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Structure of *trans*-[Rh(CO)Cl{P(C₆H₅)₃}₂]: a Centrosymmetric Triclinic Phase

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Abstract. *trans*-[Rh(CO)Cl{P(C₆H₅)₃}₂], $M_r = 690·96$, triclinic, $P\bar{1}$, $a = 9·182$ (3), $b = 9·640$ (2), $c = 10·384$ (2) Å, $\alpha = 107·56$ (2), $\beta = 89·70$ (2), $\gamma = 110·48$ (2)°, $V = 815·7$ (9) Å³, $Z = 1$, $D_x = 1·41$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 7·2$ cm⁻¹, $F(000) = 352$, $T = 298$ K, $R = 0·036$ for 2878 reflections with $I > 3\sigma(I)$. The molecule lies at an inversion center with a disordered Cl atom and CO group. Rh—C(10) = 1·759 (1), Rh—Cl = 2·380 (2), Rh—P = 2·328 (1) Å. The molecular structure is similar to the previously reported noncentrosymmetric triclinic ($P1$) structure. However, unlike the previous report, the distance between C(10) and O(10), C—O = 1·14 (1) Å, is well defined and reasonable.

Experimental. Yellow crystals of *trans*-carbonyl-chlorobis(triphenylphosphino)rhodium(I), *trans*-Rh(CO)Cl(PPh₃)₂, were grown from CH₂Cl₂/Et₂O. A suitable column-like crystal with dimensions 0·15 × 0·17 × 0·32 mm was mounted on a glass fiber on an Enraf–Nonius CAD-4 diffractometer equipped with monochromated Mo $K\alpha$ radiation. Cell constants were derived from least-squares refinement of 25 reflections having $19 < 2\theta < 24°$. Intensity data were collected at room temperature using the $\theta/2\theta$ scan technique with $2 < 2\theta < 55°$ ($h = -11$ to 11; $k = 0$ to 12; $l = -13$ to 12). Three standard reflections were monitored every 3600 s, and only small ($<2\%$) random variations were observed. A total of 3728 unique reflections were measured, of which 2878

were observed [$I > 3\sigma(I)$]. Lorentz and polarization corrections were applied. An empirical correction based on a series of ψ scans was applied to the data; $T_{\min} = 0·87$, $T_{\max} = 0·99$.

The atomic position of Rh was fixed at (0,0,0). The remaining non-H atoms were located in successive difference Fourier syntheses. H-atom positions were calculated using ideal geometry but were not included in the least-squares refinements. Atomic scattering factors including anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The structure was refined by full-matrix least squares using *Personal SDP* (Frenz, 1989) on an 80386-based IBM compatible PC. All non-H atoms were refined anisotropically. $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 4F_o^2/[\sigma^2(I) + 0·02(F_o)^2]$. A secondary-extinction coefficient refined to a value of $\chi = 5·21 \times 10^{-7}$, at which point the correction factor $(1 + \chi I)^{-1}$ was applied to F_c. Based on the centrosymmetric space group $P\bar{1}$, final $R = 0·036$, $wR = 0·045$, $R_{\text{all}} = 0·059$ and $S = 1·617$ obtained using 206 variables. The largest shift/e.s.d. was 0·02; maximum and minimum residual electron densities in the final difference Fourier map were 0·52 and $-0·40$ e Å⁻³.

The atoms Cl—Rh—C(10)—O(10) are almost collinear [angle of Cl—Rh—C(10) is 179·7 (3)°]. The disordered model in $P\bar{1}$ leads to three atom sites for these ligands on each side of the Rh. The sites are sufficiently well resolved that atomic positional and anisotropic displacement parameters can be refined satisfactorily. Refinements based on the noncentrosymmetric space group $P1$ were also carried out. CO and Cl remained disordered, and the

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Table 1. *Positional parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

	x	y	z	B (Å^2)
Rh	0·000	0·000	0·000	2·549 (7)
Cl	−0·2120 (2)	−0·1590 (2)	0·0864 (2)	4·69 (5)
P	0·01922 (9)	0·20796 (9)	0·19370 (8)	2·77 (2)
O(10)	0·2554 (8)	0·1962 (7)	−0·1060 (6)	7·1 (2)
C(10)	0·1573 (9)	0·1176 (8)	−0·0631 (7)	3·9 (2)
C(11)	0·1273 (4)	0·4061 (4)	0·1913 (4)	3·65 (8)
C(12)	0·2873 (5)	0·4510 (5)	0·1841 (4)	5·0 (1)
C(13)	0·3724 (7)	0·5991 (5)	0·1784 (5)	7·0 (1)
C(14)	0·2973 (8)	0·6993 (6)	0·1782 (6)	8·9 (2)
C(15)	0·1430 (8)	0·6549 (5)	0·1817 (6)	9·1 (2)
C(16)	0·0532 (6)	0·5082 (4)	0·1888 (5)	6·2 (1)
C(21)	−0·1719 (4)	0·2105 (4)	0·2392 (4)	3·48 (8)
C(22)	−0·2081 (4)	0·2410 (4)	0·3715 (4)	4·15 (9)
C(23)	−0·3578 (5)	0·2411 (5)	0·3981 (5)	5·8 (1)
C(24)	−0·4646 (5)	0·2156 (6)	0·2956 (5)	6·8 (1)
C(25)	−0·4294 (5)	0·1863 (6)	0·1653 (5)	6·7 (1)
C(26)	−0·2822 (5)	0·1824 (5)	0·1356 (5)	5·5 (1)
C(31)	0·1154 (4)	0·1998 (4)	0·3435 (3)	3·10 (7)
C(32)	0·1221 (4)	0·0602 (4)	0·3428 (4)	4·14 (9)
C(33)	0·1934 (5)	0·0494 (5)	0·4553 (4)	5·7 (1)
C(34)	0·2564 (5)	0·1757 (6)	0·5660 (4)	5·9 (1)
C(35)	0·2503 (6)	0·3155 (6)	0·5683 (5)	6·6 (2)
C(36)	0·1800 (5)	0·3294 (5)	0·4567 (4)	5·1 (1)

refinements were not satisfactorily convergent because of large correlations between variables related by inversion symmetry. In a previous report of this structure by Del Pra, Zanotti & Segala (1979), an unacceptable C(10)—O(10) distance of 0·74 (1) Å was obtained, assuming the noncentrosymmetric space group *P*1, whereas in the centrosymmetric space group *P*$\bar{1}$ we obtain a value of 1·14 (1) Å which is chemically reasonable. Therefore we report the structure as being disordered with space group *P*$\bar{1}$.

Final positional parameters are presented in Table 1.* Bond lengths and angles are listed in Table 2. Fig. 1 shows an *ORTEP* drawing (Johnson, 1970; Frenz, 1989) of the molecule and atomic labeling scheme.

Related literature. Del Pra, Zanotti & Segala (1979) have reported a noncentrosymmetric triclinic structure with abnormal C—O distance. Ceriotti, Ciani & Sironi (1983) and Rheingold & Geib (1987) have reported a monoclinic structure of the title compound. The iodo analog has also been reported (Basson, Leipoldt & Roodt, 1990).

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Table 2. *Bond distances (Å) and angles (°)*

Numbers in parentheses are e.s.d.'s in the least significant digits.

Rh—Cl	2·380 (2)	C(15)—C(16)	1·390 (7)
Rh—P	2·3279 (7)	C(21)—C(22)	1·380 (5)
Rh—C(10)	1·759 (7)	C(21)—C(26)	1·378 (6)
Cl—O(10)	0·522 (7)	C(22)—C(23)	1·401 (6)
Cl—C(10)	0·622 (7)	C(23)—C(24)	1·356 (7)
P—C(11)	1·826 (4)	C(24)—C(25)	1·358 (8)
P—C(21)	1·821 (4)	C(25)—C(26)	1·396 (7)
P—C(31)	1·828 (4)	C(31)—C(32)	1·366 (6)
O(10)—C(10)	1·14 (1)	C(31)—C(36)	1·377 (4)
C(11)—C(12)	1·387 (6)	C(32)—C(33)	1·390 (7)
C(11)—C(16)	1·385 (7)	C(33)—C(34)	1·344 (5)
C(12)—C(13)	1·387 (6)	C(34)—C(35)	1·362 (9)
C(13)—C(14)	1·37 (1)	C(35)—C(36)	1·392 (8)
C(14)—C(15)	1·33 (1)		

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Cl—Rh—P	87·76 (5)	C(12)—C(11)—C(16)	119·6 (4)
Cl—Rh—C(10)	179·7 (3)	C(11)—C(12)—C(13)	119·8 (5)
P—Rh—C(10)	92·1 (2)	C(12)—C(13)—C(14)	119·6 (5)
Rh—P—C(11)	118·2 (1)	C(13)—C(14)—C(15)	120·6 (5)
Rh—P—C(21)	112·17 (9)	C(14)—C(15)—C(16)	121·8 (6)
Rh—P—C(31)	112·5 (1)	C(11)—C(16)—C(15)	118·5 (5)
C(11)—P—C(21)	104·1 (2)	C(22)—C(21)—C(26)	119·8 (4)
C(11)—P—C(31)	103·2 (2)	C(21)—C(22)—C(23)	119·2 (4)
C(21)—P—C(31)	105·4 (2)	C(22)—C(23)—C(24)	120·5 (4)
Rh—C(10)—O(10)	177·2 (9)	C(23)—C(24)—C(25)	120·6 (5)
P—C(11)—C(12)	118·3 (3)	C(24)—C(25)—C(26)	120·1 (5)
P—C(11)—C(16)	122·0 (3)	C(21)—C(26)—C(25)	119·8 (4)
P—C(21)—C(22)	122·7 (3)	C(32)—C(31)—C(36)	119·2 (4)
P—C(21)—C(26)	117·5 (3)	C(31)—C(32)—C(33)	120·3 (3)
P—C(31)—C(32)	118·7 (2)	C(32)—C(33)—C(34)	120·6 (5)
P—C(31)—C(36)	122·1 (3)	C(33)—C(34)—C(35)	119·9 (5)
		C(34)—C(35)—C(36)	120·6 (4)
		C(31)—C(36)—C(35)	119·5 (5)
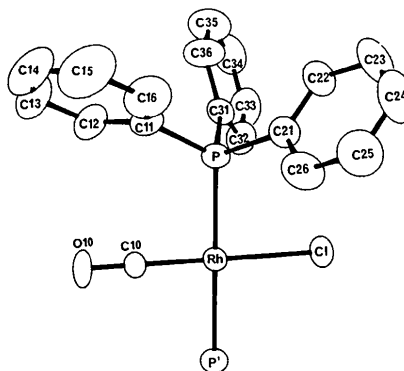
Fig. 1. *ORTEP* drawing of the title compound with 50% probability ellipsoids.

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