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Synthesis of Group 6 metal carbonyl complexes with iminophosphine ligands: crystal structure of $[Mo(CO)_4(o-Ph_2PC_6H_4-CH=NMe)]$

Gregorio Sánchez^{a,*}, José L. Serrano^b, Carmen M. López^a, Joaquín García^a, José Pérez^a, Gregorio López^a

^a Departamento de Química Inorgánica, Universidad de Murcia, 30071 Murcia, Spain

^b Departamento de Ingeniería Minera, Geológica y Cartográfica. Área de Química Inorgánica, Universidad Politécnica de Cartagena,

30203 Cartagena, Spain

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Abstract

The synthesis of new molybdenum and tungsten carbonyl complexes with the mixed donor bidentate ligands $o-Ph_2PC_6H_4-CH=NR$ is described. Two series of complexes $[M(CO)_4(o-Ph_2PC_6H_4-CH=NR)]$ (M = Mo; R = Me (**1a**), Et (**2a**), ⁱPr (**3a**), ⁱBu (**4a**), NH-Me (**5a**). M = W; R = Me (**1b**), Et (**2b**), ⁱPr (**3b**), ⁱBu (**4b**), NH-Me (**5b**)) have been prepared by direct reaction of the ligands with the corresponding precursors *cis*- $[M(CO)_4(pip)_2]$ (M = Mo or W; pip = piperidine NHC₅H₁₀). The new complexes were characterized by partial elemental analyses and spectroscopic methods (IR, ¹H, ¹³C and ³¹P NMR). The molecular structure of compound **1a** (monoclinic, $P2_1/n$) was determined by a single-crystal diffraction study. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Molybdenum complexes; Tungsten complexes; Carbonyl complexes; Iminophosphine complexes; Crystal structures

1. Introduction

There has recently been considerable interest in the chemistry of polydentate ligands with both 'soft' phosphorus and 'hard' nitrogen donor atoms [1-8], due in part to the variety of coordination possibilities displayed by them compared with classical P–P or N–N ligands [9]. Since the hard-ligand components can readily dissociate from a soft metal centre generating a vacant site on the metal ion for potential substrate binding, these N,P-ligands have also been investigated in order to design and develop new homogeneous catalytic systems [10–17]. Among the most studied ligands with this characteristic are the pyridylphosphines and the iminophosphines that we present here, which have been widely reported since 1992 in complexes of several

metals [10,11,13–21] but have not yet been studied when coordinating Group 6 metals.

Furthermore, some N,P-ligands were involved years ago in mechanistic studies of chelate ring opening in metal carbonyl complexes of the type $[M(CO)_4(N-P)]$ where M = Cr, Mo and W, and $N-P = Ph_2PCH_2$ - $CH_2N(CH_3)_2$, $Ph_2PCH_2CH_2NPh_2$, $Ph_2PCH_2CH_2NH_2$ and $Ph_2PCH_2CH_2CH_2N(CH_3)_2$ [22,23], showing that the labile nitrogen-donor group readily dissociated from the coordination sphere, allowing the entry of a new ligand.

Since then only a few examples of metal carbonyls with pyridylphosphines [24–31] have been reported, compared with the extensive and well documented bibliography of their phosphine analogues.

A recent surge of interest in carbonyl complexes with chelating phosphines is due to their ability to undergo ring-opening in order to facilitate isomerization reactions [32]. In this sense, the study of Group 6 carbonyl complexes containing iminophosphine ligands with the binding properties mentioned above should be of maxi-

^{*} Corresponding author. Tel.: + 34-968-367 454; fax: + 34-968-364 148.

E-mail address: gsg@fcu.um.es (G. Sánchez).

mum interest. On the other hand, the fact that their fundamental chemistry remains unexplored and that related examples have been recently reported [33-35] encouraged us to investigate in this field.

It is well known that to carry out carbonyl substitution reactions in Group 6 metal carbonyls, disubstituted precursors like *cis*-[M(CO)₄(pip)₂] [36] are preferred to enable the reaction to proceed smoothly in high yields, instead of direct reaction of the metal hexacarbonyl. Recent examples [34,37–40] where simple substitution reactions have occurred confirm the convenience of using the piperidine complex as metallic precursor. Here we report the synthesis and characterization of complexes of the type [M(CO)₄(*o*-Ph₂PC₆H₄-CH=NR)] (M = Mo, W; R = Me, Et, ⁱPr, ^tBu, NH-Me), prepared by direct reaction between iminophosphine ligands and the precursors *cis*-[M(CO)₄(pip)₂].

2. Experimental

C, H, and N analyses were carried out with a Perkin–Elmer 240C microanalyser. IR spectra were recorded on a Perkin–Elmer spectrophotometer 16F PC FTIR, using Nujol mulls between polyethylene sheets. NMR data were recorded on a Bruker AC 200E (¹H, ¹³C) or a Varian Unity 300 (¹H, ¹³C, ³¹P) spectrometer. Decomposition temperatures were determined on a Reichert microscope.

Reactions were carried out under a dinitrogen atmosphere using standard Schlenk techniques. The piperidine precursors cis-[M(CO)₄(pip)₂] (M = Mo, W) were prepared by the published method [36]. The iminophosphine ligands were prepared according to reported procedures [11] and all the solvents were dried by standard methods before use.

2.1. Preparation of the complexes $[M(CO)_4(o-Ph_2PC_6H_4-CH=NR)]$ (M = Mo; R = Me(1a), Et (2a), ⁱPr (3a), ⁱBu (4a), NH-Me (5a). M = W; R = Me (1b), Et (2b), ⁱPr (3b), ⁱBu (4b), NH-Me (5b))

The complexes were obtained by treating *cis*- $[M(CO)_4(pip)_2]$ (M = Mo, W) with the corresponding iminophosphine (molar ratio 1:1) in dichloromethane according to the following general method. To a dichloromethane (10 ml) yellow solution of the precursor $[M(CO)_4(pip)_2]$ (0.310 mmol) was added the stoichiometric amount of the previously prepared iminophosphine dissolved in dichloromethane (10 ml). The reaction mixture changed colour to orange-red gradually and was refluxed for 30 min (Mo compounds) or 1 h (W compounds). The hot solution was filtered through celite and then concentrated under reduced pressure to half volume. Addition of hexane caused

precipitation of the new complexes, which were filtered off, air dried and recrystallized from dichloromethane-hexane.

[Mo(CO)₄(o-Ph₂PC₆H₄-CH=NMe)] (1a) was obtained in 72% yield. *Anal.* Calc. for C₂₄H₁₈NPO₄Mo: C, 56.4; H, 3.5; N, 2.7. Found: C, 56.2; H, 3,6; N, 3.0%. M.p. 167°C dec. ¹H NMR (CDCl₃, 20°C) δ 3.67 (s, 1H, Me), 6.79 (m, 1H, H³), 7.34 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.15 (s, 1H, CH=N). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 35.97 (s).

[Mo(CO)₄(o-Ph₂PC₆H₄-CH=NEt)] (**2a**) was obtained in 83% yield. *Anal.* Calc. for C₂₅H₂₀NPO₄Mo: C, 57.2; H, 3.8; N, 2.7. Found: C, 57.0; H, 4.0; N, 2.8%. M.p. 151°C dec. ¹H NMR (CDCl₃, 20°C) δ 1.01 (t, 3H,CH₃-, $J_{\rm HH} = 7.0$), 3.82 (cd, 2H, -CH₂-, $J_{\rm HH} = 7.0$), 6.80 (m, 1H, H³), 7.48 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.21 (s, 1H, CH=N). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 34.86 (s).

[Mo(CO)₄(o-Ph₂PC₆H₄-CH=NⁱPr)] (**3a**) was obtained in 88% yield. *Anal.* Calc. for C₂₆H₂₂NPO₄Mo: C, 57.9; H, 4.1; N, 2.6. Found: C, 57.8; H, 4.1; N, 2.7%. M.p. 160°C dec. ¹H NMR (CDCl₃, 20°C) δ 1.04 (d, 6H, 2CH₃-, J_{HH} = 6.6), 4.17 (m, 1H,CH, J_{HH} = 6.6), 6.69 (m, 1H, H³), 7.37 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.22 (d, 1H, CH=N, J_{HP} = 2.1). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 36.02 (s).

[Mo(CO)₄(o-Ph₂PC₆H₄-CH=N^tBu)] (4a) was obtained in 86% yield. *Anal.* Calc. for C₂₇H₂₄NPO₄Mo: C, 58.6; H, 4.4; N, 2.5. Found: C, 58.5; H, 4.2; N, 2.7%. M.p. 138°C dec. ¹H NMR (CDCl₃, 20°C) δ 1.13 (s, 9H, 3CH₃-), 6.67 (m, 1H, H³), 7.26 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.29(d, 1H, CH=N, J_{HP} = 2,0).). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 33.96 (s).

[Mo(CO)₄(o-Ph₂PC₆H₄-CH=NNHMe)] (**5**a) was obtained in 68% yield. *Anal.* Calc. for C₂₄H₁₉N₂PO₄Mo: C, 54.8; H, 3.6; N, 5.3. Found: C, 54.5; H, 3.5; N, 5.4%. M.p. 139°C dec. ¹H NMR (CDCl₃, 20°C) δ 2.80 (d, 3H, Me, J_{HH} = 5.1), 5.62 (m, 1H, NH, J_{HH} = 4.8), 6.74 (m, 1H, H³), 7.36 (m, 13H, H⁴,H⁵,H⁶; Ph), 7.51 (s, 1H, CH=N). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 36.55 (s).

[W(CO)₄(*o*-Ph₂PC₆H₄-CH=NMe)] (**1b**) was obtained in 70% yield. *Anal.* Calc. for C₂₄H₁₈NPO₄W: C, 48.1; H, 3.0; N, 2.3. Found: C, 47.9; H, 3,1; N, 2.3%. M.p. 195°C dec. ¹H NMR (CDCl₃, 20°C) δ 3.83 (s, 1H, Me), 6.81 (m, 1H, H³), 7.35 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.08 (s, 1H, CH=N). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 24.88 (s; with ¹⁸³W satellites, $J_{WP} = 241$ Hz).

[W(CO)₄(*o*-Ph₂PC₆H₄-CH=NEt)] (**2b**) was obtained in 80% yield. *Anal.* Calc. for C₂₅H₂₀NPO₄W: C, 49.0; H, 3.3; N, 2.3. Found: C, 48.9; H, 3.0; N, 2.4%. M.p. 182°C dec. ¹H NMR (CDCl₃, 20°C) δ 0.99 (t, 3H,CH₃-, *J*_{HH} = 7.5), 3.93 (cd, 2H, -CH₂-, *J*_{HH} = 7.5), 6.81 (m, 1H, H³), 7.42 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.14 (d, 1H, CH=N, *J*_{HP} = 2.4). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 24.93 (s; with ¹⁸³W satellites, *J*_{WP} = 238 Hz).

 $[W(CO)_4(o-Ph_2PC_6H_4-CH=N^iPr)]$ (3b) was obtained in 73% yield. Anal. Calc. for $C_{26}H_{22}NPO_4W$: C, 49.8; Table 1 Crystal data and structure refinement for **1a**

Empirical formula	C ₂₄ H ₁₈ MoNO ₄ P
Formula weight	511.30
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	$P2_1/n$
Unit cell dimensions	-
a (Å)	13.817(3)
b (Å)	10.785(2)
<i>c</i> (Å)	16.077(3)
α (°)	90
β (°)	106.38(3)
γ (°)	90
Volume (Å ³)	2298.5(8)
Ζ	4
Density (calculated) (Mg m^{-3})	1.478
Absorption coefficient (mm^{-1})	0.669
F(000)	1032
Theta range for data collection (°)	2.29 to 27.50
Index ranges	$-17 \le h \le 1, -13 \le k \le 1,$
-	$-20 \le l \le 20$
Reflections collected	5105
Independent reflections	4040 $[R_{int} = 0.0159]$
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	4040/0/280
Goodness-of-fit on F^2	1.069
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0301, wR_2 = 0.0767$
R indices (all data)	$R_1 = 0.0344, \ wR_2 = 0.0797$
Largest diff. peak and hole (e \AA^{-3})	0.306 and -0.507

H, 3.5; N, 2.2. Found: C, 49.7; H, 3.3; N, 2.0%. M.p. 180°C dec. ¹H NMR (CDCl₃, 20°C) δ 1.02 (d, 6H, 2CH₃-, $J_{HH} = 6.6$), 4.38 (m, 1H,CH, $J_{HH} = 6.6$), 6.77 (m, 1H, H³), 7.41 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.18 (d, 1H, CH=N, $J_{HP} = 3.0$). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 25.43 (s; with ¹⁸³W satellites, $J_{WP} = 239$ Hz).

[W(CO)₄(o-Ph₂PC₆H₄-CH=N'Bu)] (**4b**) was obtained in 81% yield. *Anal.* Calc. for C₂₇H₂₅NPO₄W: C, 50.6; H, 3.8; N, 2.2. Found: C, 50.5; H, 3.8; N, 2.4%. M.p. 145°C dec. ¹H NMR (CDCl₃, 20°C) δ 1.24 (s, 9H, 3CH₃-), 6.76 (m, 1H, H³), 7.41 (m, 13H, H⁴,H⁵,H⁶; Ph), 8.34 (d, 1H, CH=N, $J_{HP} = 2.7$). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 24.08 (s; with ¹⁸³W satellites, $J_{WP} =$ 240 Hz).

[W(CO)₄(o-Ph₂PC₆H₄-CH=NNHMe)] (**5b**) was obtained in 69% yield. *Anal.* Calc. for C₂₄H₁₉N₂PO₄W: C, 46.9; H, 3.1; N, 4.6. Found: C, 46.9; H, 3.3; N, 4.4%. M.p. 172°C dec. ¹H NMR (CDCl₃, 20°C) δ 2.84 (d, 3H, Me, *J*_{HH} = 5.1), 5.64 (m, 1H, NH, *J*_{HH} = 5.4), 6.81 (m, 1H, H³), 7.38 (m, 13H, H⁴,H⁵,H⁶; Ph), 7.51 (s, 1H, CH=N). ³¹P-{¹H}NMR (CDCl₃, 20°C) δ 26.18 (s; with ¹⁸³W satellites, *J*_{WP} = 242 Hz).

2.2. Crystal structure determination of $[Mo(CO)_4(o-Ph_2PC_6H_4-CH=NMe)]$ (1a)

A single crystal of complex $[Mo(CO)_4(o-Ph_2PC_6H_4 -$ CH=NMe)] (1a) (approximate dimensions $0.30 \times$ 0.30×0.30 mm was mounted on a Siemens P4 diffractometer. The crystallographic data are shown in Table 1. Accurate cell parameters were determined by least-squares fitting of 42 high-angle reflections. The scan method was $\omega - 2\theta$ with the range of *hkl* ($-17 \leq$ $h \le 1, -13 \le k \le 1, -20 \le l \le 20$) corresponding to $2\theta_{\rm max} = 55^{\circ}$. Empirical ψ -scan mode absorption correction was made. The structure was solved by direct methods [41] and refined [42] by full-matrix leastsquares techniques using anisotropic thermal parameters for non-H atoms. The final R factor was 0.0301 $w = 1/[\sigma^2(F_0^2) + (0.0315P)^2 +$ where $(R_{\rm w} = 0.0767)$ 1.41P] and $P = F_o^2 + 2F_c^2/3$ over 3627 observed reflections $(I > 2\sigma(I))$.

3. Results and discussion

In dichloromethane, the disubstituted precursors *cis*-[M(CO)₄(pip)₂] (M = Mo or W) react under mild conditions with the corresponding iminophosphines previously prepared in 1:1 molar ratio, affording the expected compounds [M(CO)₄(o-Ph₂PC₆H₄-CH=NR)] in good yield (Scheme 1). The new complexes are air stable orange to red solids that show negligible molar conductances and have been characterized by elemental analysis, ¹H, ³¹P{¹H}, ¹³C{¹H} NMR and IR spec-



M = Mo, W.R = Me, Et, ⁱPr, ^tBu, NHMe.

Scheme 1.

Table 2 IR a and $\{^{1}H\}^{13}C$ (δ ppm) data for the complexes [M(CO)_4{P(o-C_6H_4-CH=N-R)Ph_2}]

Complex	v(CH=N)	v(CO)	¹³ C (SiMe ₄) ^{b,c}
1a	1622	2014, 1918, 1884, 1852, 1902(sh)	61.96 (d, 1C, Me, $J_{CP} = 3.6$) 168.32 (d, 1C, CH=N, $J_{CP} = 4.4$) 207.87 (d, 2C, $C_{3,4}$, $J_{CP} = 8.4$) 216.61 (d, 1C, C_1 , $J_{CP} = 32.8$) 220.99 (d, 1C, C_2 , $J_{CP} = 7.6$)
2a	1614	2008, 1892, 1872, 1856	16.27 (s, 1C, CH ₃ -) 67.71 (d, 1C, $-CH_{2^-}$, $J_{CP} = 2.3$) 168.25 (d, 1C, CH=N, $J_{CP} = 3.8$) 207.90 (d, 2C, $C_{3,4}$, $J_{CP} = 9.1$) 216.57 (d, 1C, C_1 , $J_{CP} = 33.6$) 221.00 (d, 1C, C_2 , $J_{CP} = 6.9$)
3a	1612	2010, 1904, 1888, 1864	22.29 (s, 2C, 2CH ₃ –) 69.58 (s, 1C, CH) 165.57 (d, 1C, CH=N, $J_{CP} = 4.6$) 207.82 (d, 2C, $C_{3,4}$, $J_{CP} = 9.1$) 216.93 (d, 1C, C_1 , $J_{CP} = 33.6$) 221.37 (d, 1C, C_2 , $J_{CP} = 7.6$)
4a	1598	2008, 1892, 1872, 1842	31.18 (s, 3C, 3CH ₃ -) 64.60 (s, 1C, C) 167.60 (d, 1C, CH=N, $J_{CP} = 4.6$) 207.75 (d, 2C, $C_{3,4}$, $J_{CP} = 8.3$) 217.04 (d, 1C, C_1 , $J_{CP} = 34.3$) 221.29 (d, 1C, C_2 , $J_{CP} = 7.7$)
5a	1584	2014, 1916, 1892, 1856	35.18 (s, 1C, Me) 142.62 (d, 1C, CH=N, $J_{CP} = 5.8$) 207.52 (d, 2C, $C_{3, 4}$, $J_{CP} = 9.2$) 216.19 (d, 1C, C_{1} , $J_{CP} = 31.3$) 220.61 (d, 1C, C_{2} , $J_{CP} = 7.6$)
1b	1586	2008, 1892, 1872, 1842, 1912(sh)	64.17 (d, 1C, Me, $J_{CP} = 3.4$) 168.59 (d, 1C, CH=N, $J_{CP} = 4.4$) 202.26 (d, 2C, $C_{3,4}$, $J_{CP} = 6.9$) 209.78 (d, 1C, C_1 , $J_{CP} = 31.9$) 211.26 (d, 1C, C_2 , $J_{CP} = 5.3$)
2b	1584	2002, 1910, 1880, 1850	22.48 (s, 1C, CH ₃ -) 70.09 (s, 1C, $-$ CH ₂ -) 168.33 (d, 1C, CH=N, $J_{CP} = 4.7$) 202.34 (d, 2C, $C_{3,4}$, $J_{CP} = 6.9$) 209.84 (d, 1C, C_1 , $J_{CP} = 31.9$) 211.11 (d, 1C, C_2 , $J_{CP} = 5.5$)
3b	1606	2004, 1880, 1860, 1846, 1898(sh)	22.31 (s, 2C, 2CH ₃ –) 72.22 (s, 1C, CH) 165.48 (d, 1C, CH=N, $J_{CP} = 4.6$) 202.36 (d, 2C, $C_{3,4}$, $J_{CP} = 6.9$) 210.35 (d, 1C, C_1 , $J_{CP} = 32.5$) 211.61 (d, 1C, C_2 , $J_{CP} = 5.7$)
4b	1592	2002, 1876, 1842, 1770, 1864(sh), 1788(sh)	31.60 (s, 3C, 3CH ₃ -) 66.57 (s, 1C, C) 168.42 (d, 1C, CH=N, $J_{CP} = 4.6$) 202.30 (d, 2C, $C_{3, 4}, J_{CP} = 7, 3$) 210.21 (d, 1C, $C_{1}, J_{CP} = 31.8$) 210.95 (d, 1C, $C_{2}, J_{CP} = 5.0$)
5b	1582	2010, 1906, 1886, 1850	35.84 (d, 1C, Me) 143.74 (d, 1C, CH=N, $J_{CP} = 4.1$) 202.27 (d, 2C, $C_{3,4}$, $J_{CP} = 7.0$) 209.49 (d, 1C, C_1 , $J_{CP} = 30.2$) 210.41 (d, 1C, C_2 , $J_{CP} = 4.5$)

^a In Nujol (cm⁻¹). ^b In CDCl₃ at 20°C. ^{c 31}P⁻¹³C coupling constants in Hz.



Fig. 1. Molecular structure of complex 1a.

troscopy (Table 2, Section 2). Compound **1a** was further characterized by a single-crystal X-ray diffraction study; the structure is discussed later.

The IR spectra of all compounds show the four-band pattern expected for the $M(CO)_4$ framework of C_s symmetry (3A' + A'') [43] together with a single medium band in the 1622–1582 cm⁻¹ region, attibuted to the C=N stretching vibration of the iminophosphine ligand coordinated to Mo or W; this appears shifted to lower frequencies than in the free ligand.

The ¹H NMR data are collected in Section 2. All the spectra display the expected resonances for the iminophosphine ligands [11], which are little perturbed on complexation and appear in the range previously observed on coordinating other metals [44]. The appreciable coupling to the phosphorus atom exhibited by the iminic proton is the only remarkable feature. The ³¹P NMR spectra of the new complexes show a singlet resonance shifted downfield from the free ligand, accompanied by ¹³⁸W satellites in **b**-compounds ($J_{WP} = 240$ Hz).

Identification of the new complexes is substantiated unambiguously by ¹³C{¹H} NMR spectroscopy. Selected data from the spectra are listed in Table 2 focusing on the aliphatic region, the iminic position of the iminophosphine ligand and the carbonyl carbon resonances. In this last region three resonances are observed at approximately 221, 217 and 208 ppm in molybdenum compounds (211, 210 and 202 ppm in tungsten complexes) assigned to C2, C1 and the equivalents C3 and C4 respectively. Thus, as $M \rightarrow CO \pi$ back-donation increases, the ¹³C chemical shift is deshielded, in agreement with previously reported data [43]. The usual trend in ³¹P–¹³C coupling constants in phosphorus-substituted derivatives of molybdenum and tungsten hexacarbonyls is also followed, all compounds displaying considerably greater J_{PC} for *trans* arrangements than for the *cis* geometry [43,45].

3.1. Molecular structure of $[Mo(CO)_4(o-Ph_2PC_6H_4-CH=NMe)]$ (1a)

The coordination about the central metal atom is a distorted octahedron (Fig. 1). Selected bond distances and angles are presented in Table 3. A large deviation from ideal octahedral geometry is observed for the P-Mo-N angle, for which a contraction from 90 to 77.78(7)° occurs. Small P-M-N angles also have been

Table 3 Selected bond lengths (Å) and angles (°) for **1a**

Mo-C(22)	1.947(3)	C(22)-Mo-C(23)	90.07(14)
Mo-C(23)	1.996(3)	C(22)-Mo-C(21)	88.73(13)
Mo-C(21)	2.024(3)	C(22)-Mo-C(20)	84.19(15)
Mo-C(20)	2.028(3)	C(22)–Mo–P	100.25(10)
Mo–N	2.279(2)	C(23)-Mo-N	92.12(12)
Мо–Р	2.4977(8)	C(21)-Mo-N	93.90(11
C(20)-Mo-N	93.27(14)	C(21)-Mo-C(20)	172.44(15)
N–Mo–P	77.78(7)	C(22)-Mo-N	176.62(13)
C(21)-Mo-P	87.21(11)	C(23)-Mo-P	168.74(9)
C(20)-Mo-P	96.64(10)	C(23)-Mo-C(21)	88.53(16)
C(23)–Mo–C(20)	88.83(14)		

found in compounds with the same ligand coordinating Pd (square-planar) [16], Rh [11] and Ru [46].

The Mo–P and Mo–N distances are similar to those found in related compounds [47,48]. The mutually *trans* CO groups bind at an average distance of 2.026(3) Å. The carbonyl ligand *trans* to P is found at a distance of 1.996(3) Å while the M–CO bond distance opposite the imine group is 1.947(3) Å. The distances follow the expected trends based on the electronic properties of their associated *trans* ligands [43], increasing as the π -acceptor ability of the ligand *trans* to the bond in question increases (N-donor < P-donor < CO).

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