

Cl3—Cr—N3	174.02 (5)	C7—C8—C9	111.4 (3)
N1—Cr—N2	82.56 (7)	C8—C9—C10	115.4 (3)
N1—Cr—N3	82.02 (7)	N2—C11—C12	116.0 (2)
N2—Cr—N3	82.74 (9)	C11—C12—C13	112.8 (2)
C1—N1—C6	110.8 (2)	C12—C13—C14	112.8 (3)
C1—N1—C7	109.2 (2)	N3—C15—C16	116.9 (3)
C6—N1—C7	109.8 (2)	C15—C16—C17	110.8 (3)
C2—N2—C3	111.3 (2)	C16—C17—C18	114.9 (4)
C2—N2—C11	109.2 (2)		
C6—N1—C1—C2	134.1 (2)	C4—N3—C5—C6	133.3 (2)
C7—N1—C1—C2	-104.8 (3)	C15—N3—C5—C6	-104.2 (3)
C1—N1—C6—C5	-68.5 (3)	C4—N3—C15—C16	51.7 (3)
C7—N1—C6—C5	170.8 (2)	C5—N3—C15—C16	-70.4 (3)
C1—N1—C7—C8	-74.8 (3)	N1—C1—C2—N2	-49.2 (3)
C6—N1—C7—C8	46.9 (3)	N2—C3—C4—N3	-49.2 (3)
C3—N2—C2—C1	-65.4 (3)	N3—C5—C6—N1	-47.4 (3)
C11—N2—C2—C1	173.9 (2)	N1—C7—C8—C9	168.3 (3)
C2—N2—C3—C4	133.9 (2)	C7—C8—C9—C10	168.3 (3)
C11—N2—C3—C4	-105.3 (2)	N2—C11—C12—C13	124.9 (3)
C2—N2—C11—C12	60.0 (3)	C11—C12—C13—C14	174.7 (3)
C3—N2—C11—C12	-62.0 (3)	N3—C15—C16—C17	168.4 (3)
C5—N3—C4—C3	-67.3 (3)	C15—C16—C17—C18	172.9 (3)
C15—N3—C4—C3	171.0 (2)		

Isotropic displacement parameters for the ring H atoms range 2.4 (5)–4.2 (6) Å² and the C—H distances range 0.89 (2)–0.99 (2) Å. The H atoms of the butyl groups have *B* values in the range 2.9 (5)–19 (2) Å² and C—H distances in the range 0.86 (2)–1.08 (4) Å. Programs used include the Enraf–Nonius *SDP* system (Frenz, 1978) and *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BK1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-trans-[N₃P₃Cl₄(C₆H₅)(PPh₂)]Mn-(CO)₂(bipy){P(OPh)₃}PF₆. A Cationic Manganese–Carbonyl Complex with a Phosphazenyolphosphine Ligand

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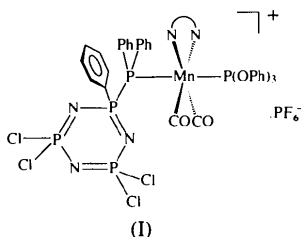
Abstract

The structure of *cis-trans*-(bipyridine-*N,N'*)dicarbonyl-[diphenyl(2-phenyl-4,4,6,6-tetrachloro-2-cyclophosphazenyolphosphine-*P*)(triphenyl phosphite-*P*)manganese(I) hexafluorophosphate, [Mn(C₁₈H₁₅O₃P)(C₁₀H₈N₂)(C₁₈H₁₅Cl₄N₃P₄)(CO)₂]PF₆, consists of discrete complex cations and PF₆⁻ anions linked by several weak C—H···O and C—H···F interactions. The cation is a very distorted octahedral manganese–dicarbonyl fragment having *cis-trans* stereochemistry and bearing triphenylphosphite, bipyridine and phosphazenyolphosphine [N₃P₃Cl₄(Ph)(PPh₂)] ligands. Analysis of the structure of the complex reveals that the coordination of the phosphazenyolphosphine ligand to the cationic manganese–carbonyl fragment induces only minor changes in the phosphazene ring, affecting only the P5—P3 bond length.

Comment

The presence of phosphine in a phosphazene ring molecule, attached either directly to the ring P atoms or indirectly *via* pendant groups, is of interest because the resulting ligands and their complexes may be useful as models for high polymeric analogues (Allcock, Manners, Mang & Parvaez, 1990). The phosphazenyolphosphine ligand N₃P₃Cl₄(C₆H₅)(PPh₂) has been coordinated previously to a neutral Cr(CO)₅ fragment and the X-ray structure determination of the resulting complex showed that no significant changes took place in the structure of the phosphazene ring upon coordination of the metal to the ligating PPh₂ group (Allcock, Manners, Mang & Parvaez, 1990). In order to determine the effect of a similar oc-

tahedral fragment, only this time with a positive charge, we prepared the title ionic complex, *cis-trans*- $[\{N_3P_3Cl_4(C_6H_5)(PPh_2)\}Mn(CO)_2(bipy)\{P(OPh)_3\}]PF_6$, (I), and determined its structure by X-ray diffraction.



The structure consists of discrete complex cations and octahedral PF_6^- anions (Fig. 1). The mean geometric parameters of the P environment are: P—N(ring) 1.580 (7), P—Cl 1.998 (3), P1—O 1.597 (5) Å, Mn—P1—O 116.5 (2), P—N—P(ring) 121.4 (4), N—P—N(ring) 117.7 (3) and Cl—P—Cl 100.5 (2)°. Although the position of the F atoms of the PF_6^- anion are affected by some disorder, as suggested by the anisotropic displacement parameters, it is possible that in the crystal there are several weak H-atom interactions between the cation and the anion. Thus, the two mutu-

ally *cis* F atoms (F2 and F5) of the PF_6^- anion interact weakly with the C25—H25 and C13—H13 bonds of the bipyridine ligand. The shortest anion—cation separation is H13···F5 [2.1 (2) Å]. There are also weak cation—cation hydrogen-bond contacts between atoms C14, C24 and C25 of the bipyridine ligand and the O atoms of the carbonyl ligands. A few very weak intramolecular hydrogen-bond interactions can also be observed within the cation, the shortest of which are between the phosphazene ring N atoms, N3 and N5, and the C—H bonds of the phenyl rings attached to P2 and P3.

The complex cation consists of an Mn atom bonded to two mutually *cis* carbonyl ligands, two N atoms of a chelated bipyridine ligand and two P atoms, one of the phosphite $P(OPh)_3$ and the other of the PPh_2 group of the phosphazenylyphosphine ligand. There are no intramolecular interactions between the Mn atom and the N or Cl atoms of the phosphazene ring. The stereochemistry around the Mn atom is only approximately octahedral and there are many distortions from ideal geometry. Thus the angles between the P2→Mn vector and the equatorial Mn—N2 [98.2 (2)°] and Mn—C2 [86.0 (2)°] bonds are significantly different from 90°. The large P2—Mn—N2 angle appears to be due mainly to the position in the molecule of the sterically demanding $-P(C_6H_5)_3N_3P_3Cl_4$ moiety relative to the Mn—N2 internuclear distance. The reason this bond is the more affected can be understood by considering that the torsion angle N2—Mn—P2—P3 is only $-24.5 (2)^\circ$ while N1—Mn—P2—P3 is $-102.6 (2)^\circ$ and Mn—P2—P3—N3 is $47.7 (2)^\circ$. The same explanation may be given for the deviation from linearity of the C2—Mn—N2 angle [171.4 (3)°]. Other deviations from octahedral geometry, such as the angle N1—Mn—N2 [78.3 (2)°], are due to the geometrical demands of the bidentate bipyridine ligand and are normal. The Mn—P2 bond distance [2.362 (3) Å] is rather large (Orpen, Brammer, Allen, Kennard, Watson & Taylor, 1989). This is similar to the corresponding Cr—P distance of 2.387 Å in the Cr analogue $[\{N_3P_3Cl_4(C_6H_5)(PPh_2)\}Cr(CO)_5]$ (Allcock, Manners, Mang & Parvaez, 1990). However, in the case of the manganese complex cation an important *trans* influence is expected due to the presence of the $P(OPh)_3$ ligand [P2—Mn—P1 is $174.34 (7)^\circ$ and the $^2J_{PMnP}$ coupling constant is 122 Hz].

The dimensions and angles of the $P_3N_3Cl_4$ ring (Table 2) are very close to those observed in both the free phosphazenylyphosphine $N_3P_3Cl_4(C_6H_5)(PPh_2)$ and in the chromium complex $[\{N_3P_3Cl_4(C_6H_5)(PPh_2)\}Cr(CO)_5]$ (Allcock, Manners, Mang & Parvaez, 1990). The N_3P_3 phosphazene ring is approximately planar, the largest deviation from the least-squares plane being 0.123 (7) Å for the atom N3. The angle between this plane and the plane of the bipyridine ligand is $68.3 (2)^\circ$. The bipyridine ligand is also planar with a maximum deviation from the ideal least-squares plane of $-0.0336 (9)$ Å for atom C14. Also, as in the chromium analogue, the

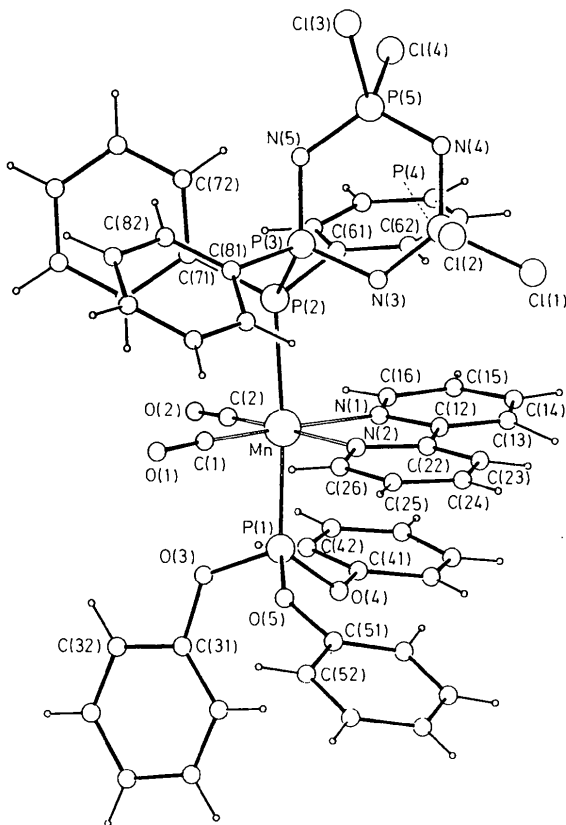


Fig. 1. EUCLID (Spek, 1982) plot of the title complex cation showing the atomic numbering scheme.

P3—P2 bond length in the cationic manganese complex [2.245 (2) Å] is significantly longer than in the free phosphine [2.199 (2) Å]. This suggests that the effect of the Cr(CO)₅ fragment on the P3—P5 bond is nearly the same as that of the cationic Mn(CO)₂(bipy){P(OPh)₃}⁺ fragment.

Refinement

Refinement on F^2
 $R(F) = 0.0442$
 $wR(F^2) = 0.1082$
 $S = 0.957$
 7302 reflections
 682 parameters
 H atoms refined as riding
 with common isotropic U

$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.055$
 $\Delta\rho_{\max} = 0.68 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{Å}^{-3}$
 Atomic scattering factors
 from *International Tables*
 for *Crystallography* (1992),
 Vol. C, Tables 4.2.6.8 and
 6.1.1.4)

Experimental

The phosphine [N₃P₃Cl₄(C₆H₅)(PPh₂)] (Allcock, Manners, Mang & Parvaez, 1990) and the complex [Mn(CO)₂(bipy)-{P(OPh)₃}] (Carriedo, Crespo, Riera, Valín, Moreiras & Solans, 1986) were prepared following the published procedures. The title compound was prepared by stirring a mixture of the phosphine (0.1 g, 0.185 mmol) with the complex (0.09 g, 0.128 mmol) and TlPF₆ (0.1 g) in CH₂Cl₂ (15 ml) for 2.5 h at room temperature, filtering the resulting mixture, concentrating it *in vacuo* and adding diethyl ether to give a yellow precipitate (0.1 g, 60%). The product was crystallized from CH₂Cl₂/ether (slow diffusion). Relevant spectroscopic data: FTIR spectrum (cm⁻¹): $\nu(\text{CO})$ absorptions (in CH₂Cl₂ solution), 1971(s), 1905(s); other bands (nujol mull), 1197(vs) [$\nu(\text{PN})$, with shoulders of medium intensity corresponding to the P(OPh)₃ ligand], 952(w), 932(m), 907(w) [P(OPh)₃], 842(vs) [$\nu(\text{PF})$ of PF₆⁻]. ³¹P NMR (in CH₂Cl₂, measured with reference to down-field external aqueous H₃PO₄): 18.0 (dd, ²J_{PNP} = 16 Hz, ³J_{PPNP} = 8 Hz, PCl₂); 31.9 [ddt, ¹J_{PP} = 85 Hz, ²J_{PNP} = 16 Hz, ³J_{PPMnP} = 8 Hz, P(Ph)], 50.3 (dd, ¹J_{PP} = 85 Hz, ²J_{PMnP} = 122 Hz, the signal is too broad to see the ³J_{PPNP} of 8 Hz, PPh₂), 154 [d, broad, ²J_{PMnP} = 122 Hz, P(OPh)₃].

Crystal data

[Mn(C₁₈H₁₅O₃P)(C₁₀H₈-
 N₂)(C₁₈H₁₅Cl₄N₃P₄)-
 (CO)₂]PF₆

$M_r = 1261.39$

Triclinic

$P1$

$a = 10.563 (2) \text{ Å}$

$b = 10.603 (3) \text{ Å}$

$c = 12.250 (3) \text{ Å}$

$\alpha = 97.68 (4)^\circ$

$\beta = 99.34 (3)^\circ$

$\gamma = 93.11 (2)^\circ$

$V = 1337.6 (5) \text{ Å}^3$

$Z = 1$

$D_x = 1.566 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ Å}$

Cell parameters from 25

reflections

$\theta = 15\text{--}20^\circ$

$\mu = 0.698 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prismatic plate

$0.13 \times 0.13 \times 0.07 \text{ mm}$

Yellow

$R_{\text{int}} = 0.050$

$\theta_{\text{max}} = 24.97^\circ$

$h = -10 \rightarrow 12$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 14$

3 standard reflections

monitored every 200

reflections

frequency: 60 min

intensity variation:

12.00%

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Mn1	0.0000	0.0000	0.0000	0.0295 (2)
C11	-0.4009 (2)	-0.4136 (2)	-0.2245 (2)	0.0591 (5)
C12	-0.2239 (2)	-0.5486 (2)	-0.3713 (2)	0.0771 (7)
C13	-0.3004 (3)	-0.2937 (2)	-0.6486 (2)	0.0756 (7)
C14	-0.4416 (2)	-0.0979 (2)	-0.5158 (2)	0.0705 (6)
P1	0.0908 (2)	0.0135 (2)	0.1773 (2)	0.0373 (4)
P2	-0.0858 (2)	0.0050 (2)	-0.1897 (2)	0.0290 (4)
P3	-0.1010 (2)	-0.1726 (2)	-0.3148 (2)	0.0299 (4)
P4	-0.2821 (2)	-0.3748 (2)	-0.3271 (2)	0.0393 (4)
P5	-0.3084 (2)	-0.2227 (2)	-0.4901 (2)	0.0398 (4)
P6	-0.5638 (2)	-0.5400 (2)	-0.9677 (2)	0.0669 (6)
F1	-0.4183 (7)	-0.5677 (8)	-0.9821 (8)	0.155 (3)
F2	-0.6994 (6)	-0.5224 (8)	-0.946 (2)	0.203 (5)
F3	-0.5973 (8)	-0.6842 (7)	-0.989 (1)	0.169 (4)
F4	-0.5930 (9)	-0.552 (2)	-1.0960 (8)	0.190 (4)
F5	-0.521 (1)	-0.4030 (8)	-0.9504 (13)	0.235 (6)
F6	-0.520 (1)	-0.542 (2)	-0.8444 (8)	0.233 (6)
O1	0.2605 (5)	-0.0284 (5)	-0.0491 (5)	0.069 (2)
O2	0.0296 (5)	0.2814 (5)	0.0241 (5)	0.060 (2)
O3	0.2038 (5)	0.1263 (5)	0.2105 (4)	0.052 (2)
O4	0.0095 (5)	0.0287 (4)	0.2772 (4)	0.046 (2)
O5	0.1699 (5)	-0.1026 (5)	0.2091 (4)	0.056 (2)
N1	-0.1835 (5)	-0.0043 (5)	0.0354 (4)	0.029 (2)
N2	-0.0391 (5)	-0.1909 (4)	0.0028 (4)	0.035 (2)
N3	-0.1656 (5)	-0.2856 (5)	-0.2601 (5)	0.036 (2)
N4	-0.3641 (6)	-0.3313 (6)	-0.4320 (5)	0.050 (2)
N5	-0.1757 (5)	-0.1483 (5)	-0.4326 (5)	0.040 (2)
C1	0.1590 (7)	-0.0146 (6)	-0.0316 (6)	0.037 (2)
C2	0.0189 (6)	0.1727 (7)	0.0131 (5)	0.035 (2)
C12	-0.2360 (6)	-0.1209 (7)	0.0464 (6)	0.044 (2)
C13	-0.3598 (7)	-0.1367 (8)	0.0713 (7)	0.054 (2)
C14	-0.4305 (7)	-0.0337 (9)	0.0807 (7)	0.061 (2)
C15	-0.3798 (7)	0.0855 (8)	0.0708 (6)	0.049 (2)
C16	-0.2548 (6)	0.0955 (7)	0.0490 (5)	0.039 (2)
C22	-0.1533 (7)	-0.2281 (6)	0.0289 (6)	0.042 (2)
C23	-0.1885 (8)	-0.3503 (7)	0.0377 (8)	0.059 (2)
C24	-0.1024 (9)	-0.4425 (8)	0.0228 (8)	0.070 (2)
C25	0.0136 (9)	-0.4092 (7)	-0.0029 (7)	0.061 (2)
C26	0.0442 (7)	-0.2827 (7)	-0.0111 (6)	0.049 (2)
C31	0.2781 (6)	0.1700 (7)	0.3148 (6)	0.047 (2)
C32	0.3497 (7)	0.2833 (7)	0.3180 (7)	0.053 (2)
C33	0.4257 (8)	0.3347 (8)	0.4163 (8)	0.068 (2)
C34	0.4355 (8)	0.2757 (9)	0.5110 (8)	0.067 (2)
C35	0.3622 (9)	0.1629 (9)	0.5024 (7)	0.066 (2)
C36	0.2841 (8)	0.1107 (8)	0.4073 (6)	0.063 (2)
C41	-0.0797 (7)	0.1206 (7)	0.2916 (6)	0.046 (2)
C42	-0.0562 (7)	0.2442 (7)	0.2725 (6)	0.049 (2)
C43	-0.1499 (9)	0.3265 (7)	0.2878 (6)	0.056 (2)
C44	-0.261 (1)	0.2914 (9)	0.3220 (7)	0.069 (2)
C45	-0.2809 (8)	0.1674 (9)	0.3437 (7)	0.064 (2)
C46	-0.1910 (7)	0.0804 (7)	0.3287 (6)	0.051 (2)
C51	0.1579 (7)	-0.2105 (6)	0.2613 (5)	0.043 (2)
C52	0.2609 (9)	-0.2807 (8)	0.2678 (8)	0.075 (3)
C53	0.260 (2)	-0.388 (1)	0.319 (2)	0.103 (4)

C54	0.151 (2)	-0.4236 (9)	0.3609 (9)	0.093 (3)
C55	0.0486 (9)	-0.3518 (8)	0.3561 (8)	0.073 (3)
C56	0.0511 (8)	-0.2443 (7)	0.3044 (8)	0.064 (2)
C61	-0.2531 (6)	0.0461 (6)	-0.2080 (5)	0.032 (2)
C62	-0.3509 (6)	-0.0415 (7)	-0.2045 (5)	0.040 (2)
C63	-0.4779 (7)	-0.0079 (9)	-0.2029 (6)	0.057 (2)
C64	-0.4983 (8)	0.118 (2)	-0.2022 (7)	0.069 (3)
C65	-0.4033 (9)	0.2075 (9)	-0.2059 (7)	0.066 (2)
C66	-0.2812 (7)	0.1734 (7)	-0.2091 (6)	0.045 (2)
C71	-0.0081 (6)	0.1185 (6)	-0.2636 (5)	0.031 (2)
C72	-0.0666 (7)	0.1461 (6)	-0.3651 (6)	0.042 (2)
C73	-0.0110 (8)	0.2333 (7)	-0.4209 (6)	0.049 (2)
C74	0.1086 (8)	0.2957 (7)	-0.3725 (7)	0.056 (2)
C75	0.1695 (7)	0.2703 (7)	-0.2728 (7)	0.056 (2)
C76	0.1127 (7)	0.1825 (7)	-0.2157 (6)	0.045 (2)
C81	0.0608 (6)	-0.2053 (6)	-0.3295 (6)	0.033 (2)
C82	0.1279 (7)	-0.1356 (7)	-0.3911 (6)	0.045 (2)
C83	0.2509 (7)	-0.1675 (8)	-0.4045 (7)	0.059 (2)
C84	0.3022 (7)	-0.2665 (8)	-0.3614 (7)	0.060 (2)
C85	0.2386 (7)	-0.3369 (7)	-0.2979 (7)	0.055 (2)
C86	0.1148 (7)	-0.3060 (7)	-0.2831 (7)	0.053 (2)

Table 2. Selected geometric parameters (Å, °)

Mn1—C1	1.793 (7)	P1—O4	1.603 (5)
Mn1—C2	1.814 (7)	P1—O3	1.606 (5)
Mn1—N2	2.050 (5)	P2—P3	2.245 (2)
Mn1—N1	2.053 (5)	P3—N5	1.588 (6)
Mn1—P1	2.210 (2)	P3—N3	1.621 (5)
Mn1—P2	2.362 (2)	P4—N3	1.556 (5)
Cl1—P4	1.979 (3)	P4—N4	1.566 (7)
Cl2—P4	2.008 (2)	P5—N4	1.569 (6)
Cl3—P5	2.004 (3)	P5—N5	1.579 (6)
Cl4—P5	2.000 (3)	O1—C1	1.141 (7)
P1—O5	1.581 (5)	O2—C2	1.139 (7)
C2—Mn1—N2	171.4 (3)	O4—P1—Mn1	122.4 (2)
C1—Mn1—N1	173.8 (3)	O3—P1—Mn1	111.3 (2)
C1—Mn1—P1	86.2 (2)	P3—P2—Mn1	120.1 (1)
C2—Mn1—P1	88.9 (2)	N5—P3—N3	115.5 (3)
N2—Mn1—P1	87.2 (2)	N3—P4—N4	119.2 (3)
N1—Mn1—P1	93.6 (2)	Cl1—P4—Cl2	101.0 (2)
C1—Mn1—P2	91.5 (2)	N4—P5—N5	118.5 (3)
C2—Mn1—P2	86.0 (2)	Cl4—P5—Cl3	100.0 (2)
N2—Mn1—P2	98.2 (2)	P4—N3—P3	121.1 (3)
N1—Mn1—P2	89.2 (2)	P4—N4—P5	120.3 (4)
P1—Mn1—P2	174.3 (1)	P5—N5—P3	122.8 (3)
O5—P1—Mn1	115.8 (2)		
N2—Mn1—P2—P3	-24.5 (2)	Mn1—P2—P3—N3	47.7 (2)
N1—Mn1—P2—P3	-102.6 (2)		

Data were collected (Enraf–Nonius, 1989) with profile analysis over all reflections (Lehmann & Larsen, 1974; Grant & Gabe, 1978). Friedel pairs were measured from 0 to 20° (θ). Standard drift corrections were applied. Cell refinement was performed using *DIRDIF CRYSDA* (Beurskens *et al.*, 1992). *DATAR* (Van der Maelen Uría, 1991) was used for data reduction. The structure was completed by the *DIRDIF* phase-expansion procedure. Isotropic least-squares refinement used a local version of *SHELX76* (Sheldrick, 1976; Van der Maelen Uría, 1991). Final refinement used *SHELXL93* (Sheldrick, 1993). The absolute configuration was checked with a new version of the *BIJVOET* program (Beurskens, Noordik & Beurskens, 1980) giving a Bijvoet coefficient of 0.933 (2) for the strongest 100 Friedel pairs. Further geometrical calculations were made with *PARST* (Nardelli, 1983) and Fig. 1 was produced using the *EUCLID* package (Spek, 1982).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and hydrogen-bonding geometry have been deposited with the IUCr (Reference: HR1019). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[MoH₄(dppe)₂].thf (dppe = Ph₂PCH₂CH₂PPh₂; thf = Tetrahydrofuran)

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Abstract

The structure of tetrahydridobis[1,2-bis(diphenylphosphino)ethane-*P,P'*]molybdenum(IV). [MoH₄(dppe)₂] (dppe = Ph₂PCH₂CH₂PPh₂), is reported as