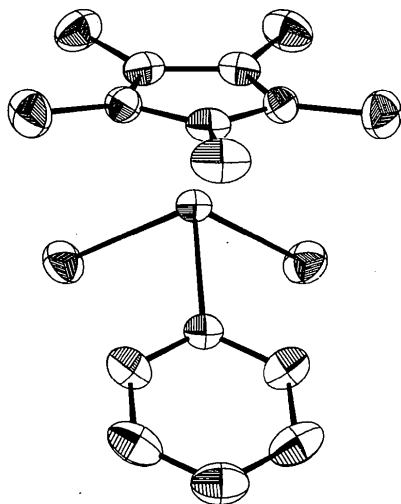**Table 1.** *Atomic coordinates of the non-H atoms with e.s.d.'s in parentheses*

B_{iso} is the mean of the principal axes of the thermal ellipsoid.

	x	y	z	B_{iso} (Å²)
Ru	0.87654 (6)	¾*	0.33182 (5)	2.173 (22)
Cl	1.07911 (17)	0.88628 (9)	0.30806 (14)	3.50 (5)
N	1.0151 (7)	¾*	0.5930 (6)	2.50 (20)
C(101)	1.0432 (7)	0.6645 (4)	0.6782 (6)	3.27 (19)
C(102)	1.1008 (7)	0.6630 (4)	0.8484 (6)	3.96 (23)
C(103)	1.1300 (11)	¾*	0.9374 (8)	3.9 (3)
C(1)	0.5864 (8)	¾*	0.3756 (7)	2.65 (25)
C(11)	0.5424 (10)	¾*	0.5390 (8)	3.9 (3)
C(2)	0.6078 (6)	0.6637 (3)	0.2848 (5)	2.48 (16)
C(21)	0.5929 (7)	0.5582 (4)	0.3322 (7)	3.93 (22)
C(3)	0.6377 (6)	0.6969 (3)	0.1318 (5)	2.65 (17)
C(31)	0.6485 (7)	0.6322 (4)	−0.0094 (6)	3.86 (22)

* y coordinate fixed by symmetry.

Fig. 1. Structure of $[\eta\text{-}C_5(CH_3)_5]RuCl_2(NC_5H_5)$.

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

Ru—Cl	2.386 (1)	Cl—Ru—Cl(a)	100.21 (5)
Ru—N	2.150 (5)	Cl—Ru—N	88.28 (9)
Ru—C(1)	2.227 (6)	Cl—Ru—Cp*	123.4 (2)
Ru—C(2)	2.195 (4)	N—Ru—Cp*	123.6 (2)
Ru—C(3)	2.163 (4)		
Ru—Cp*	1.819 (4)		

Cp* is the centroid of the C_5 ring of the $C_5(CH_3)_5$ group [obtained by averaging the coordinates of C(1), C(2), C(2)a, C(3) and C(3)a].

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Structure of *trans*-Di-μ-chloro-dichlorobis(triphenylphosphite)dipalladium

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Abstract. $[Pd_2Cl_4(C_{18}H_{15}O_3P)_2]$, $M_r = 975.19$, monoclinic, $P2_1/c$, $a = 10.034$ (2), $b = 16.278$ (2), $c = 13.129$ (2) Å, $\beta = 112.04$ (1)°, $V = 1987.5$ (5) Å³, $Z = 2$, $D_x = 1.63$ g cm⁻³, λ(Mo $K\alpha$) = 0.7107 Å, μ(Mo $K\alpha$) = 12.77 cm⁻¹, $F(000) = 968$, $T = 294$ K, $R = 0.0504$ for 1811 unique observed reflections, $I > 2\sigma(I)$. The structure consists of centrosymmetric dimers, with the Pd^{II} ions joined by unsymmetrical double Cl⁻ bridges. A terminal Cl⁻ and a P(OPh)₃ ligand complete the near square-planar coordination of each Pd. Bond lengths: Pd—P 2.187 (3), Pd—Cl(terminal) 2.269 (3), Pd—Cl(bridging, *trans* to P) 2.413 (2), Pd—Cl(bridging, *trans* to Cl) 2.309 (2) Å.

Experimental. Compound isolated from the reaction of orthomanganated triphenylphosphite, (PhO)₂-($\overline{C_6H_4O}$)PMn(CO)₄ with PdCl₂; a more direct synthesis is available (Chatt & Venanzi, 1957). Plate-like

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Table 1. *Final positional and equivalent isotropic thermal parameters* ($\AA^2$) *for* $[Pd_2Cl_4\{P(OPh)_3\}_2]$

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

	x	y	z	U_{eq}
Pd(1)	0.64793 (8)	0.03379 (4)	1.11214 (5)	0.065
P(1)	0.7953 (3)	-0.0114 (2)	1.2713 (2)	0.073
Cl(1)	0.4780 (2)	0.0876 (1)	0.9417 (2)	0.075
Cl(2)	0.7696 (3)	0.1543 (2)	1.1568 (2)	0.113
O(1)	0.7655 (7)	-0.1046 (4)	1.2774 (5)	0.088
O(2)	0.7866 (7)	0.0327 (4)	1.3757 (5)	0.087
O(3)	0.9633 (7)	-0.0009 (4)	1.3063 (5)	0.078
C(11)	0.712 (1)	-0.2288 (7)	1.3417 (9)	0.114
C(12)	0.728 (2)	-0.2874 (9)	1.419 (1)	0.144
C(13)	0.832 (2)	-0.2760 (9)	1.523 (1)	0.134
C(14)	0.912 (1)	-0.2051 (9)	1.5494 (9)	0.106
C(15)	0.894 (1)	-0.1450 (6)	1.4693 (8)	0.085
C(16)	0.795 (1)	-0.1612 (6)	1.3675 (8)	0.077
C(21)	0.672 (2)	0.1430 (8)	1.4245 (9)	0.104
C(22)	0.558 (2)	0.1763 (9)	1.443 (1)	0.127
C(23)	0.438 (2)	0.131 (1)	1.415 (1)	0.131
C(24)	0.428 (2)	0.056 (1)	1.372 (1)	0.139
C(25)	0.541 (2)	0.0226 (8)	1.3562 (9)	0.110
C(26)	0.663 (1)	0.0662 (7)	1.3844 (7)	0.071
C(31)	1.105 (1)	-0.1099 (8)	1.280 (1)	0.103
C(32)	1.192 (2)	-0.1420 (8)	1.229 (1)	0.123
C(33)	1.213 (2)	-0.100 (1)	1.150 (1)	0.121
C(34)	1.154 (2)	-0.023 (1)	1.121 (1)	0.132
C(35)	1.066 (2)	0.0104 (8)	1.171 (1)	0.119
C(36)	1.041 (1)	-0.0356 (8)	1.2480 (8)	0.077

Table 2. *Selected bond lengths* ($\AA$) *and angles* (°) *for* $[Pd_2Cl_4\{P(OPh)_3\}_2]$

Pd(1)—P(1)	2.187 (3)	Pd(1)—Cl(1)	2.413 (2)
Pd(1)—Cl(1)'	2.309 (2)	Pd(1)—Cl(2)	2.269 (3)
P(1)—O(1)	1.553 (7)	P(1)—O(2)	1.578 (6)
P(1)—O(3)	1.582 (7)	O(1)—C(16)	1.44 (1)
O(2)—C(26)	1.39 (1)	O(3)—C(36)	1.40 (1)

P(1)—Pd(1)—Cl(1)	176.8 (1)	O(1)—C(16)—C(15)	122.1 (9)
P(1)—Pd(1)—Cl(2)	86.5 (1)	Cl(1)—Pd(1)—Cl(2)	92.9 (1)
Cl(1)—Pd(1)—Cl(1)'	84.9 (1)	O(1)—C(16)—C(11)	114 (1)
Pd(1)—Cl(1)—Pd(1)'	95.1 (1)	Pd(1)—P(1)—O(1)	107.6 (2)
Pd(1)—P(1)—O(2)	116.0 (3)	Pd(1)—P(1)—O(3)	120.4 (3)
O(1)—P(1)—O(2)	109.0 (4)	O(1)—P(1)—O(3)	107.1 (4)
O(2)—C(26)—C(21)	117 (1)	O(2)—P(1)—O(3)	95.7 (4)
O(2)—C(26)—C(25)	121 (1)	P(1)—O(1)—C(16)	133.1 (6)
P(1)—O(2)—C(26)	126.1 (6)	P(1)—O(3)—C(36)	123.4 (5)

pale yellow crystals from CH_2Cl_2, $0.50 \times 0.22 \times 0.18$ mm, Enraf–Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$ X-radiation ($\lambda = 0.7107$ Å), cell parameters from 25 reflections ($2\theta = 20$–$25°$). For data collection, ω–2θ scans, $2\theta_{max} = 50°$, h $0 \rightarrow 13$, k $0 \rightarrow 21$, l $-17 \rightarrow 17$, no significant crystal movement or decay, empirical absorption correction applied using φ scans (transmission factors 0.999 maximum, 0.873 minimum), 2851 unique reflections giving 1811 with $I > 2\sigma(I)$ for structure solution (the Pd position from Patterson methods, other non-H atoms from a subsequent difference map) and refinement (using full-matrix least squares based on F^2, 227 parameters refined). All non-H atoms were assigned anisotropic thermal parameters and refined without constraint while H atoms were included in calculated positions (d_{C-H} 0.96 Å) with a common
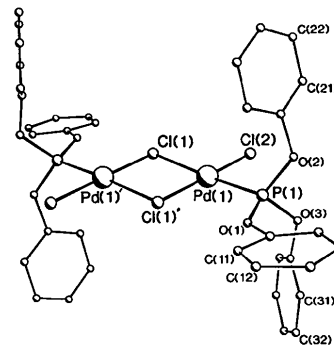
Fig. 1. A *PLUTO* (Motherwell & Clegg, 1978) diagram of the structure of $[Pd_2Cl_4\{P(OPh)_3\}_2]$ showing atom numbering.

isotropic temperature factor. At convergence R, wR = 0.0504, 0.0460. The weighting scheme $w = [\sigma(F)^2 + 0.000334F^2]^{-1}$ gave satisfactory agreement analyses. Final maximum $\Delta/\sigma = 0.03$, residual peaks $+0.75/-0.38$ e $\AA^{-3}$ adjacent to Pd. Scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol IV) and calculations used the *SHELX* programs (Sheldrick, 1976, 1986). Atom coordinates and equivalent isotropic thermal parameters are given in Table 1,* selected molecular-geometry parameters appear in Table 2 and the structure is illustrated in Fig. 1.

Related literature. Structures have been reported for related Pt complexes $Pt_2Cl_4(PR_3)_2$ where PR_3 = triethylphosphine (Blake, Gould, Marr, Rankin & Schroder, 1989), PR_3 = tri-n-propylphosphine (Black, Mais & Owston, 1969), PR_3 = di-*tert*-butylcyclopropylphosphine (Simms, Shang, Lu, Youngs & Ibers, 1987) and for a mixed Pt–Pd analogue $PdPtCl_4(PEt_3)_2$ (Clark, Ferguson, Jain & Parvez, 1985). For each of these the same basic geometry prevails.

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Diaqua-*N*,*N*′-trimethylenebis(5′-chlorosalicylideneaminato)manganese(III) Perchlorate Monohydrate: a Schiff-Base Complex Linked into Infinite Spirals by Hydrogen and π Bonds

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Abstract. $[Mn(C_{17}H_{18}Cl_2N_2O_4)]ClO_4.H_2O$, $M_r = 557.65$, orthorhombic, $P2_12_12_1$, $a = 13.650\,(1)$, $b = 20.784\,(9)$, $c = 7.773\,(2)$ Å, $V = 2205\,(2)$ Å^3, $Z = 4$, $D_x = 1.679$ Mg m^{-3}, $F(000) = 1136$, $\lambda(Mo\,K\alpha) = 0.71069$ Å, $\mu = 0.992$ mm^{-1}, $T = 293$ K, $R = 0.057$, 1565 unique reflexions $[I \geq 2\sigma(I)]$. The role of manganese in photosynthesis has focused attention on how individual complex molecules are able to associate to form multinuclear structures. In the title molecule the Schiff base forms a square-planar belt around manganese with Mn—N distances of 2.029 (8), 2.044 (8) Å and Mn—O distances of 1.894 (6), 1.866 (6) Å. Two capping water molecules at 2.230 (6) and 2.206 (7) Å complete a distorted octahedral arrangement. Infinite spirals of the complex, generated by a screw axis along **c**, are linked by hydrogen bonds from coordinated water to phenoxy oxygen and π bonds between overlapping aromatic rings. The hydrogen-bonding scheme is completed by lattice water, which, in addition to bridging alternate members of the spiral *via* coordinated water, forms two links to perchlorate anions.

Experimental. The compound was prepared following the method of Boucher (Boucher & Coe, 1975). To a solution of the ligand (2.85 mmol) in ethanol $Mn(ClO_4)_2.6H_2O$ (2.85 mmol) in methanol was added. The mixture was stirred in air for five days before filtering. On standing the filtrate at room temperature, small dark-green crystals were deposited. These were separated by filtration and washed with ethanol and diethyl ether before being dried in air.

Crystal dimensions $0.4 \times 0.3 \times 0.1$ mm, Rigaku AFC-6S diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centered reflexions ($21.0 \leq 2\theta \leq 30.8°$), ω–2θ scan mode, ω scan width $(0.94 + 0.30\tan\theta)°$ and scan speed of 4° min^{-1} with up to 2 additional scans of weak reflexions $[I < 10\sigma(I)]$, $0 \leq h \leq 16$, $0 \leq k \leq 24$, $0 \leq l \leq 9$, $0 \leq \theta \leq 25°$, 2264 reflexions measured, 2252 unique 1565 observed $[I \geq 2\sigma(I)]$, intensity standards $(25\bar{1}, 15\bar{1}, \bar{1}80)$ measured every 150 reflexions showed 24% decline, Lp, decomposition and absorption corrections applied (ψ-scan method, min. and max. transmission 0.91 and 1.00 respectively), *MITHRIL* (Gilmore, 1984) used to solve the phase problem, all non-H atoms found in Fourier map, H atoms placed in chemically reasonable positions except for those attached to water, which were located from a ΔF map and refined, full-matrix least-squares refinement based on F using *TEXSAN* crystallographic software (Molecular Structure Corporation, 1985), final $R = 0.057$ $\{wR = 0.058$, $w = 1/[\sigma^2(F_o) + (0.03F_o)^2]\}$; a parallel refinement of the inverted structure gave a final $R = 0.058$ and $wR = 0.059$, indicating that the correct enantiomer had been chosen, anisotropic thermal parameters for heavier atoms, isotropic for hydrogen. Maximum fluctuation in final ΔF map in range -0.47 to 0.46 e Å^{-3}, maximum Δ/σ 0.08. Scattering factors from Cromer & Waber (1974), computation