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THE SYNTHESIS OF AND NUCLEAR MAGNETIC RESONANCE STUDIES ON BIMETALLIC CHROMIUM(0), OR MOLYBDENUM(0) CARBONYL COMPLEXES CONTAINING BRIDGING *trans*-$Ph_2PCH{=}CHPPh_2$ LIGANDS: CRYSTAL STRUCTURE OF $[Mo_2(CO)_8(\mu\text{-}trans\text{-}Ph_2PCH{=}CHPPh_2)_2]$

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Summary

The *trans*-$Ph_2PCH{=}CHPPh_2$ (*t*-dppe)-bridged bimetallic 10-membered ring complexes $[M_2(CO)_8(\mu\text{-}t\text{-dppe})_2]$ (M = Cr or Mo) have been synthesised by treatment of the metal hexacarbonyl with one equivalent of *t*-dppe at elevated temperature. The fluxional process in the Mo(μ-*t*-dppe)Mo ring has been studied by variable temperature $^{31}P\text{-}\{^1H\}$ NMR spectroscopy and is characterised by a change from an A_4 spin system at +20 °C to an AA′BB′ system at −100 °C. The bimetallic complex $[Mo_2(CO)_6(\mu\text{-}t\text{-dppe})_3]$ has been prepared and $^{31}P\text{-}\{^1H\}$ NMR studies indicate that in solution all four phosphorus nuclei are equivalent, even at −115 °C. Attempts to prepare related heterobimetallic complexes are also described. Crystals of $[Mo_2(CO)_8(\mu\text{-}trans\text{-}Ph_2PCH{=}CHPPh_2)_2]$ are monoclinic, space group $P2_1/a$, with *a* 2020.2(5), *b* 1423.1(5), *c* 2145.1(5) pm, β 113.95(2)°, and $Z = 4$; final *R* factor 0.0465 for 5668 observed reflections. The structure shows two $[Mo(CO)_4]$ moieties linked by two *trans*-dppe bridges to give a 10-membered $Mo_2P_4C_4$ ring.

The chemistry of the diphosphine ligand $Ph_2PCH_2CH_2PPh_2$ (dppe) is dominated by the formation of stable, five-membered ring chelate complexes which one would expect to be thermodynamically more favoured than compounds containing the 10-membered ring moiety $M^1(\mu\text{-dppe})_2M^2$. However, we showed in a recent publication that bimetallic Group 6 metal carbonyl complexes of dppe could be prepared by treating a complex of type *cis*-$[M^1(CO)_4(PPh_2H)_2]$ with one of type *cis*-$[M^2(CO)_4\{PPh_2(CH{=}CH_2)\}_2]$ in the presence of Bu^tOK as catalyst [1]. The coordinative chemistry of the related unsaturated diphosphine *cis*-$Ph_2PCH{=}CHPPh_2$ (*c*-dppe) should parallel that of dppe, however the chemistry of the isomeric *trans*-$Ph_2PCH{=}CHPPh_2$ (*t*-dppe) should be dominated by bridging or monodentate interactions since, of course, chelation is sterically impossible. There have been several reports of the use of *t*-dppe as a ligand in transition metal chemistry. These

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include the preparation of polymeric complexes of rhodium, [Rh(μ-*t*-dppe)(CO)Cl]$_n$ [2], monodentate or bridging complexes of manganese, [(η-C_5H_5)Mn(CO)(NO)(*t*-dppe-*P*)][PF_6] and {[(η-C_5H_5)Mn(CO)(NO)]$_2$(μ-*t*-dppe)}[PF_6]$_2$ [3], binuclear bisthiolene complexes of iron and cobalt [4], the monodentate tungsten complex [W$(CO)_5$(*t*-dppe-*P*)] [5], polymeric nickel, cobalt, and palladium complexes [6], the gold complex [AuCl(*t*-dppe-*P*)] [7] and the vanadium complexes [Et_4N]$_2$-[{V$(CO)_5$}$_2$(μ-*t*-dppe)], [{(η-C_5H_5)V$(CO)_3$}$_2$(μ-*t*-dppe)], and [{(η-C_5H_5)V$(CO)_2$}$_2$-(μ-*t*-dppe)$_2$] [8]. Only the last complex was formulated as having a 10-membered ring structure and there appear to be no reports of X-ray structural investigations of metal complexes containing the ligand *t*-dppe.

In this paper we describe methods for the synthesis of chromium and molybdenum homobimetallic complexes containing bridging *t*-dppe ligands and, in addition, describe attempts to prepare related heterobimetallic species.

Results and discussion

We find that treatment of [Mo$(CO)_6$] with one equivalent of *t*-dppe in boiling light petroleum (b.p. 100–120 °C) gives the bimetallic complex [$Mo_2(CO)_8$(μ-*t*-dppe)$_2$] (**1b**) in 65% isolated yield. The chromium analogue (**1a**) was made similarly, however, in this case the major product was an insoluble, possibly polynuclear, material which was not characterised. Attempts to substitute [W$(CO)_6$] with *t*-dppe met with failure. Even after boiling in light petroleum (b.p. 100–120 °C) for 48 h, no reaction had occurred (^{31}P-{^{1}H} NMR evidence). Treatment of [Mo$(CO)_6$] with two equivalents of *t*-dppe gave the complex [$Mo_2(CO)_6$(μ-*t*-dppe)$_3$] (**2**). **2** was also prepared, quantitatively, by treating **1b** with *t*-dppe. Preparative details are given in the Experimental section. The complexes were characterised by elemental analysis and ^{1}H and ^{31}P-{^{1}H} NMR and IR spectroscopy (Tables 1, 2, and 3 respectively). In addition, the structure of complex **1b** has been determined by X-ray crystallography (see below).

The IR absorption spectra of **1a** and **1b** [ν(CO) region] were very similar, showing four bands in CH_2Cl_2 solution, typical of the pattern expected for a *cis*-[M$(CO)_4L_2$] stereochemistry. The IR spectrum of **2** showed two strong ν(CO) absorptions characteristic of a *fac*-[M$(CO)_3L_3$] stereochemistry [9].

TABLE 1

COLOURS, YIELDS, AND MICROANALYTICAL DATA

Complex	Colour	Yield (%)	Analysis (Found (calcd.) (%))			Mol.wt. [a]
			C	H	P	
1a	yellow	18	63.5 (64.3)	4.1 (3.95)	11.2 (11.05)	–
1b	pale-yellow	65	59.75 (59.6)	3.75 (3.65)	10.5 (10.25)	1216 (1209)
2	pale-yellow	65	64.85 (65.1)	4.1 (4.3)	11.85 (12.0)	–
3	pale-yellow	57	59.35 (59.6)	3.55 (3.65)	10.5 (10.25)	–

[a] Measured in $CHCl_3$.

TABLE 2

^{31}P-{^{1}H} [a] AND ^{1}H-{^{31}P} [b] NMR DATA

Complex	^{31}P-{^{1}H}				^{1}H-{^{31}P}(δ, ppm)	
	δ(P) (ppm)		J(MoP) (Hz)	N [c] (Hz)	+20 °C	−115 °C
	+20 °C	−100 °C				
1a	49.6 [d]	–	–	–	6.02	–
1b	31.1 [d]	32.1, 27.7 [e]	n.r. [f]	42	6.18	6.18, 5.89
2	28.7 [d]	27.5 [e]	125	–	6.23	6.02
4	31.1, −6.9 [e]	–	–	14	–	–

[a] Spectra measured in CD_2Cl_2; chemical shifts (δ) in ppm (±0.1) to high frequency of 85% H_3PO_4 and coupling constants (J) in Hz (±3). [b] Spectra measured in CD_2Cl_2; chemical shifts (δ) ppm (±0.01) to high frequency of $SiMe_4$. Spectra at +20 °C measured at 100 MHz. Spectra at −115 °C measured at 360 MHz. [c] $N = J(AX) + J(AX')$. [d] ^{31}P-{^{1}H} spectra measured at 40.5 MHz. [e] ^{31}P-{^{1}H} spectra measured at 162 MHz. [f] Coupling to ^{95}Mo not resolved.

The ^{31}P-{^{1}H} NMR spectra of **1a** and **1b** were single lines at +20 °C. When the solution was cooled, the spectrum of **1b** first broadened then split into two resonances with a coalescence temperature of ca. −85 °C. At −100 °C the spectrum of complex **1b** consists of two deceptively simple triplets interpreted as an AA′BB′ spin system with $|N|$ 42 Hz. The ^{31}P-{^{1}H} NMR spectrum of **2** was a

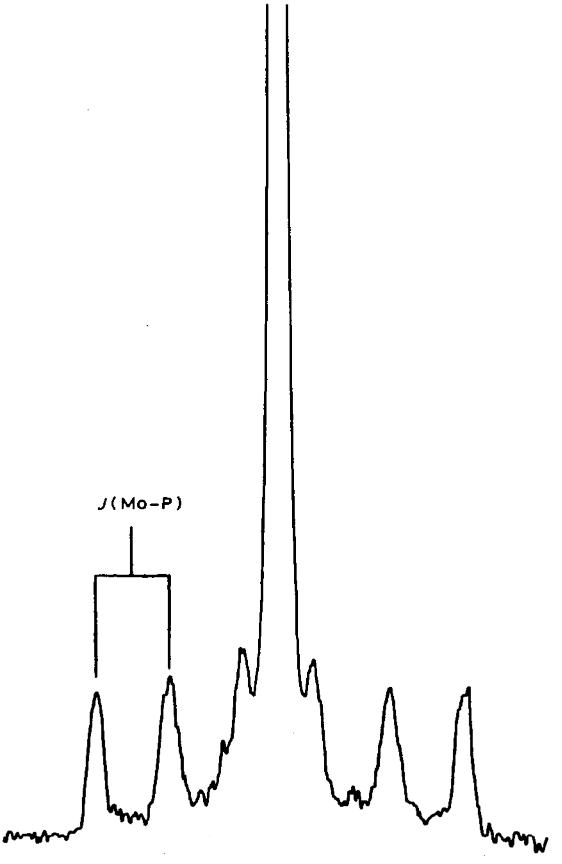

Fig. 1. ^{31}P-{^{1}H} NMR spectrum (40.5 MHz) of $[Mo_2(CO)_6(\mu\text{-}t\text{-dppe})_3]$ (**2**) in CD_2Cl_2 at +20 °C.

TABLE 3

IR ABSORPTION MAXIMA[a]

Complex	$\nu(C{\equiv}O)$ (cm^{-1})
1a	2015sh, 2010s, 1905sh, 1890s
1b	2030sh, 2025s, 1925sh, 1900s
2	1944s, 1856s
3[b]	2020s, 1915sh, 1905s, 1875s

[a] Measured in CH_2Cl_2 solution. [b] Nujol mull (sh = shoulder, s = strong).

single line even at −115 °C. At +20 °C six equally spaced satellites due to coupling to ^{95}Mo ($I = \frac{5}{2}$, 15.5%) are observed (Fig. 1). The ^1H-{^{31}P} NMR spectra of **1a** and **1b** at +20 °C showed a single resonance at ca. 6 ppm assigned to the ethenyl protons of the *t*-dppe ligands. However, when the solution was cooled to −115 °C the spectrum of **1b** showed two resonances which could be assigned to the ethenyl protons. The ^1H-{^{31}P} NMR spectrum of **2** showed a single resonance assigned to the ethenyl protons even at −115 °C. On the basis of these data the complexes **1a** and **1b** have been formulated as binuclear species with two bridging *t*-dppe ligands and a *cis*-geometry around each metal whereas **2** has been formulated as a binuclear species with three bridging *t*-dppe ligands and a *fac*-geometry around each metal.

Crystal structure of $[Mo_2(CO)_8(\mu\text{-t-dppe})_2]$ (1b)

Complex **1b** was crystallised from dichloromethane/ethanol (1/3). The structure is shown in Fig. 2 and selected intramolecular distances and angles are in Table 4. The $[Mo(CO)_4]$ moieties are linked by two *trans*-dppe bridges to give a ten-membered $Mo_2P_4C_4$ ring. Both metal centres show little distortions from their idealised

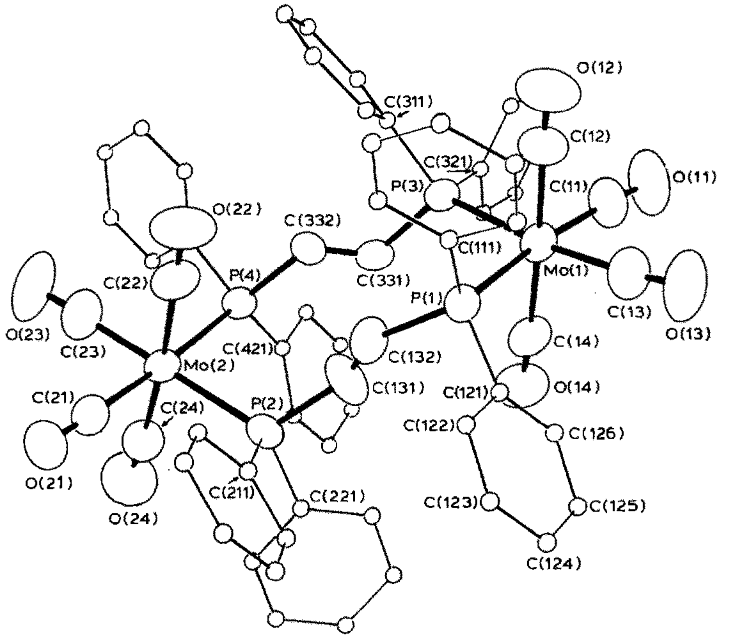

Fig. 2. Molecular structure of $[Mo_2(CO)_8(\mu\text{-}t\text{-dppe})_2]$ (**1b**), showing the principal atomic numbering.

TABLE 4

SELECTED DISTANCES BETWEEN INTERATOMIC VECTORS (pm) AND ANGLES (°) FOR $[Mo_2(CO)_8(\mu\text{-}t\text{-dppe})_2]$ (**1b**)

P(1)–Mo(1)	252.5(5)	P(2)–Mo(2)	253.9(4)
P(3)–Mo(1)	253.9(4)	P(4)–Mo(2)	254.0(5)
C(111)–P(1)	182.7(6)	C(121)–P(1)	183.1(5)
C(132)–P(1)	178.0(9)	C(131)–P(2)	174.2(10)
C(211)–P(2)	183.5(7)	C(221)–P(2)	182.5(5)
C(311)–P(3)	182.3(5)	C(321)–P(3)	183.3(7)
C(331)–P(3)	184.4(8)	C(332)–P(4)	181.0(8)
C(411)–P(4)	183.9(5)	C(421)–P(4)	182.2(6)
C(132)–C(131)	135.2(9)	C(332)–C(331)	132.7(9)
C(11)–Mo(1)	198.5(11)	C(12)–Mo(1)	201.6(10)
C(13)–Mo(1)	199.2(8)	C(14)–Mo(1)	202.5(10)
C(21)–Mo(2)	198.0(10)	C(22)–Mo(2)	202.0(11)
C(23)–Mo(2)	196.8(8)	C(24)–Mo(2)	203.8(11)
P(3)–Mo(1)–P(1)	100.9(2)	P(4)–Mo(2)–P(2)	94.6(2)
C(132)–P(1)–Mo(1)	124.8(4)	C(131)–P(2)–Mo(2)	116.4(4)
C(331)–P(3)–Mo(1)	118.8(3)	C(332)–P(4)–Mo(2)	116.1(3)
C(132)–C(131)–P(2)	130.8(6)	C(131)–C(132)–P(1)	130.5(5)
C(332)–C(331)–P(3)	122.7(5)	C(331)–C(332)–P(4)	127.2(5)

octahedral geometries. Figure 3 shows another view of the framework of complex **1b** which illustrates the stereochemistry of the ten-membered ring, in particular the relative orientation of the –C = C– bonds. Presumably, this structure is reflected in the low temperature NMR spectra discussed above. At ambient temperatures rapid ring flipping must occur to account for the NMR spectral changes.

Other studies

We also attempted to prepare complex **1b**, by treating the labile molybdenum complex $[Mo(CO)_4(nbd)]$ (nbd = norbornadiene) with one equivalent of *t*-dppe in

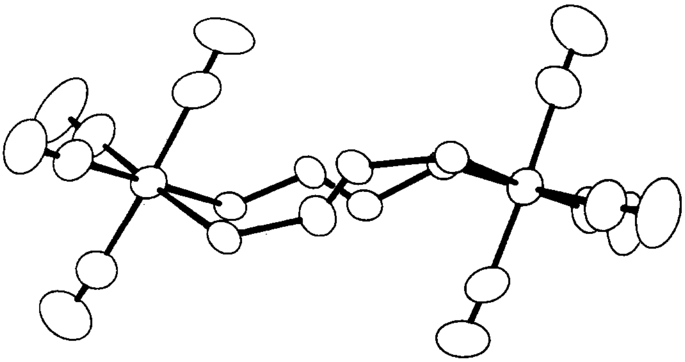

Fig. 3. A view of the 10-membered ring in **1b** showing the stereochemistry of the *t*-dppe ligands.

benzene. However, a material which was insoluble in most common organic solvents was isolated and we formulate this as the polynuclear complex $[Mo(CO)_4(\mu\text{-}t\text{-dppe})]_n$ (**3**). **3** was characterised by elemental analysis (Table 1) and IR spectroscopy (Table 3). The presence of four absorptions assigned to $\nu(CO)$ suggests a *cis*-$[Mo(CO)_4L_2]$ geometry.

We were interested in extending this work to the preparation of heterobimetallic complexes containing bridging *t*-dppe ligands. We therefore attempted to prepare the bis-monodentate complex $[Mo(CO)_4(t\text{-dppe-}P)_2]$ which we anticipated would be a useful precursor to bimetallic species. Treatment of $[Mo(CO)_4(nbd)]$ with an excess of *t*-dppe in dichloromethane gave a mixture of products [^{31}P-{^1H} NMR evidence]. These were identified as unreacted *t*-dppe, the bimetallic complex **1b**, and a new complex tentatively formulated as the hoped for $[Mo(CO)_4(t\text{-dppe-}P)_2]$ (**4**). The ^{31}P-{^1H} NMR spectrum of **4** showed the expected AA′XX′ spin system. The chemical shift of the low field branch was very close to that of **1b** suggesting coordination to molybdenum whereas the shift of the high field branch was close to that of free *t*-dppe (ca. 7 ppm) suggesting no coordination. However, repeated attempts to obtain pure samples of this complex met with failure, even by varying the stoichiometry of the reaction.

Treatment of a solution of the monodentate *t*-dppe complex **4** prepared in situ as above, with other metal complexes such as $HgCl_2$, $[Rh_2Cl_2(CO)_4]$ or $[PtCl_2(COD)]$ (COD = 1,5-cyclooctadiene) gave heterobimetallics [^{31}P-{^1H} NMR spectroscopic evidence] but we were unable to isolate these pure. Thus treatment of a dichloromethane solution of **4**, prepared in situ with $HgCl_2$ gave a product with a well-defined AA′XX′ ^{31}P-{^1H} NMR pattern + ^{199}Hg satellites viz. $\delta(P_A)$ (on Mo) 34.5 ppm, $\delta(P_B)$ (on Hg) 23.8 ppm, $^1J(HgP_B)$ 4135 Hz and $N = J(P_AP_B) + J(P_AP_{B'})$ = 30 Hz. This was the major species present in solution but we could not isolate it. Similarly for the Mo–Rh or Mo–Pt species.

Experimental

General methods were as previously described in recent papers from this laboratory [10]. The complex $[Mo(CO)_4(nbd)]$ was prepared by the literature method [11].

Preparations

$[Mo_2(CO)_8(\mu\text{-}t\text{-dppe})_2]$ (1b) from $[Mo(CO)_6]$ and t-dppe. A solution of $[Mo(CO)_6]$ (1.32 g, 5 mmol) and *t*-dppe (1.98 g, 5 mmol) in light petroleum solution (b.p. 100–120 °C) (25 cm^3) was refluxed for 16 h. The solution was cooled to room temperature and the solvent evaporated under reduced pressure. The residue was recrystallised from dichloromethane/ethanol to give the desired product as pale-yellow microcrystals. Yield 65%. **1a** was made similarly in 18% yield.

$[Mo_2(CO)_6(\mu\text{-}t\text{-dppe})_3]$ (2) from $[Mo(CO)_6]$ and t-dppe. A solution of $[Mo(CO)_6]$ (0.264 g, 1 mmol) and *t*-dppe (0.8 g, 2 mmol) in light petroleum solution (b.p. 100–120 °C) (25 cm^3) was refluxed for 40 h. The solution was cooled to room temperature and the solvent removed under reduced pressure. The residue was recrystallised from dichloromethane/ethanol to give the desired product as pale-yellow microcrystals. Yield 65%.

$[Mo_2(CO)_6(\mu\text{-}t\text{-dppe})_3]$ (2) from $[Mo_2(CO)_8(\mu\text{-}t\text{-dppe})_2]$ (1b) and t-dppe. A solution of $[Mo_2(CO)_8(\mu\text{-}t\text{-dppe})_2]$ (0.2 g, 0.166 mmol) and *t*-dppe (0.133 g, 0.336

TABLE 5

ATOMIC PARAMETERS FOR [Mo(CO)$_8$(μ-*t*-dppe)$_2$] (**1b**)

Atom	*x*	*y*	*z*
Mo(1)	3591.0(3)	3505.5(4)	3971.3(2)
Mo(2)	2604.3(3)	6450.6(4)	857.6(2)
P(1)	4125(1)	3468(1)	3092(1)
P(2)	3839(1)	6015(1)	1762(1)
P(3)	2369(1)	4258(1)	3289(1)
P(4)	1993(1)	6517(1)	1684(1)
C(111)	4000(2)	2335(2)	2658(2)
C(112)	4371(2)	1574(2)	3057(2)
C(113)	4250(2)	669(2)	2783(2)
C(114)	3758(2)	525(2)	2111(2)
C(115)	3387(2)	1286(2)	1712(2)
C(116)	3508(2)	2191(2)	1986(2)
C(121)	5106(1)	3638(3)	3401(2)
C(122)	5452(1)	3436(3)	2971(2)
C(123)	6183(1)	3657(3)	3174(2)
C(124)	6567(1)	4079(3)	3805(2)
C(125)	6221(1)	4280(3)	4235(2)
C(126)	5490(1)	4060(3)	4033(2)
C(131)	3848(4)	5211(5)	2378(3)
C(132)	3899(4)	4264(5)	2396(3)
C(211)	4478(7)	5483(3)	1454(2)
C(212)	4212(2)	4914(3)	875(2)
C(213)	4691(2)	4469(3)	651(2)
C(214)	5436(2)	4594(3)	1006(2)
C(215)	5702(2)	5163(3)	1585(2)
C(216)	5223(2)	5607(3)	1809(2)
C(221)	4384(2)	6957(2)	2308(2)
C(222)	4686(2)	6915(2)	3019(2)
C(223)	5099(2)	7664(2)	3400(2)
C(224)	5209(2)	8455(2)	3069(2)
C(225)	4907(2)	8497(2)	2358(2)
C(226)	4494(2)	7747(2)	1978(2)
C(311)	1671(2)	3620(3)	2595(2)
C(312)	1891(2)	3012(3)	2206(2)
C(313)	1378(2)	2614(3)	1612(2)
C(314)	645(2)	2824(3)	1408(2)
C(315)	425(2)	3432(3)	1797(2)
C(316)	938(2)	3830(3)	2391(2)
C(321)	1926(2)	4579(3)	3854(2)
C(322)	1998(2)	5476(3)	4137(2)
C(323)	1705(2)	5673(3)	4610(2)
C(324)	1340(2)	4973(3)	4800(2)
C(325)	1268(2)	4076(3)	4517(2)
C(326)	1561(2)	3879(3)	4044(2)
C(331)	2341(3)	5382(4)	2851(3)
C(332)	2054(3)	5463(4)	2177(3)
C(411)	1008(1)	6606(3)	1195(2)
C(412)	706(1)	7488(3)	967(2)
C(413)	−27(1)	7566(3)	538(2)
C(414)	−458(1)	6763(3)	337(2)
C(415)	−155(1)	5881(3)	565(2)

continued

TABLE 5 (continued)

Atom	x	y	z
C(416)	578(1)	5802(3)	994(2)
C(421)	2186(2)	7426(3)	2332(2)
C(422)	2831(2)	7932(3)	2539(2)
C(423)	3012(2)	8578(3)	3071(2)
C(424)	2547(2)	8718(3)	3398(2)
C(425)	1901(2)	8212(3)	3191(2)
C(426)	1721(2)	7566(3)	2659(2)
C(11)	3312(4)	3427(5)	4756(4)
O(11)	3190(3)	3365(4)	5228(3)
C(12)	3182(4)	2207(6)	3680(4)
O(12)	2979(3)	1455(4)	3525(3)
C(13)	4492(4)	2864(5)	4597(3)
O(13)	4979(3)	2436(4)	4966(3)
C(14)	3988(3)	4825(6)	4215(3)
O(14)	4189(3)	5581(4)	4328(3)
C(21)	3012(3)	6445(5)	160(3)
O(21)	3231(3)	6461(4)	−253(2)
C(22)	2364(4)	5067(6)	728(3)
O(22)	2217(3)	4272(4)	651(3)
C(23)	1662(4)	6695(6)	100(4)
O(23)	1123(3)	6846(6)	−362(3)
C(24)	2754(4)	7869(6)	926(4)
O(24)	2805(4)	8659(4)	927(4)

mmol) in light petroleum (b.p. 100–120 °C) (15 cm^3) was refluxed for 24 h. The solution was cooled to room temperature and the solvent removed under reduced pressure. The residue was dissolved in dichloromethane and examined by ^{31}P-{^{1}H} NMR spectroscopy which indicated quantitative conversion of **1b** to **2**.

$[Mo(CO)_4(\mu\text{-}t\text{-}dppe)]_n$ (3) from $[Mo(CO)_4(nbd)]$ and t-dppe. *t*-Dppe (0.398 g, 1 mmol) was added to a solution of $[\text{Mo(CO)}_4(\text{nbd})]$ (0.300 g, 1 mmol) in benzene (20 cm^3). After 2 h the pale-yellow precipitate was filtered off, washed with benzene and dried in vacuo. Yield 57%.

$[Mo(CO)_4(t\text{-}dppe\text{-}P)_2]$ (4) from $[Mo(CO)_4(nbd)]$ and t-dppe. *t*-Dppe (2.40 g, 6 mmol) was added to a solution of $[\text{Mo(CO)}_4(\text{nbd})]$ (0.30 g, 1 mmol) in dichloromethane (75 cm^3). After 24 h the volume of the solution was reduced to 20 cm^3 and excess ethanol added to precipitate the product as pale-yellow microcrystals. Yield 0.56 g. ^{31}P-{^{1}H} NMR analysis of this product indicated the present of *t*-dppe, (**1b**), and a species formulated as $[\text{Mo(CO)}_4(t\text{-dppe-}P)_2]$ (**4**).

Crystallography

Suitable crystals of $[\text{Mo}_2(\text{CO})_8(\mu\text{-}t\text{-dppe})_2]$ (**1b**) were grown from dichloromethane/ethanol (1/3). All crystallographic measurements were made on a Syntex P2_1 diffractometer operating in the ω–2θ scan mode using graphite monochromated Mo-K_α radiation (λ 71.069 pm) following a standard procedure described elsewhere in detail [12]. The data set was corrected for absorption once the structure had been solved [13].

Crystal data. $C_{60}H_{44}Mo_2O_8P_4$, $M = 1208.78$, monoclinic, *a* 2020.2(5), *b* 1423.1(5), *c* 2145.1(5) pm, β 113.95(2)°, *U* 5.636 nm^3, space group $P2_1/a$ ($P2_1/c$, no. 14), $Z = 4$, D_c 1.42 g cm^{-3}, μ(Mo-K_α) 5.37 cm^{-1}, $F(000) = 2448$, T 290 K. 7943 data were collected, 5668 were considered observed [$I > 2.0\sigma(I)$]. Scans running from 1.0° below K_{α_1}, to 1.0° above K_{α_2}, scan speeds 2.0–29.3° min^{-1}, $4.0 \leqslant 2\theta \leqslant 45.0°$.

Structure determination and refinement

The structure was determined via standard heavy atom procedures and refined by full-matrix least squares using the SHELX program system [14]. All non-hydrogen atoms were assigned anisotropic thermal parameters with phenyl rings included in refinement as rigid bodies with idealised hexagonal symmetry (C–C 139.5 pm). All hydrogen atoms were included in calculated positions (C–H 108 pm) and assigned to an overall isotropic thermal parameter. The weighting scheme $w = [\sigma^2(F_o) + 0.0005(F_o)^2]^{-1}$ was used at the end of refinement to give a flat analysis of variance with increasing sin θ and $[F \mid F_{max}]^{1/2}$. Final R values are $R = 0.0465$ and $R' = 0.0507$ (574 parameters). The final atomic coordinates are given in Table 5. Lists of thermal parameters and structure factors are available from the authors.

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