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Synthesis and structural characterization of an unusual η^3-ligated 1,3-diphosphacyclobutadiene molybdenum complex via cyclodimerization of a phosphaalkyne. Crystal structure of $[Mo(\eta^5\text{-}C_9H_7)(CO)_2\{P\text{-}\eta^3\text{-}({}^tBu)CPC({}^tBu)PFBF_3\}]$

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Abstract

The phosphaalkyne ${}^tBuCP$ undergoes cyclodimerization at a molybdenum centre to afford a 1,3-diphosphacyclobutadiene complex in which, unusually, the four-membered ring is η^3 ligated to the metal.

Key words: Phosphaalkyne; Molybdenum; Boron; Bridging ligand; Cyclobutadiene; Crystal structure

1. Introduction

The chemistry of mononuclear transition metal complexes which contain coordinated alkynes is of considerable current interest [1–4]. It has been shown that the alkynes in some of these complexes can act as either two, three or four-electron donors. Green and coworkers [2] have shown that, in the complexes $[Mo(\eta^5\text{-}C_9H_7)(CO)(PEt_3)(MeC_2Me)][BF_4]$ and $[Mo(\eta^5\text{-}C_9H_7)(PMe_3)_2(MeC_2Me)][BF_4]$, each molybdenum atom attains a formal 18-electron configuration if the but-2-yne ligand functions as a four-electron donor.

Likewise, in the complexes $[Mo(\eta^5\text{-}C_5H_5)(CO)(MeC_2Me)_2][BF_4]$ and $[Mo(\eta^5\text{-}C_5H_5)(NCMe)(MeC_2Me)]_2[BF_4]$, whose structures have been confirmed by single-crystal X-ray diffraction studies, each alkyne can be considered to donate three electrons to the molybdenum atom [5]. Complexes which have alkynes bonded in the above bonding modes exhibit unusually low field ${}^{13}C$ chemical shifts [6].

The complex $[Mo(\eta^5\text{-}C_9H_7)(CO)_2(CH_3CN)_2][BF_4]$ has been extensively used as the starting material for the preparation of bis(alkyne)monocarbonyl cations, $[Mo(\eta^5\text{-}C_9H_7)(CO)(RC{\equiv}CR')_2]^+$ ($R = R'$ = alkyl or aryl) ($R = H$, R' = alkyl or aryl), and in view of the previously discussed analogy in the behaviour of alkynes and phosphaalkynes [7,8] it was of interest to carry out similar reactions with phosphaalkynes in order to see whether η^2-(3e or 4e)-phosphaalkyne complexes can be obtained.

2. Results and discussion

Treatment of $[Mo(\eta^5\text{-}C_9H_7)(CO)_2(CH_3CN)_2][BF_4]$ with an excess of ${}^tBuC{\equiv}P$ gave a red–brown mixture whose ${}^{31}P\{{}^1H\}$ NMR spectrum exhibited two strong singlets ($\delta(P) = -122.1$ ppm and $\delta(P) = -149.9$ ppm) and temperature-dependent multiplets ($\delta(P) = -65.0$ to -40.1 ppm). It was only possible to identify conclusively the complex responsible for the singlet resonance at $\delta(P) = -121.1$ ppm, which was isolated with a 54% yield. The ${}^1H$ NMR spectrum of this yellow product

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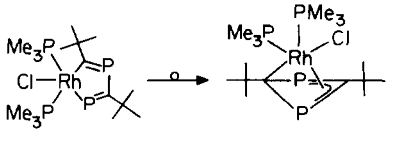
$[\mathrm{Mo}(\eta^5\text{-}\mathrm{C_9H_7})(\mathrm{CO})_2(^t\mathrm{BuCP})_2\mathrm{BF_4}]$ (**1**) (ν(CO) = 2039, and 1976 $\mathrm{cm^{-1}}$) confirmed that two moles of ^{t}BuCP had been incorporated, and the unusual structure of the complex was confirmed by a single-crystal X-ray diffraction study.

The molecular structure shown in Fig. 1 reveals that, in contrast with the known behaviour of alkynes, the acetonitrile ligands have been displaced by two phosphaalkynes to form a 1,3-diphosphacyclobutadiene complex. The solid state structure shows that the 1,3-diphosphacyclobutadiene ring in **1** is unlike that in several related η^4-1,3-diphosphacyclobutadiene complexes $[\mathrm{M}(\eta^5\text{-}(\mathrm{C_5R_5})(^t\mathrm{BuCP})_2]$ (R = H or Me) (M = Co, Rh or Ir) [9–11]. The most significant structural feature is the η^3-C–P–C allylic fragment which is symmetrically bonded to the molybdenum, and only the three atoms C(1), C(2) and P(2) of the $\mathrm{P_2C_2}^t\mathrm{Bu_2}$ ring take part in the bonding to the molybdenum atom. As a result, the 1,3-diphosphacyclobutadiene ring is no longer planar, and the remaining phosphorus atom, which is attached to one of the fluorine atoms of the tetrafluoroborate anion, is directed out of the C(2)–P(2)–C(1) plane. The neutral complex is best described as $[\mathrm{Mo}(\eta^5\text{-}\mathrm{C_9H_7}(\mathrm{CO})_2\{\eta^3\text{-}(^t\mathrm{Bu})\mathrm{CPC}(^t\mathrm{Bu})\mathrm{PFBF_3}\}]$.

Several tetrafluoroborate anion interactions with metals have been previously reported [12–17] but the compound reported here, to our knowledge, is the first example of interaction of a tetrafluoroborate anion with a main group element. Selected bond lengths and bond angles for **1** are listed in Table 1. Although the complex crystallized with two independent molecules (structures **1a** and **1b**) in the asymmetric unit, no significant geometric differences were found between the two, and dimensions averaged over the two molecules are used in the subsequent discussion. The Mo(1)–P(2) bond distance (2.447(2) Å) is slightly shorter than the mean Mo–P bond distance (2.537 Å) in the complex $[\mathrm{Mo}(\eta^5\text{-}\mathrm{C_5H_5}(\mathrm{CO})_2(\eta^3\text{-}\mathrm{P_3})]$ [18] and the Mo(1)–C(2) (2.323(7) Å) and Mo(1)–C(1) (2.294(8) Å) distances are equidistant within the limits of experimental error.

The structure of **1** represents the first example of η^3 ligation of the diphosphacyclobutadiene ring system, but Binger et al. [19] have shown that similar unexpected ligating modes of the "$\mathrm{P_2C_2}^t\mathrm{Bu_2}$" system can be observed in certain rhodium complexes. Thus treatment of $[\mathrm{Rh_2Cl_2(C_2H_4)_2}]$ with an excess of ^{t}BuCP in the presence of $\mathrm{PMe_3}$ gives a mixture of the complexes **2** (8%) and **3** (28%).



(2) (3)

In solution, **2** rearranges slowly to **3**, in which the four-membered ring exhibits considerable η^1,η^3-character [19]. Interestingly, a side product in the originally described [10] cyclodimerization of ^{t}BuCP in the presence of η^5-indenylbis(ethene)rhodium has recently been shown [20] to have the structure **4**.

This contains two η^3-PCP fragments resulting from a cotrimerization of ^{t}BuCP and 1 mol of $\mathrm{P_2}$, which

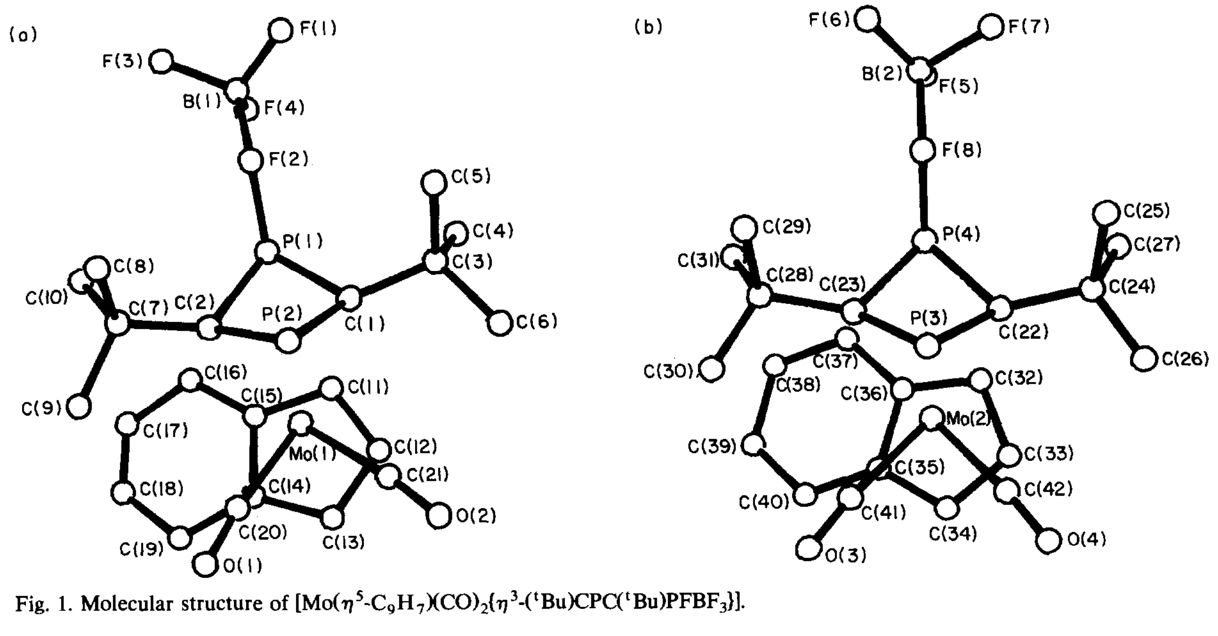


Fig. 1. Molecular structure of $[\mathrm{Mo}(\eta^5\text{-}\mathrm{C_9H_7})(\mathrm{CO})_2\{\eta^3\text{-}(^t\mathrm{Bu})\mathrm{CPC}(^t\mathrm{Bu})\mathrm{PFBF_3}\}]$.

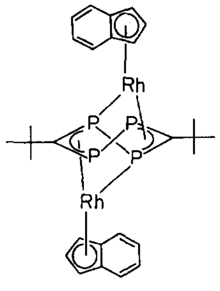
must stem from the metathesis of ^{t}BuCP during the reaction.

The $^{31}P\{^1H\}$ NMR spectrum of complex **1** is unaffected by changes in temperature, and both ^{19}F and ^{11}B NMR spectra show broad singlets at $\delta(F) = -142.6$ ppm and $\delta(B) = -2$ ppm respectively, implying that **1** may have a different structure in solution from that in the solid state.

TABLE 1. Selected intramolecular distances (Å) and angles (°) with estimated standard deviations in parentheses

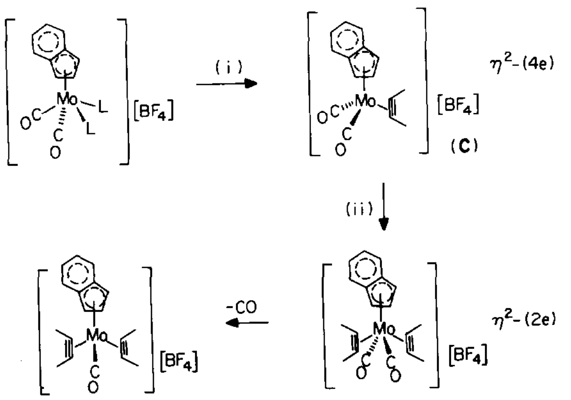
Bond lengths			
Mo(1)-C(20)	2.018(8)	Mo(1)-C(21)	1.977(10)
Mo(1)-P(2)	2.450(2)	Mo(1)-C(1)	2.308(7)
Mo(1)-C(2)	2.320(6)	Mo(1)-C(11)	2.374(8)
Mo(1)-C(12)	2.294(9)	Mo(1)-C(13)	2.268(10)
Mo(1)-C(14)	2.380(10)	Mo(1)-C(15)	2.458(9)
Mo(2)-C(41)	2.034(11)	Mo(2)-C(42)	1.982(8)
Mo(2)-P(3)	2.444(2)	Mo(2)-C(22)	2.279(8)
Mo(2)-C(23)	2.326(7)	Mo(2)-C(32)	2.403(10)
Mo(2)-C(33)	2.302(12)	Mo(2)-C(34)	2.258(9)
Mo(2)-C(35)	2.398(9)	Mo(2)-C(36)	2.485(9)
C(20)-O(1)	1.134(10)	C(21)-O(2)	1.138(13)
C(41)-O(3)	1.135(14)	C(42)-O(4)	1.158(10)
P(1)-F(2)	1.536(5)	P(1)-C(1)	1.739(9)
P(1)-C(2)	1.766(6)	P(2)-C(1)	1.823(8)
P(2)-C(2)	1.824(8)	P(3)-C(22)	1.825(9)
P(3)-C(23)	1.825(7)	P(4)-F(8)	1.539(5)
P(4)-C(22)	1.770(7)	P(4)-C(23)	1.740(9)
F(1)-B(1)	1.293(14)	F(2)-B(1)	1.477(11)
F(3)-B(1)	1.356(14)	F(4)-B(1)	1.287(14)
F(5)-B(2)	1.30(2)	F(6)-B(2)	1.29(2)
F(7)-B(2)	1.39(2)	F(8)-B(2)	1.431(13)
C(1)-C(3)	1.539(12)	C(2)-C(7)	1.523(10)
Mo(1)-Cen1	2.006	Mo(1)-Cen2	2.052
Mo(2)-Cen3	2.034	Mo(2)-Cen4	2.040
Bond angles			
F(2)-P(1)-C(1)	117.5(4)	F(2)-P(1)-C(2)	117.2(3)
C(1)-P(1)-C(2)	84.9(3)	C(1)-P(2)-C(2)	80.9(3)
C(22)-P(3)-C(23)	81.1(3)	F(8)-P(4)-C(22)	116.3(3)
F(8)-P(4)-C(23)	116.1(4)	C(22)-P(4)-C(23)	85.0(4)
P(1)-F(2)-B(1)	131.1(6)	P(4)-F(8)-B(2)	134.7(6)
P(1)-C(1)-P(2)	91.9(4)	P(1)-C(1)-C(3)	124.8(6)
P(2)-C(1)-C(3)	126.9(7)	P(1)-C(2)-P(2)	91.0(3)
P(1)-C(2)-C(7)	124.4(5)	P92)-C(2)-C(7)	126.6(5)
C(1)-C(3)-C(4)	108.4(9)	C(1)-C(3)-C(5)	109.2(8)
C(1)-C(3)-C(6)	111.3(8)	C(4)-C(3)-C(5)	109.6(9)
C(4)-C(3)-C(6)	106.7(9)	C(5)-C(3)-C(6)	111 (1)
F(1)-B(1)-F(2)	108(1)	F(1)-B(1)-F(3)	103(1)
F(1)-B(1)-F(4)	112.4(9)	F(2)-B(1)-F(3)	109.3(8)
F(2)-B(1)-F(4)	114.1(8)	F(3)-B(1)-F(4)	110(1)
F(5)-B(2)-F(6)	121(1)	F(5)-B(2)-F(7)	105 (1)
F(5)-B(2)-F(8)	112.8(8)	F(6)-B(2)-F(7)	98(1)
F(6)-B(2)-F(8)	112(1)	F(7)-B(2)-F(8)	106.3(9)
Cen1-Mo(1)-Cen2	141	Cen1-Mo(1)-C(20)	112
Cen1-Mo(1)-C(21)	109	Cen2-Mo(1)-C(20)	100
Cen2-Mo(1)-C(21)	99	C(20)-Mo(1)-C(21)	79
Cen3-Mo(2)-Cen4	142	Cen3-Mo(2)-C(41)	112
Cen3-Mo(2)-C(42)	110	Cen4-Mo(2)-C(41)	100
Cen4-Mo(2)-C(42)	97	C(41)-Mo(2)-C(42)	79
Mo(1)-C(20)-O(1)	172.6(8)	Mo(1)-C(21)-O(2)	174(1)
Mo(2)-C(41)-O(3)	173.8(8)	Mo(2)-C(42)-O(4)	174.0(8)

Cen1 is the centroid of C(11), C(12), C(13), C(14), C(15); Cen2 is the centroid of C(2), P(2), C(1); Cen3 is the centroid of C(32), C(33), C(34), C(35), C(36); Cen4 is the centroid of C(22), P(3), C(23).



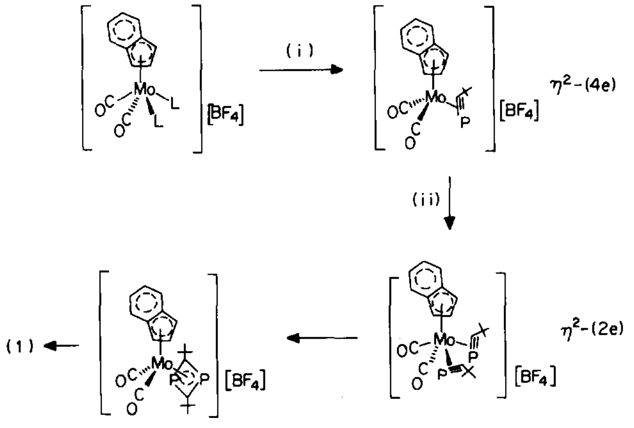
(4)

It is conceivable that in solution the structure involves an η^4-ligated diphosphacyclobutadiene ring A, Fig. 2, in which the positive charge becomes mainly localized to molybdenum. In structure B, on the contrary, the positive charge is concentrated on the phosphorus atom P(1), and the complex could then be regarded as containing a phosphenium ion which is interacting with the $[BF_4]^-$ anion, ultimately resulting the P–F bond formation and elongation of one B–F



(i) $MeC\equiv CMe$, CH_2Cl_2, RT; (L = MeCN)
(ii) $MeC\equiv CMe$

Scheme 1.



(i) ${}^tBuC\equiv P$, CH_2Cl_2, RT; L = MeCN
(ii) ${}^tBuC\equiv P$

Scheme 2.

TABLE 2. Fractional atomic coordinates ($\times 10^4$) with the estimated standard deviations in parentheses

	x	y	z
Mo(1)	1282.2(4)	791.7(3)	3424.0(4)
Mo(2)	4889.7(5)	2483.6(4)	2354.7(4)
P(1)	−81(1)	1695(1)	2320(1)
P(2)	1711(1)	1676(1)	2582(1)
P(3)	4268(1)	1251(1)	2390(1)
P(4)	5960(1)	1150(1)	2352(1)
F(1)	−1721(6)	2450(8)	600(4)
F(2)	−545(4)	2251(3)	1689(3)
F(3)	−1668(6)	3166(4)	1484(7)
F(4)	−2130(4)	2056(4)	1554(5)
F(5)	7701(5)	348(4)	2273(8)
F(6)	7198(7)	−607(5)	2810(7)
F(7)	6765(8)	−479(7)	1664(6)
F(8)	6204(4)	333(3)	2346(3)
O(1)	3112(4)	1330(5)	4688(4)
O(2)	2890(5)	−127(4)	3118(6)
O(3)	3820(5)	2864(5)	3557(4)
O(4)	2793(5)	2756(4)	1361(4)
C(1)	714(5)	1077(4)	2138(4)
C(2)	895(4)	1999(4)	3079(4)
C(3)	542(8)	640(5)	1390(5)
C(4)	−415(10)	225(7)	1228(6)
C(5)	495(11)	1155(7)	747(6)
C(6)	1309(11)	53(7)	1467(8)
C(7)	938(5)	2728(4)	3489(4)
C(8)	1224(7)	3331(4)	3003(6)
C(9)	1654(8)	2724(6)	4288(6)
C(10)	−45(6)	2919(5)	3563(5)
C(11)	−147(5)	159(4)	3311(5)
C(12)	640(6)	−343(5)	3464(5)
C(13)	1300(7)	−179(5)	4199(6)
C(14)	897(7)	417(5)	4536(5)
C(15)	−5(5)	619(4)	3988(4)
C(16)	−605(7)	1147(5)	4150(6)
C(17)	−332(9)	1425(7)	4874(7)
C(18)	510(12)	1265(7)	5387(7)
C(19)	1139(9)	792(7)	5284(6)
C(20)	2444(6)	1184(5)	4214(6)
C(21)	2287(6)	218(5)	3191(6)
C(22)	4882(5)	1431(4)	1686(4)
C(23)	5447(5)	1422(4)	3044(4)
C(24)	4551(7)	1251(5)	837(5)
C(25)	4495(19)	452(9)	727(8)
C(26)	3746(13)	1647(12)	399(7)
C(27)	5408(12)	1392(11)	507(8)
C(28)	5807(6)	1222(5)	3908(5)
C(29)	5680(12)	420(7)	3993(7)
C(30)	5378(17)	1634(12)	4377(8)
C(31)	6843(9)	1354(10)	4177(8)
C(32)	6059(6)	2966(5)	1826(5)
C(33)	5191(7)	3382(6)	1581(6)
C(34)	5009(7)	3705(5)	2225(7)
C(35)	5796(7)	3551(5)	2876(6)

TABLE 2. (continued)

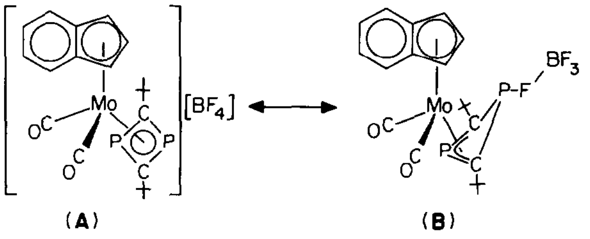
	x	y	z
C(36)	6444(6)	3098(4)	2608(5)
C(37)	7351(7)	2904(5)	3141(6)
C(38)	7621(9)	3187(6)	3898(7)
C(39)	6916(11)	3625(7)	4137(8)
C(40)	5996(9)	3783(7)	3629(8)
C(41)	4232(6)	2697(5)	3154(5)
C(42)	3568(6)	2622(5)	1702(5)
B(1)	−1545(7)	2453(6)	1335(6)
B(2)	7025(8)	−70(6)	2335(8)

bond (B(1)–F(2), 1.454(12) Å), the other B(1)–F bonds having a mean length of 1.319(14) Å.

An attempt to confirm the presence of A in solution by reaction of 1 with $LiBEt_3H$ was unsuccessful. Interestingly, analogous cationic η^4-cyclobutadiene complexes have been synthesized by Rheingold and coworkers [21] by treating η^3-oxocyclobutenyl complexes of molybdenum and tungsten with biscyclopentadienyl titanacyclobutane followed by protonation with $HBF_4 \cdot Et_2O$.

The formation of the η^3-1,3-diphosphacyclobutadienyl complex 1 is particularly interesting. As is summarized in Scheme 1, the reaction of, for example, but-2-yne with the labile complex $[Mo(\eta^5\text{-}C_9H_7)(CO)_2(CH_3CN)_2][BF_4]$ probably proceeds via the intermediate η^2-(4e)-donor alkyne complex C, and replacement of a second carbonyl group by a but-2-yne molecule is assisted by a switch $[\eta^2\text{-(4e)} \rightarrow \eta^2\text{-(2e)}]$ in the bonding mode of the alkyne. By analogy, it might have been expected that a similar sequence of reactions would taken place on reaction of the bis-acetonitrile complex with $^tBuC{\equiv}P$. However, instead of CO loss, head-to-tail coupling of the two coordinated (η^2-(2e)) phosphaalkyne ligands apparently occurs, to give initially a cationic η^4-1,3-diphosphacyclobutadiene complex, which then collapses through a BF_4^--phosphorus interaction to give the isolated complex 1 (Scheme 2).

The reasons for this difference in behaviour are not clear, and it is interesting to note that η^4-cyclobutadiene formation at a Mo(II) η^4-cyclopentadienyl substituted centre has not been observed, although it is important to note that, when a toluene solution of $[MoCl(\eta^5\text{-}C_5H_5)(\eta^2\text{-}PhC_2Ph)_2]$ is heated, disproportionation occurs [22], to yield as principal products a Mo(II) metallacyclopentatriene and the paramagnetic η^4-cyclobutadiene complex $[MoCl_2(\eta^5\text{-}C_5H_5)(\eta^4\text{-}C_4Ph_4)]$. Clearly, more work is needed to provide understanding of the formation of 1.



(A)
(solution)
(B)
(solid)

Fig. 2.

3. Experimental details

3.1. Reaction of $[Mo(\eta^5\text{-}C_9H_7)(CO)_2(CH_3CN)_2][BF_4]$ with $^tBuC{\equiv}P$

A solution of $[Mo(\eta^5\text{-}C_9H_7)(CO)_2(CH_3CN)_2][BF_4]$ (109 mg, 0.25 mmol) in CH_2Cl_2 (10 ml) was frozen and degassed at −197°C. After addition of $^tBuC{\equiv}P$ (69 mg, 0.69 mmol), the mixture was slowly warmed to room temperature and stirred for 16 h, during which a red–brown solution was formed. The solvent was removed under reduced pressure and the residue was kept under vacuum for a further 4 h to remove any remaining displaced acetonitrile. The resulting red–brown complex was redissolved in CH_2Cl_2 (2 ml). The $^{31}P\{^1H\}$ NMR spectrum (CH_2Cl_2) exhibited two strong singlets at δ(P [illegible].1 ppm and $\delta(P) = -149.4$ ppm and a temperature-dependent multiplet at $\delta(P) = -65.0$ to 40.1 ppm. After slow addition of petroleum ether (30–40°C), the solution was kept at room temperature for 4 days to afford pale-golden-yellow crystals. The mother liquor was syringed off and the crystals washed several times with petroleum ether (30–40°C) and dried in a flow of dry dinitrogen. The compound was identified as dicarbonyl-η^5-indenyl-η^3-[1,3-diphospha-2,4-di-tert-butyl-1-{μ-fluorotrifluoroborane}-cyclobutadiene]-molybdenum, (yield, 58 mg (54%); mp 175°C (dec)).

Anal. Found: C, 45.45; H, 4.21. $C_{21}H_{25}BF_4MoO_2P_2$ calc.: C, 45.52; H, 4.55%. IR (CH_2Cl_2): ν(CO) 2039m and 1976br, m cm^{-1}. $^{31}P\{^1H\}$ NMR (CH_2Cl_2): −122.1 (s) ppm. ^1H NMR ($CDCl_3$): δ 0.95 (s, 18H, ^tBu); 5.46 (t, 1H, H^1, $^1J(H^1H^2) = 2.93$ Hz); 6.13 (d, 2H, H^2, $^1J(H^1H^2) = 2.93$ Hz); 7.10–7.70 (m, 4H, aromatic H) ppm.

3.2. X-ray structural analysis of $[Mo(\eta^5\text{-}C_9H_7)\text{-}(CO)_2\text{-}\{\eta^3\text{-}(^tBuCPC(^tBu)PFBF_3\}]$

3.2.1. Crystal data

$C_{21}H_{25}BF_4MoO_2P_2$; $M = 554.1$; monoclinic; space group, $P2_1/c$; $a = 14.880(6)$ Å, $b = 18.280(8)$ Å and $c = 18.331(9)$ Å; $\beta = 107.77(4)°$; $F(000) = 2040$; $U = 4748.3$ Å^3; $Z = 8$; $D_c = 1.55$ g cm^{-3}; monochromated Mo Kα radiation, $\lambda = 0.71069$ Å; $\mu = 7.2$ cm^{-1}. Data were collected using a crystal of approximate dimensions 0.5 mm × 0.3 mm × 0.3 mm on an Enraf–Nonius CAD4 diffractometer. A total of 8963 unique reflections were measured for $2° < \theta < 25°$, $+h+k \pm l$, and 6079 reflections with $|F^2| > \sigma(F^2)$ were used in the refinement where $\sigma(F^2) = [\sigma^2(I) + (0.04I)^2]^{1/2}/Lp$. The structure was solved by routine heavy-atom methods and refined by the full-matrix least-squares method with anisotropic temperature factors. Hydrogen atoms were omitted. The weighting scheme was $w = 1/\sigma^2(F)$ and the final residuals were $R = 0.063$ and $R' = 0.092$. A final difference map had a maximum density of 1.1. electrons Å^{-3} close to the heavy atom. Programs from the Enraf–Nonius SDP-PLUS package were run on a PDP 11/34 computer.

Atom coordinates are listed in Table 2. A complete list of bond lengths and angles and a table of thermal parameters have been deposited at the Cambridge Crystallographic Data Centre.

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