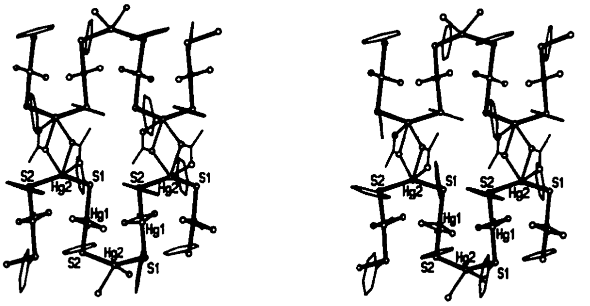

Fig. 2. Stereoview of part of the layers in the structure. Infinite (—Hg—*R*S—)$_n$ chains, running parallel to the *b* axis, are linked by the acetate ligands. Hg—O bonds >2·8 Å are not drawn.

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# Structure of a Chelating Phosphine Rhenium(I) Complex: *fac*-Tricarbonylchloro-[1,1,1-tris(diphenylphosphinemethyl)ethane]rhenium(I)

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**Abstract.** [ReCl(CO)$_3$(C$_{41}$H$_{39}$P$_3$)], $M_r = 930·372$, triclinic, $P\bar{1}$, $a = 9·804\ (3)$, $b = 9·815\ (2)$, $c = 21·339\ (5)$ Å, $\alpha = 103·74\ (2)$, $\beta = 93·25\ (2)$, $\gamma = 97·62\ (2)°$, $U = 1968·7\ (4)$ Å$^3$, $Z = 2$, $D_m = 1·58$, $D_x = 1·57$ Mg m$^{-3}$ (by flotation in CCl$_4$/hexane mixture), $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·9907$ mm$^{-1}$, $F(000) = 928$, $T = 296\ (4)$ K, $R(F) = 0·55$ for 7317 observed reflections [$I > 3\sigma(I)$]. The coordination geometry about Re is a six-coordinate octahedron comprising Cl, two P atoms and three carbonyl groups. The six-membered ring in this complex is characterized by a large ring strain and takes a chair form.

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**Introduction.** The reaction between an organometallic cluster compound and a multidentate ligand, such as 1,2-bis(diphenylphosphino)ethane, bis(diphenylphosphino)methane (Lugan, Bonnet & Ibers, 1985), tris(diphenylphosphino)methane (Harding, Nicholls & Smith, 1982; Bahsoun, Osborn, Voelker, Bonnet & Lavigne 1982) and tris(dibutylphosphino)methylsilane (de Boer, van Doorn & Masters, 1978), has attracted much attention in recent years. The main thrust in this area is the belief that the multidentate ligand chelated cluster will maintain the integrity of the metal cluster core under severe reaction conditions. Furthermore, the multidentate ligand may serve as a template in synthesizing new cluster compounds. To enhance our knowledge concerning the chemistry of multidentate ligands, we investigated the reaction of a multidentate tripod ligand, 1,1,1-tris(diphenylphosphinomethyl)ethane (tdppme) with $Re_2(CO)_{10}$ under photolytic conditions. This report presents the structure of the major monometallic complex, which reflects the stereochemistry of the tripod ligand tdppme.

**Experimental.** 30 ml dichloromethane solution containing 528 mg (0·81 mmol) of $Re_2(CO)_{10}$ and 560 mg (0·90 mmol) tdppme tripod ligand was photolyzed using a 450 W mercury lamp for 8 hours. After evaporating the solvent, the residue was separated by preparative thin-layer chromatography using dichloromethane and *n*-hexane mixed solvent (2:1). The fraction with $R_f = 0{\cdot}58$ is the major product (225 mg). It was recrystallized from a mixture of chloroform and *n*-hexane. Its $^{31}P$ NMR consists of two peaks at −11·26 and −11·43 p.p.m. with intensity ratio of 2:1.

Experimental data and structure solution parameters together with the refinement procedures are listed in Table 1. All hydrogen atoms were located theoretically and their temperature factors were initially assigned according to $B_H = B_n + 1$ where $n$ is the atom to which the H atom is bonded (Lee, Lee, Juang & Chung, 1985). With the H atoms included in the last cycle of least-squares calculations, $R(F)$ reduced to 0·055.

Table 1. *Experimental data and structure refinement parameters*

| | |
|---|---|
| Crystal size (mm) | 0·3 × 0·3 × 0·25 |
| Diffractometer and data collection technique used | $\omega$–$2\theta$ scan, four-circle diffractometer (Nonius CAD-4) with graphite monochromator |
| Scan width ($2\theta$) (°) | $2(0{\cdot}6 + 0{\cdot}35\tan\theta)$ |
| Number and $\theta$ range used for measuring lattice parameters | 25 reflections with $11 < 2\theta < 20°$ |
| Max., min. transmission factor | 0·7806, 0·7429 |
| $2\theta$ range in intensity measurement | 2 to 55° |
| Range of *hkl* | 0, −13, −28 to 13,13,28 |
| Standard reflections and intensity variation | $0,\bar{3},11$, $0,3,\overline{11}$, $5\bar{3}\bar{2}$, $\bar{5}32$ < 3% |
| Number of reflections measured | 12 300 |
| Number of unique reflections | 7317 [$I > 3\sigma(I)$] |
| Method used to solve the structure | Patterson and Fourier methods |
| $(\Delta\rho)_{max}$ and $(\Delta\rho)_{min}$ in final difference Fourier synthesis | 1·35 (around Re), 0·135 e Å$^{-3}$ |
| Parameters refined, nonhydrogen atoms | Coordinates and anisotropic temperature factors (468 parameters) |
| hydrogen atoms | Isotropic temperature factors (39 parameters) |
| Quantity minimized | $\sum w(\Delta F)^2$; $w = 1/\sigma_F^2$ |
| Atomic scattering factors $f'$ and $f''$ | *International Tables for X-ray Crystallography* (1968) |
| Numbering of reflections per parameter | 14 |
| $R(F)$ and $wR$ | 0·055 and 0·059 |
| $S$ | 3·779 |
| $R_{int}$ | 0·032 |
| Average, max. $\Delta/\sigma$ | 0·06, 0·71 |
| Programs used | *XTAL*83 (Stewart, Hall, Alden, Olthof-Hazekamp, Doherty, Pagoaga & Norden, 1983); *ORTEP*II (Johnson, 1976). |
| Computers | CDC Cyber-840 and IBM PC-XT |

**Discussion.** The atomic coordinates and temperature factors are listed in Table 2.* Fig. 1 illustrates the geometry and labeling of the molecule together with its stereoview. The coordination geometry about Re is an approximate octahedron comprising three carbonyl groups, Cl and two P atoms. As can be seen from Fig. 2, the deviations of Re and C(3) from the least-squares plane through P(1), C(1), P(2) and C(2) are −1·064 and +0·703 Å respectively. Thus, the six-membered ring Re—P(1)—C(1)—C(3)—C(2)—P(2) exhibits a chair conformation.

The Re—Cl bond length, 2·494 (3) Å, and Re—C(carbonyl) bond lengths ranging from 1·854 (10) to 1·926 (12) Å are slightly shorter than those in rhenium pentacarbonyl chloride (Cotton & Daniels, 1983). The C≡O bond lengths are consistent with the normal carbonyl bond length within one standard deviation. The C—C bond lengths are normal. Bond distances and angles are given in Table 3.

One of the phosphorus coordination site atoms, P(3), in the tripod ligand tdppme [$(PPh_2CH_2)_3CCH_3$] is not coordinated to any rhenium. Examination of the distances among the phosphorus atoms, P(1), P(2), P(3), reveals that they form an approximate isosceles triangle. The distance between the two coordinated phosphorus atoms, P(1) and P(2), is 3·430 (3) Å. P(3) is 5·045 (3) and 4·938 (4) Å away from P(1) and P(2), respectively. Steric factors prevent P(3) from coordinating to the central rhenium atom.

It is well established that in chelate coordination compounds, the most favorable geometry is a five-membered ring system. In organometallic chelating compounds, the five-membered ring structure also seems to be preferred. Some examples of rhenium compounds with bidentate phosphine ligands will be given to illustrate the basic ring structure.

Dppm [bis(diphenylphosphino)methane] can easily coordinate to two rhenium atoms as in $Re_2Cl_4(\mu_2\text{-}dppm)_2$, $Re_2Cl_3(OC_2H_5)(dppm)_2$ (Barder, Cotton,

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* Lists of structure factors, anisotropic thermal parameters, C—C bond distances and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43237 (62 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Lewis, Schwotzer, Tetrick & Walton, 1984), $Re_2(CO)_6(\mu_2\text{-dppm})H_2$, $Re_2(CO)_6(\mu_2\text{-dppm})(NCHMe)$ (Mays, Prest & Raithby, 1980), and $(\mu\text{-H})(\mu\text{-OH})Re_2(CO)_6(\mu_2\text{-dppm})$ (Lee, Pennington, Cordes & Brown, 1984). In these dirhenium chelate ring structures, the Re—P bond lengths are in the range of 2·45–2·48 Å and the P—P distances are in the neighborhood of 3·0 Å. The P—C—P angles are close to the expected tetrahedral angle of 109°.

Dppe [bis(diphenylphosphino)ethane)] tends to form complexes with a single rhenium to complete a five-membered ring, as in the compounds $ReCl(dppe)_2$ (Hughes, Pombeiro, Pickett & Richards, 1980) and *fac*-$Re(CO)_3(dppe)\{C(CO)SiPh_3\}$ (Anglin, Calhoun & Graham, 1977). In these complexes, Re—P bond lengths are 2·25–2·45 Å, the P—Re—P angles are 80–82°, and the P—P distances 3·0–3·2 Å. Dppe can also coordinate to two rhenium atoms to form a six-membered ring structure as in $Re_2(CO)_8(dppe)$, $Re_2(CO)_6(\mu\text{-H})(\mu\text{-OH})(dppe)$ and $Re_2(CO)_6(\mu\text{-H})(\mu\text{-Cl})(dppe)$ (Lee, Pennington, Cordes & Brown, 1984). However, no detailed structure is known.

Table 2. *Atomic positional and thermal parameters of non-hydrogen atoms.*

$B_{eq} = \frac{4}{3}\sum_i\sum_j a_i a_j B_{ij}$, where $a_i$'s are the length vectors in direct space.

| | $x$ | $y$ | $z$ | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Re | 0·03271 (4) | 0·10916 (4) | 0·20113 (2) | 3·1 |
| P(1) | 0·1710 (2) | 0·3445 (3) | 0·2514 (1) | 2·9 (1) |
| P(2) | −0·1389 (2) | 0·2328 (3) | 0·1585 (1) | 2·8 (1) |
| P(3) | 0·1744 (3) | 0·3762 (3) | 0·6440 (1) | 3·9 (2) |
| Cl | −0·0875 (3) | 0·1599 (3) | 0·3022 (1) | 4·3 (2) |
| C(1) | 0·0655 (9) | 0·4787 (10) | 0·2829 (4) | 3·3 (5) |
| C(2) | 0·1666 (9) | 0·6064 (10) | 0·7830 (4) | 3·2 (5) |
| C(3) | 0·0481 (9) | 0·4829 (9) | 0·7595 (4) | 3·3 (5) |
| C(4) | 0·0128 (10) | 0·5614 (10) | 0·1815 (4) | 4·0 (6) |
| C(5) | 0·1095 (10) | 0·3578 (10) | 0·7215 (4) | 3·7 (6) |
| C(111) | 0·2965 (8) | 0·4308 (10) | 0·2065 (4) | 3·0 (5) |
| C(112) | 0·3237 (10) | 0·3643 (11) | 0·1461 (5) | 4·2 (6) |
| C(113) | 0·4185 (11) | 0·4307 (13) | 0·1126 (5) | 5·1 (8) |
| C(114) | 0·4863 (11) | 0·5632 (13) | 0·1399 (6) | 5·3 (8) |
| C(115) | 0·4590 (11) | 0·6281 (12) | 0·1997 (6) | 6·0 (9) |
| C(116) | 0·3663 (11) | 0·5664 (11) | 0·2357 (5) | 4·7 (7) |
| C(121) | 0·2818 (9) | 0·3425 (10) | 0·3232 (5) | 3·8 (6) |
| C(122) | 0·4179 (11) | 0·3219 (12) | 0·3161 (5) | 4·6 (7) |
| C(123) | 0·5017 (11) | 0·3111 (12) | 0·3685 (5) | 5·1 (8) |
| C(124) | 0·4505 (12) | 0·3190 (15) | 0·4274 (6) | 6·3 (9) |
| C(125) | 0·3158 (13) | 0·3393 (19) | 0·4342 (5) | 8·2 (12) |
| C(126) | 0·2310 (11) | 0·3490 (16) | 0·3820 (5) | 6·2 (9) |
| C(211) | −0·1327 (10) | 0·2829 (10) | 0·0809 (4) | 3·7 (6) |
| C(212) | −0·2410 (12) | 0·3444 (13) | 0·0613 (5) | 5·1 (8) |
| C(213) | −0·2428 (13) | 0·3823 (14) | 0·0052 (6) | 6·6 (9) |
| C(214) | −0·1407 (15) | 0·3595 (14) | −0·0331 (5) | 7·3 (10) |
| C(215) | −0·0322 (13) | 0·2985 (13) | −0·0155 (5) | 6·2 (9) |
| C(216) | −0·0282 (11) | 0·2592 (12) | 0·0420 (5) | 4·7 (7) |
| C(221) | −0·3075 (9) | 0·1199 (10) | 0·1443 (5) | 3·5 (6) |
| C(222) | −0·3488 (11) | 0·0292 (12) | 0·0840 (5) | 5·0 (7) |
| C(223) | −0·4702 (12) | −0·0657 (12) | 0·0753 (5) | 5·9 (8) |
| C(224) | −0·5477 (12) | −0·0739 (13) | 0·1234 (6) | 6·2 (9) |
| C(225) | −0·5087 (11) | 0·0117 (13) | 0·1792 (7) | 7·0 (10) |
| C(226) | −0·3852 (11) | 0·1142 (13) | 0·1978 (7) | 6·0 (9) |
| C(311) | 0·3049 (9) | 0·2596 (10) | 0·6345 (4) | 3·5 (6) |
| C(312) | 0·3028 (11) | 0·1413 (11) | 0·6597 (5) | 4·6 (7) |
| C(313) | 0·4080 (12) | 0·0621 (12) | 0·6507 (5) | 5·5 (8) |
| C(314) | 0·5169 (12) | 0·0973 (14) | 0·6193 (6) | 6·0 (9) |
| C(315) | 0·5236 (11) | 0·2143 (14) | 0·5951 (5) | 5·7 (9) |
| C(316) | 0·4176 (10) | 0·2967 (12) | 0·6038 (5) | 4·5 (7) |
| C(321) | 0·0319 (11) | 0·2693 (14) | 0·5852 (5) | 5·6 (8) |
| C(322) | −0·0294 (12) | 0·1389 (16) | 0·5863 (6) | 7·8 (10) |
| C(323) | −0·1365 (15) | 0·0688 (20) | 0·5409 (9) | 12·3 (16) |
| C(324) | −0·1754 (18) | 0·1305 (27) | 0·4928 (9) | 16·4 (20) |
| C(325) | −0·1217 (18) | 0·2513 (27) | 0·4901 (7) | 13·6 (20) |
| C(326) | −0·0134 (15) | 0·3279 (21) | 0·5381 (7) | 9·5 (14) |
| C(401) | 0·1208 (9) | 0·0592 (10) | 0·1262 (5) | 4·1 (6) |
| C(402) | 0·1502 (10) | 0·0116 (11) | 0·2449 (6) | 4·9 (8) |
| C(403) | −0·0902 (10) | −0·0659 (9) | 0·1699 (5) | 3·9 (6) |
| O(401) | 0·1719 (8) | 0·0225 (8) | 0·0778 (4) | 5·9 (6) |
| O(402) | 0·2169 (8) | −0·0440 (9) | 0·2734 (5) | 6·9 (7) |
| O(403) | −0·1696 (8) | −0·1691 (7) | 0·1549 (4) | 5·7 (5) |

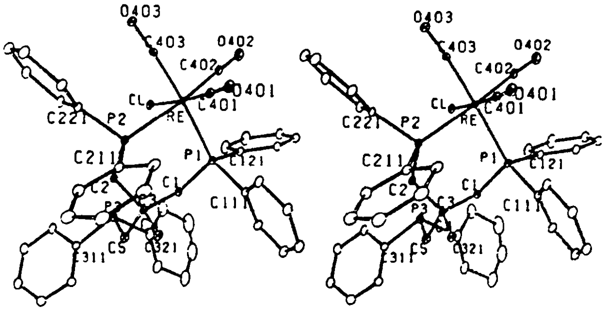

Fig. 1. Stereoview of title complex, showing the atomic numbering scheme. Thermal ellipsoids are depicted at the 50% probability level. H atoms attached to C atoms are not plotted.

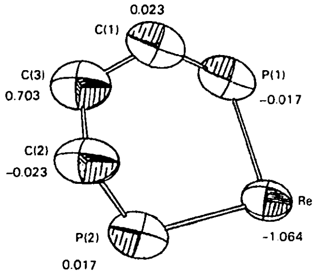

Fig. 2. Perspective view of partial structure of the title complex, showing the six-membered ring to be a chair form and showing displacements of atoms from the plane through P(1), C(1), C(2) and P(2) (Å). The e.s.d.'s are around 0·005 Å. Thermal ellipsoids are depicted at the 50% probability level.

Table 3. *Bond distances* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Bond distances and bond angles (*trans*) around the coordination sphere | | | |
| Re—P(1) | 2·480 (2) | Re—C(401) | 1·854 (10) |
| Re—P(2) | 2·454 (3) | Re—C(402) | 1·926 (12) |
| Re—Cl | 2·494 (3) | Re—C(403) | 1·914 (8) |
| P(1)—Re—C(401) | 99·8 (3) | Cl—Re—C(401) | 176·3 (3) |
| P(1)—Re—C(402) | 92·9 (3) | Cl—Re—C(402) | 87·0 (4) |
| P(1)—Re—C(403) | 172·9 (3) | Cl—Re—C(403) | 90·0 (3) |
| P(2)—Re—Cl | 84·76 (9) | C(401)—Re—C(402) | 90·6 (5) |
| P(2)—Re—C(401) | 97·5 (3) | C(401)—Re—C(403) | 87·1 (4) |
| P(2)—Re—C(403) | 89·2 (3) | C(402)—Re—C(403) | 88·8 (4) |
| P(2)—Re—C(402) | 171·5 (4) | | |
| P—C bond distances | | | |
| P(1)—C(1) | 1·810 (10) | P(2)—C(221) | 1·839 (10) |
| P(1)—C(111) | 1·842 (9) | P(3)—C(5) | 1·845 (10) |
| P(1)—C(121) | 1·833 (10) | P(3)—C(311) | 1·815 (10) |
| P(2)—C(2) | 1·827 (9) | P(3)—C(321) | 1·848 (10) |
| Other relevant bond distances and bond angles | | | |
| C(1)—C(3) | 1·537 (13) | C(401)—O(401) | 1·175 (13) |
| C(2)—C(3) | 1·530 (11) | C(402)—O(402) | 1·142 (16) |
| C(3)—C(5) | 1·519 (13) | C(403)—O(403) | 1·159 (11) |
| C(3)—C(4) | 1·550 (14) | | |

Rhenium phosphine chelate complexes involving other ring systems are very rare. Brown *et al.* recently reported the structure of Re$_2$(CO)$_5$(dppm)($\mu$-$\eta^1$,$\eta^2$-CH=CH$_2$)($\mu$-$\eta^1$,$\eta^2$-C=CPh) (Lee, Pennington, Cordes & Brown, 1985). The dppm and one of the rheniums form a four-membered ring. The Re—P bond lengths of 2·42–2·44 Å are normal. However, the bite angle P—Re—P of 68·9° is substantially less than that in the five-membered ring complex. The P—C—P angle of 96·4° is 13° less than the regular tetrahedral angle. H$_3$Re$_3$(CO)$_{10}$(tedip)$_2$ [tedip stands for (EtO)$_2$POP(OEt)$_2$] was also characterized to have a four-membered ring (Prest, Mays, Raithby & Orpen, 1982), but the detailed structure is not reported.

The Re(CO)$_3$Cl(tdppme) reported here is the only chelating phosphine monorhenium compound with a six-membered ring. The Re—P bond lengths of 2·445 (3)–2·480 (2) Å are slightly longer than those in the normal range. The P(1)—Re—P(2) angle 88·07 (8)° is only 6–8° larger than those in Re(dppe) complexes. However, the P(2)—C(2)—C(3) and P(1)—C(1)—C(3) angles of 118·9 (6)–121·1 (6)° are substantially larger than the normal 109°. It is an indication that the large ring strain disfavors the geometry of the six-membered ring. Dppp [1,3-bis(diphenylphosphinopropane)] has been reported to form a six-membered ring complex, RhCl$_2$(COPh)(dppp) (McGuiggan, Doughty & Pignolet, 1980). The bite angle P—Re—P 94° is 12–14° larger than that in the dppe rhenium complexes with five-membered rings. All the other angles in the six-membered ring ranging from 114–117° are larger than the expected tetrahedral angle. Thus, the six-membered ring complexes are characterized with a large ring strain as reflected in the bond angles.

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# Structure of an Octacoordinate Cadmium(II) Complex: Di-$\mu$-aqua-bis[1,1,1-triaqua-$\mu$-(*o*-phenylenediamine-*N*,*N*,*N*′,*N*′-tetraacetato-$O^1$,$O^2$,$O^3$:*N*,*N*′,$O^1$,$O^2$,$O^4$,$O^5$)-dicadmium(II)] Octahydrate

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**Abstract.** [Cd(H$_2$O)C$_{14}$H$_{12}$N$_2$O$_8$Cd(H$_2$O)$_3$]$_2$.8H$_2$O, $M_r$ = 1410·4, triclinic, $P\bar{1}$, $a$ = 11·357 (2), $b$ = 12·260 (2), $c$ = 9·905 (1) Å, $\alpha$ = 114·74 (1), $\beta$ = 102·54 (1), $\gamma$ = 101·99 (2)°, $V$ = 1151·3 (4) Å$^3$, $Z$ = 1, $D_m$ = 2·03, $D_x$ = 2·03 g cm$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·5417 Å, $\mu$ = 149·7 cm$^{-1}$, $F(000)$ = 700, $T$ = 293 K. Final $R$ = 0·055 for 3365 observed reflections. The title compound is a tetranuclear complex bridged by carboxylate and water oxygen atoms. The chelated cadmium(II) ion is dihydrated and octacoordinate, whereas

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