, 1891 s, 1818 m, 1748 w cm^{-1}

Compound IV: ^{1}H NMR (CD_2Cl_2) δ 5.14 (s, 5H, C_5H_5), 5.12 (s, 5H, C_5H_5), 1.89 (s, 3H, =CMe'Me), 1.64 (s, 9H, CMe_3), 1.33 ppm (s, 3H, =CMe'Me). ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 250 19 (CO), 241.96 (CO), 236.21 (CO), 233.19 (CO), 121.61 (^{t}BuN*C*CCMe$_2$), 95.33 (C_5H_5), 93.22 (C_5H_5), 88.59 (^{t}BuNCC*C*Me$_2$), 85.48 (^{t}BuNC*C*CMe$_2$), 59.28 (*C*Me$_3$), 35.56 (=C*Me'*Me), 31.58 (=CMe'*Me*), 30.96 ppm (C*Me*$_3$). IR: ν(NC) (CH_2Cl_2) 2153, ν(CO) 1916 w, 1886 s, 1817 m, 1798 w cm^{-1}.

Compound V: ^{1}H NMR (CD_2Cl_2)· δ 5.19 (s, 5H, C_5H_5), 4.95 (s, 5H, C_5H_5), 3.20 [dd, 1H, =CH'H, *J*(HP) 24.8, *J*(HH) 1.6 Hz], 1.81 [d, 9H, PMe$_3$, *J*(HP) 9.5 Hz], 1.40 ppm [dd, 1H, =CH'H, *J*(HP) 27.7, *J*(HH) 1.6 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 255.4 [d, CO, *J*(CP) 10.3 Hz], 242.2 (CO), 237.6 (CO), 234.6 [d, CO, *J*(CP) 1 9 Hz], 112.3 [d, *C*=CH$_2$, *J*(CP) 31.7 Hz], 92.6 (C_5H_5), 91.2 (C_5H_5), 55.5 [d, C=*C*H$_2$, *J*(CP) 9.3 Hz], 16.5 ppm (P*Me*$_3$). IR: ν(CO) (CH_2Cl_2) 1911 m, 1877 s, 1798 m, 1780 m cm^{-1}.

Compound VI: ^{1}H NMR (CD_2Cl_2)· δ 5.08 (s, 5H, C_5H_5), 4.97 [d, 5H, C_5H_5, *J*(HP) 2.02 Hz], 2.34 [d, 3H, =CMe'Me, *J*(HP) 2.38 Hz], 1.63 ppm [d, 9H, P*Me*$_3$, *J*(HP) 9.65 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 259 60 [d, CO, *J*(CP) 24.62 Hz], 246.81 (CO), 244 35 (CO), 230.80 [d, CO, *J*(CP) 7.0 Hz], 229.64 [d, *C*=CMe$_2$, *J*(CP) 36.8 Hz], 152.10 [d, C=*C*Me$_2$, *J*(CP) 3.35 Hz], 94 28 (C_5H_5), 93 50 (C_5H_5), 31.25 (=*C*Me'Me), 31.12 (=CMe'Me), 17.55 ppm [d, P*Me*$_3$, *J*(CP) 32.8 Hz]. IR: ν(CO) (CH_2Cl_2) 1911 m, 1883 s, 1834 m, 1756 w cm^{-1}.

Compound VII: ^{1}H NMR (CD_2Cl_2), δ 5.15 (s, 5H, C_5H_5), 5.07 [d, 5H, C_5H_5, *J*(HP) 1 71 Hz], 3.79 [d, 9H, P(OMe)$_3$, *J*(HP) 11.3 Hz], 2.34 [d, 3H, =CMe'Me, *J*(HP) 2.34 Hz], 2.33 ppm [d, 3H, =CMe'Me, *J*(HP) 2.55 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 252.50 [d, CO, *J*(CP) 31.8 Hz], 246.21 (CO), 244.26 (CO), 231 10 [d, CO, *J*(CP) 6.9 Hz], 225.47 [d, *C*=CMe$_2$, *J*(CP) 50.61 Hz], 152.96 [d,

C=*C*Me$_2$, *J*(CP) 3.94 Hz], 94.54 (C_5H_5), 93.55 (C_5H_5), 53.95 [d, P(OMe)$_3$, *J*(CP) 27.15 Hz], 31.02 ppm (=C*Me*$_2$). IR: ν(CO) (CH_2Cl_2) 1893 br, 1827 m, 1785 w cm^{-1}.

Compound VIIIa: ^{1}H NMR (CD_2Cl_2): δ 5.44 [d, 5H, C_5H_5, *J*(HP) 1.77 Hz], 5.05 (s, 5H, C_5H_5), 1.68 (s, 3H, =CMe′Me), 1.63 (s, 3H, =CMe′Me), 1.59 ppm [d, 9H, P*Me*$_3$, *J*(HP) 9.06 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2). δ 298.72 [d, Mo=*C*, *J*(CP) 34.16 Hz], 249.06 (CO), 248.69 [d, CO, *J*(CP) 15.42 Hz], 236.74 [d, CO, *J*(CP) 4.67 Hz], 95.47 (C_5H_5), 92.93 (C_5H_5), 68.04 (C=*C*Me$_2$), 31.02 ppm [d, =CMe′Me, *J*(CP) 30.68 Hz].

Compound VIIIb: ^{1}H NMR (CD_2Cl_2): δ 5.40 [d, 5H, C_5H_5, *J*(HP) 1.94 Hz], 5.26 (s, 5H, C_5H_5), 1.67 (s, 3H, =CMe^{1}Me^2), 1.62 (s, 3H, =CMe^{1}Me^2), 1.58 ppm [d, 9H, P*Me*$_3$, *J*(HP) 9.10 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 305.10 [d, Mo=C, *J*(CP) 24.2 Hz], 246.75 (CO), 246.70 (CO), 236.26 (CO), 94.02 (C_5H_5), 93.20 (C_5H_5), 30.88 [d, =CMe^{1}Me^2, *J*(CP) 2.27 Hz], 30.24 (s, =CMe^{1}Me^2), 25.83 ppm [d, P*Me*$_3$, *J*(CP) 30.92 Hz].

Compound IXa: ^{1}H NMR (CD_2Cl_2): δ 5.50 [d, 5H, C_5H_5, *J*(HP) 1.53 Hz], 5.09 (s, 5H, C_5H_5), 3.59 [d, 9H, P(OMe)$_3$, *J*(HP) 11.77 Hz], 1.64 [d, 3H, =CMe^{1}Me^2, *J*(HP) 1.88 Hz], 1.50 ppm [d, 3H, =CMe^{1}Me^2, *J*(HP) 1.85 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2). δ 300.03 [d, Mo=*C*, *J*(CP) 48.7 Hz], 246.79 (CO), 241.56 [d, CO, *J*(CP) 21.95 Hz], 235.35 [d, CO, *J*(CP) 4.98 Hz], 94.03 (C_5H_5), 91.57 (C_5H_5), 67.25 [d, C=*C*Me$_2$, *J*(CP) 3.44 Hz], 52.3 [d, P(OMe)$_3$, *J*(CP) 2.28 Hz], 28.72 (=C*Me*^{1}Me^2), 28.43 ppm (=CMe^1*Me*^{2}).

Compound IXb: ^{1}H NMR (CD_2Cl_2): δ 5.37 [d, 5H, C_5H_5, *J*(HP) 1.39 Hz], 5.26 (s, 5H, C_5H_5), 3.57 [d, 9H, P(OMe)$_3$, *J*(HP) 12.02 Hz], 1.61 [d, 3H, =CMe^{1}Me^2, *J*(HP) 0.80 Hz], 1.52 ppm [d, 3H, =CMe^{1}Me^2, *J*(HP) 0.73 Hz]. ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 310.80 [d, Mo=C, *J*(CP) 38.11 Hz], 246.42 (CO), 238.52 [d, CO, J(CP) 21.61 Hz], 232.72 (CO), 93.96 (C_5H_5), 92.08 (C_5H_5), 69.57 (C=*C*Me$_2$), 51.90 [P(OMe)$_3$], 28.82 (=CMe^{1}Me^2), 28.40 ppm (=CMe^{1}Me^2).

Compound X: ^{1}H NMR ($CDCl_3$), δ 5.82 [d, 1H, =CH^{1}H^2, *J*(HH) 2.5 Hz], 5.48 (s, 5H, C_5H_5), 5.20 [d, 1H, =CH^{1}H^2, *J*(HH) 2.5 Hz], 5.01 ppm (s, 5H, C_5H_5). ^{13}C-{^{1}H} NMR ($CDCl_3$): δ 245.7 (CO), 237.4 (CO), 235.4 (CO), 235.1 (CO), 185.6 (S*C*=CH$_2$), 107.7 (C=*C*H$_2$), 94.1 (C_5H_5), 94.0 ppm (C_5H_5). IR: ν(CO) (hexane) 1969 w, 1946 s, 1881 m, 1837 w cm^{-1}

Compound XI: ^{1}H NMR ($CDCl_3$): δ 5.43 (s, 5H, C_5H_5), 5.04 (s, 5H, C_5H_5), 2.28 (s, 3H, =CMe^{1}Me^2), 1.86 ppm (s, 3H, =CMe^{1}Me^2). ^{13}C-{^{1}H} NMR (CD_2Cl_2): δ 246.3 (CO), 237.8 (CO), 232.1 (CO), 171.5 (*C*=CMe$_2$), 131.3 (C=*C*Me$_2$), 93.7 (C_5H_5), 93.1 (C_5H_5), 31.6 (=CMe^{1}Me^2), 22.1 ppm (=CMe^{1}Me^2).

Compound XII: ^{1}H NMR ($CDCl_3$): δ 5.73 (s, 10H, C_5H_5), 3.05 ppm (s, 3H, Me). ^{13}C-{^{1}H} NMR ($CDCl_3$): δ 222.8 (CO), 222.0 (CO), 120.8 (S*C*Me), 96.0 (C_5H_5), 33.1 ppm (Me). IR ν(CO) (CH_2Cl_2) 2061 m, 2012 s, 1919 m cm^{-1}.

Compound XIII: ^{1}H NMR (acetone-d_6): δ 6.00 (s, 10H, C_5H_5), 3.48 [heptet, 1H, C*H*, *J*(HH) 6.7 Hz], 1.39 ppm [d, 6H, Me, *J*(HH) 6.7 Hz]. ^{13}C-{^{1}H} NMR (acetone-d_6): δ 220.0 (CO), 221.5 (CO), 135.5 (S*C*^{i}Pr), 94.9 (C_5H_5), 43.6 (*C*H), 26.6 ppm (Me). IR: ν(CO) (CH_2Cl_2) 2061 m, 2009 s, 1917 m cm^{-1}.

Compound XIV: ^{1}H NMR ($CDCl_3$): δ 5.43 (s, 5H, C_5H_5), 5.10 (s, 5H, C_5H_5), 1.92 [q, 1H, C*H*Me, *J*(HH) 3.0 Hz], 1.84 ppm [d, 3H, Me, *J*(HH) 3.0 Hz]. ^{13}C-{^{1}H} NMR ($CDCl_3$): δ 245.7 (CO), 235.9 (CO), 235.4 (CO), 230.7 (CO), 93.6 (C_5H_5), 92.6 (C_5H_5), 81.1 [*C*H(Me)], 31.4 ppm (Me). IR: ν(CO) (hexane) 1958 w, 1931 s, 1877 m, 1831 w cm^{-1}.

5 Crystal data for IV $C_{23}H_{25}Mo_2NO_4$, $M = 571.34$, monoclinic, space group $P2_1/n$, $a = 8.140(1)$, $b = 13.148(1)$, $c = 21.158(4)$ Å, $\beta = 96.03(1)°$, $U = 2251.9$ Å^3, $Z = 4$, $D_c = 1.69$ g cm^{-3}, μ(Mo-K_α) = 11.1 cm^{-1}, $T = 293$ K

V: $C_{19}H_{21}Mo_2O_4P$, $M = 536.23$, monoclinic, space group $P2_1/n$, $a = 8.316(2)$, $b = 15.522(2)$, $c = 15.719(4)$ Å, $\beta = 97.23(1)°$, $U = 2012.9$ Å^3, $Z = 4$, $D_c = 1.77$ g cm^{-3}, μ(Mo-K_α) = 13.2 cm^{-1}.

In both cases the positions of the hydrogen atoms were calculated according to ideal geometry (C–H distance 0.95 Å) and refined as riding atoms, otherwise all atoms were directly located and refined. Final residues R were 0.026 ($R_w = 0.032$) and 0.027 ($R_w = 0.030$) for 3217 and 2992 unique, observed [$F_o > 3\sigma(F_o)$] intensity data respectively.

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