with the numbering scheme, and Table 2 gives selected interatomic bond distances and angles. A unit-cell diagram is shown in Fig. 2.*

Related literature. Structural work on related organometallic complexes $[Zr(C_5H_5)_2Cl]_2O$ (Clarke & Drew, 1974) and $[Zr(C_5H_5)_2CH_3]_2O$ (Hunter, Hrncir, Bynum, Penttila & Atwood, 1983) has been reported.

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Orthorhombic Crystal Form of *trans*-Carbonylchlorobis(triphenylphosphine)rhodium(I) Dichloromethane Solvate

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Abstract. $[RhCl(C_{18}H_{15}P)_2(CO)].CH_2Cl_2$, $M_r = 775{\cdot}9$, orthorhombic, *Pbca*, $a = 23{\cdot}345\,(3)$, $b = 20{\cdot}644\,(7)$, $c = 7{\cdot}965\,(3)$ Å, $V = 3821{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, monochromated Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 7{\cdot}6$ cm^{-1}, $F(000) = 1576$, $T = 298$ K, $R = 0{\cdot}056$ and $wR = 0{\cdot}063$ for 587 observed reflections with $|F^2| > 3\sigma(F^2)$. The geometry at Rh is close to square planar and bond lengths and angles are similar to those in the previous determinations of the unsolvated structure.

Experimental. Large, very thin plates of *trans*-$[Rh(PPh_3)_2(CO)Cl]$ were isolated from an attempted purification of *trans*-$[Rh_2(PPh_3)_2(CO)_2\{\mu\text{-}S(CH_2)_4S\}]$ and a fragment *ca* $0{\cdot}3 \times 0{\cdot}1 \times 0{\cdot}008$ mm was used for data collection. Unit-cell parameters by least-squares fit of 25 reflections in the range $6{\cdot}7 < \theta < 11{\cdot}3°$, space group *Pbca* from systematic absences of $0kl$, k odd; $h0l$, l odd; $hk0$, h odd, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan, $\Delta\theta = (0{\cdot}8 + 0{\cdot}35\tan\theta)°$, maximum scan time 1 min, 2087 unique reflections measured for $2 < \theta < 20°$ and h $0\rightarrow7$, k $0\rightarrow19$, l $0\rightarrow22$, 587 observed reflections with $|F^2| > 3\sigma(F^2)$, $\sigma(F^2) = [\sigma^2(I) + (0{\cdot}04I)^2]^{1/2}/\mathrm{Lp}$. Two standard reflections remeasured every 30 min showed no significant change, Lorentz–polarization corrections, no absorption or extinction corrections. The structure was solved by routine heavy-atom methods and showed disorder between carbonyl and chloride ligands. The carbonyl C and O atoms were fixed at idealized positions with Rh—C = 1·82 and C—O = 1·14 Å (Rheingold & Geib, 1987). Rh atom anisotropic, otherwise non-H atoms were refined isotropically by

Table 1. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

	x	y	z	U_{iso}/U_{eq}*
Rh	0	0	0	45 (1)
Cl	−534 (5)	988 (5)	−443 (15)	67 (4)
P	−661 (3)	−628 (3)	−1463 (8)	44 (2)
C1	−1094 (7)	−1140 (7)	−53 (35)	34 (5)
C2	−953 (9)	−1237 (10)	1510 (29)	49 (7)
C3	−1263 (10)	−1631 (11)	2586 (35)	77 (8)
C4	−1770 (9)	−1896 (11)	1910 (30)	64 (8)
C5	−1942 (9)	−1801 (10)	258 (35)	71 (7)
C6	−1586 (9)	−1409 (11)	−759 (28)	57 (7)
C7	−297 (9)	−1172 (9)	2909 (27)	42 (6)
C8	126 (9)	−894 (9)	−3904 (28)	58 (7)
C9	423 (9)	−1276 (10)	−4995 (39)	67 (6)
C10	286 (9)	−1938 (10)	−5201 (37)	72 (7)
C11	−145 (9)	−2176 (10)	−4203 (27)	69 (8)
C12	−443 (10)	−1807 (11)	−3067 (29)	59 (7)
C13	−1195 (8)	−224 (8)	−2771 (25)	34 (6)
C14	−1601 (8)	164 (9)	−2056 (27)	55 (7)
C15	−2023 (10)	480 (11)	−2952 (20)	66 (8)
C16	−2018 (10)	388 (11)	−4662 (33)	80 (8)
C17	−1619 (9)	4 (16)	−5444 (29)	93 (8)
C18	−1192 (9)	−301 (10)	−4497 (27)	60 (7)
C19	3410 (18)	1777 (21)	2908 (58)	99 (16)
Cl2	3336 (5)	2571 (8)	2532 (24)	128 (4)
Cl3	2827 (6)	1413 (7)	3787 (19)	120 (5)
C20	387	−750	403	68 (15)
O	629	−1221	656	138 (17)

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Selected intramolecular distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Rh—Cl	2·414 (11)	Rh—P	2·323 (6)
P—C1	1·84 (2)	P—C7	1·82 (2)
P—C13	1·82 (2)		
Cl—Rh—P	93·3 (3)	Rh—P—C1	112·1 (8)
Rh—P—C7	110·8 (7)	Rh—P—C13	118·8 (6)
C1—P—C7	106·6 (9)	C1—P—C13	103·7 (9)
C7—P—C13	103·8 (9)		

full-matrix least squares. H atoms were held fixed at calculated positions with $U_{iso} = 1·3U_{eq}$ for the parent atom. The occupancy for the atoms of the CH_2Cl_2 solvate molecule refined to 0·65. The crystal data are calculated for the ideal crystal with full solvate occupancy. The refinement converged at $R = 0·056$, $wR = 0·063$ with 101 variables, $w = 1/\sigma^2(F)$, $S = 1·8$, $(\Delta/\sigma)_{max} = 0·1$, $(\delta\rho)_{max,min} = +0·70$, $-0·30$ e Å^{-3}. Atomic scattering factors were from *International Tables for X-ray crystallography* (1974, Vol. IV). Computer programs from the Enraf–Nonius *SDP-Plus* package (Frenz, 1985) were run on a Micro-VAX computer. Atomic parameters are given in Table 1 and bond distances and angles are presented in Table 2.* Atomic numbering is shown in Fig. 1 and packing in Fig. 2.

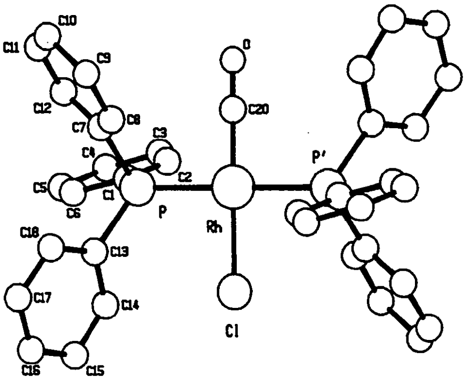

Fig. 1. Molecular structure and numbering scheme for *trans*-[Rh(PPh_3)$_2$(CO)Cl].

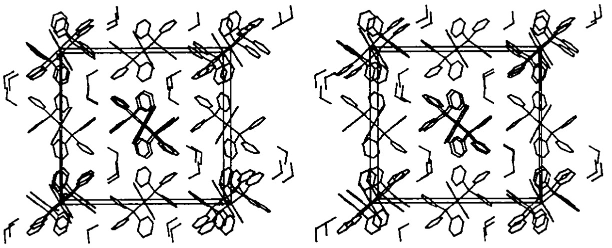

Fig. 2. Packing diagram for *trans*-[Rh(PPh_3)$_2$(CO)Cl].CH_2Cl_2.

Related literature. The structure of the unsolvated complex has been determined three times before. The first determination was of a triclinic form (Del Pra, Zanotti & Segala, 1979), reported as $P1$, with some rather unusual bond lengths. Refinement in $P\bar{1}$ (Ceriotti, Ciani & Sironi, 1983) with the Rh atom on an inversion centre and CO/Cl disorder gave more reasonable bond lengths. The other two determinations (Ceriotti, Ciani & Sironi, 1983; Rheingold & Geib, 1987) are of the same modification, a monoclinic form $P2_1/n$, with the a and c axes interchanged between the two determinations. The bond lengths and angles here are quite similar to those established in the earlier determinations. Here, and in the triclinic form, the phosphines are staggered, whereas in the monoclinic form they are eclipsed.

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