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The Structures of *trans*-Dichloro(*N*-methylsalicylideneaminato)oxo(triphenylphosphine)-rhenium(V) and *cis*-Dichloro(*N*-methylsalicylideneaminato)oxo(triphenylphosphine)-rhenium(V), $[Re(C_8H_8NO)Cl_2O\{P(C_6H_5)_3\}]$

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Abstract. *trans*: $M_r = 669\cdot5$, orthorhombic, $Pca2_1$, $a = 14\cdot802$ (2), $b = 12\cdot375$ (2), $c = 13\cdot798$ (2) Å, $U = 2527\cdot5$ (7) Å^3, $Z = 4$, $D_x = 1\cdot76$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 5\cdot17$ mm^{-1}, $F(000) = 1304$, $T = 298$ K, $R = 0\cdot025$ for 2125 reflections with $I > 3\sigma(I)$. *cis*: $M_r = 669\cdot5$, monoclinic, $P2_1/n$, $a = 9\cdot929$ (2), $b = 15\cdot860$ (3), $c = 15\cdot666$ (3) Å, $\beta = 93\cdot33$ (2)°, $U = 2463$ (1) Å^3, $Z = 4$, $D_x = 1\cdot80$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 5\cdot31$ mm^{-1}, $F(000) = 1304$, $T = 298$ K, $R = 0\cdot045$ for 2794 reflections with $I > 3\sigma(I)$. In both compounds the coordination is nearly octahedral, differing only by the *cis* and *trans* arrangement of the two Cl atoms. The geometry of the coordination polyhedron is discussed by comparing the present structures with all oxorhenium(V) quasi-octahedral complexes with oxygenated bidentate ligands of known molecular structure.

Introduction. Re^{V} is known to form easily quasi-octahedral oxorhenium complexes where a variety of spectroscopic, structural and magnetic data agree in indicating the formation of a strong triple Re≡O bond. In the last few years we have reported the crystal-structure determinations of some of these complexes with Schiff bases such as Me-sal or Ph-sal (*N*-methyl- or *N*-phenylsalicylideneaminato) and salen [*N*,*N*′-ethylenebis(salicylideneaminato)].* They include $Re_2O_2Cl_4(PPh_3)$(salen) (Bombieri, Mazzi, Gilli & Hernández-Cano, 1978), $ReOCl_2$(Me-sal)$_2$ (Gilli, Sacerdoti, Bertolasi & Rossi, 1982), *trans*-$ReOBr_2(PPh_3)$(Ph-sal) monoclinic form (Bertolasi, Sacerdoti, Gilli & Mazzi, 1982) and triclinic form (Sacerdoti, Bertolasi, Gilli & Duatti, 1984). The present paper reports the crystal structures of *trans*- and *cis*-$ReOCl_2(PPh_3)$(Me-sal) (hereafter TRANS and CIS, respectively) whose preparations have already been reported (Mazzi, Roncari, Rossi, Bertolasi, Traverso & Magon, 1980; Roncari, Mazzi, Rossi, Duatti & Magon, 1981). An attempt will be made to review the stereochemical features of this class of compounds, particularly as regards the Re–X (X = O$_{oxo}$, O, N, P, Cl, Br) distances in Re^{V} octahedral complexes.

Experimental. TRANS: olive green prism, 0·20 × 0·31 × 0·07 mm, automatic Enraf–Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$, cell parameters from 25 reflections in the range $11 < \theta < 13°$, $\omega/2\theta$ scan, $2 \leq \theta \leq 27°$, 2864 independent reflections ($0 \leq h \leq 16$, $0 \leq k \leq 15$, $0 \leq l \leq 16$), 2125 having $I > 3\sigma(I)$, 1 stable standard reflection monitored every 2 h, empirical absorption correction (0·49 ≤ transmission factor ≤1); Patterson and Fourier methods, full-matrix least squares on F, anisotropic non-H's, calculated H's, $1/w^2 = \sigma_I^2 + 0\cdot070|F|^2$, $R = 0\cdot025$, $R_w = 0\cdot026$, max. $\Delta/\sigma = 0\cdot06$, $S = 1\cdot41$, final $\Delta\rho$ excursions –0·4–0·4 e Å^{-3} outside the first Re coordination sphere. The molecule is chiral in a non-centrosymmetric space group and the solution given corresponds to the enantiomer with the smaller R factor.

CIS: emerald green prism, 0·25 × 0·16 × 0·07 mm, automatic Enraf–Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$, cell parameters from 25 reflections in the range $11 < \theta < 13°$, $\omega/2\theta$ scan, $2 \leq \theta \leq 27°$, 5355 independent reflections ($0 \leq h \leq 12$, $0 \leq k \leq 20$, $-19 \leq l \leq 19$), 2794 having $I > 3\sigma(I)$, 1 stable standard reflection monitored every 2 h, empirical absorption correction (0·79 ≤ transmission factor ≤ 1); Patterson and Fourier methods, full-matrix least squares on F, anisotropic non-H's, calculated H's, $1/w^2 = \sigma_I^2 + 0\cdot020|F|^2$, $R = 0\cdot045$, $R_w = 0\cdot054$, max. $\Delta/\sigma = 0\cdot01$, $S = 1\cdot16$, final $\Delta\rho$ excursions –0·4–0·4 e Å^{-3} outside the first Re coordination sphere.

Scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations done with the CAD-4-*SDP* system of programs (Frenz, 1978).

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* The ligand salicylideneaminato is also known as salicylideneiminato.

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Discussion. Final atomic positional and thermal (Hamilton, 1959) parameters for both compounds are given in Table 1.* The *ORTEP* (Johnson, 1971) views of the molecules are reported in Figs. 1 and 2. Bond distances and angles are given in Table 2.

In both compounds the coordination is nearly octahedral. They differ in the *trans* and *cis* arrangements of the two Cl atoms in the equatorial plane but are identical as far as the *trans* $O{-}Re{\equiv}O_{oxo}$ positions of the two oxygens on the axial line are concerned.

Neither Me-sal ligand is planar. Atoms C(1)–C(7) define an approximate plane ($\chi^2 = 25{\cdot}0$ for TRANS and 85·5 for CIS), from which the O(2), N, C(8) and Re atoms are displaced by −0·068 (4), 0·166 (7), 0·047 (11) and 0·600 (1) Å in TRANS and 0·056 (8), −0·005 (10), −0·292 (14) and 0·396 (1) Å in CIS. In TRANS the conformation of the PPh_3 moiety is described by the torsion angles O(2)—Re—P—C(21) = 1·0 (3), Re—P—C(15)—C(16) = −6·7 (8), Re—P—C(21)—C(26) = 63·5 (6) and Re—P—C(9)—C(10) = 43·0 (7)°. A situation where one P—C bond nearly eclipses the Re—O(2) bond has been found also in both polymorphic forms of *trans*-$Re^VOBr_2(PPh_3)$(Ph-sal) (Bertolasi *et al.*, 1982; Sacerdoti *et al.*, 1984) though with two different arrangements of the phenyl groups which produce the two conformational isomers. In TRANS the phenyl conformation is different again, suggesting that there must be several conformational-energy minima connected with the PPh_3-octahedron junction which can give rise to a variety of conformational polymorphs. In CIS the PPh_3 conformation is completely different. One P—C bond nearly eclipses the Re—N bond [N—Re—P—C(15) = −5·4 (4)°] while the Re—P—C(15)—C(16), Re—P—C(9)—C(14) and Re—P—C(21)—C(26) angles are respectively 102·8 (7), 13·0 (9) and −44·5 (9)°. The geometry of the coordination polyhedron is better discussed from a more general point of view. Table 3 reports some geometrical parameters of oxo complexes of Re^V with bidentate oxygenated ligands (mostly salicylideneaminato derivatives).

It is seen that in all compounds the oxygen atom of the chelate ligand is *trans* to O_{oxo}. This *trans* $O{-}Re{\equiv}O_{oxo}$ arrangement is probably due to the *trans* weakening caused by the multiple bond being smaller for the $Re{-}O^-$ bond (Gilli *et al.*, 1982). The mean $Re{-}O_{oxo}$ distance is 1·683 [3] Å and is indicative of a strong triple bond. The observed value for CIS [1·660 (8) Å] is somewhat shorter and must be taken with caution because of the high thermal parameters found in this structure. One effect of the triple bond is to repel the *cis* atoms and, in fact, the average over all structures of the mean $X_{cis}{-}Re{-}O_{oxo}$ angle is 94·5 [3]°. Values for TRANS and CIS are not far from this average [94·3 (1) and 94·1 (2)°, respectively]. Re—O distances are in the range 1·92–2·01 Å and values for TRANS and CIS are very close to the average of 1·970 [8] Å. One value (2·10 Å) is much greater and has been excluded from the averaging. The reason for the larger value is as follows. The *trans* weakening of the $Re{-}O^{x-}$ bond by the multiple $Re{\equiv}O_{oxo}$ bond is inversely related to the negative charge *x* on the oxygen (Shustorovich, Porai-Koshitz & Buslaev, 1975). Then it is irrelevant for *R*-sal or salen where $x = 1$ but becomes significant for acac where *x* averages to $\frac{1}{2}$. This point of view is supported by the very long Re—O distance [2·29 (2) Å] observed in the $O_{oxo}{-}Re{-}OH_2$ moiety of $Re^VO(H_2O)$-Cl_3(thiourea) (Lis, 1977), where $x = 0$. Re—N and

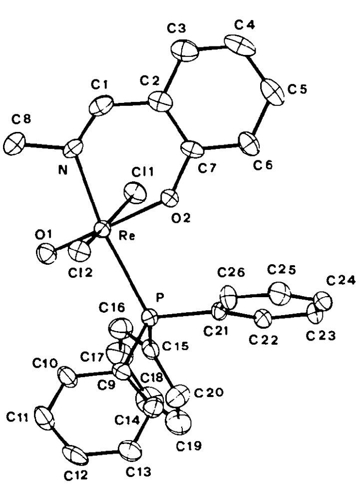

Fig. 1. Molecule of TRANS illustrating the thermal ellipsoids at 40% probability (Johnson, 1971).

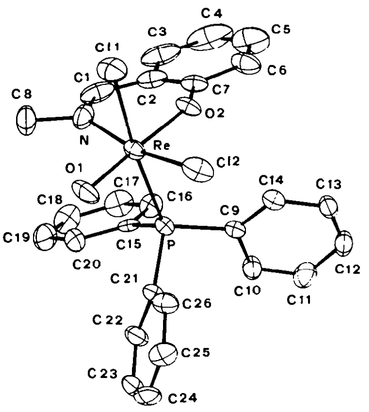

Fig. 2. Molecule of CIS illustrating the thermal ellipsoids at 40% probability (Johnson, 1971).

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* Lists of structure factors and anisotropic thermal parameters for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39074 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Re—P are found to lie in the intervals 2·10–2·17 and 2·43–2·49 Å, respectively. The total variation range for Re—Cl is 2·34–2·43 Å with an average of 2·389 [7] Å. Comparison of the two Re—Cl distances in CIS shows that the bond *trans* to P is 0·042 Å larger than that *trans* to N. This *trans* influence seems to be confirmed by the parallel differences observed in the other two *cis*-dichloro compounds of Table 3, that is $Re_2O_2Cl_4(PPh_3)_2$(salen) (Bombieri *et al.*, 1978) (differences of 0·039 and 0·049 Å) and $ReOCl_2(PPh_3)\{Ph(O)CNNCMe_3\}$ (Hursthouse, Jayaweera & Quick, 1979) (difference of 0·044 Å).

Table 1. *Positional parameters* (×10⁴) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

	CIS				TRANS			
	x	*y*	*z*	U_{eq}	*x*	*y*	*z*	U_{eq}
Re	5643·2 (4)	7183·1 (3)	6467·9 (3)	42·4 (2)	5651·3 (2)	7930·8 (2)	5000	32·1 (1)
Cl(1)	4968 (3)	6603 (2)	7789 (2)	76 (2)	7078 (1)	7683 (2)	4158 (2)	47 (1)
Cl(2)	3596 (3)	6660 (2)	5843 (2)	72 (2)	4349 (1)	7919 (2)	6005 (2)	47 (1)
P	6502 (3)	7630 (2)	5079 (2)	37 (1)	4713 (1)	7393 (2)	3599 (1)	45 (3)
O(1)	5232 (9)	8189 (5)	6590 (5)	74 (5)	5617 (4)	9258 (5)	4682 (4)	38 (3)
O(2)	6505 (7)	6106 (4)	6234 (5)	46 (3)	5813 (3)	6397 (4)	5272 (3)	44 (4)
N	7557 (10)	7423 (7)	7137 (6)	64 (6)	6433 (5)	8110 (6)	6282 (5)	31 (1)
C(1)	8653 (11)	7011 (9)	7091 (7)	71 (7)	6924 (6)	7391 (8)	6670 (7)	51 (5)
C(2)	8821 (11)	6236 (8)	6604 (7)	60 (7)	7050 (5)	6275 (8)	6347 (5)	45 (4)
C(3)	10116 (12)	5879 (11)	6587 (8)	90 (8)	7690 (6)	5636 (9)	6784 (6)	60 (6)
C(4)	10310 (14)	5130 (13)	6220 (10)	126 (10)	7824 (6)	4596 (9)	6505 (8)	68 (6)
C(5)	9241 (16)	4696 (10)	5887 (10)	112 (10)	7286 (6)	4147 (7)	5786 (7)	57 (5)
C(6)	7953 (13)	4999 (8)	5871 (8)	73 (8)	6601 (6)	4745 (7)	5372 (6)	46 (4)
C(7)	7770 (10)	5800 (7)	6237 (7)	47 (6)	6477 (5)	5819 (7)	5644 (6)	40 (4)
C(8)	7594 (19)	8124 (9)	7736 (9)	96 (9)	6372 (8)	9179 (9)	6744 (7)	68 (6)
C(9)	6522 (10)	6848 (6)	4227 (6)	39 (5)	3549 (6)	7875 (7)	3650 (6)	39 (4)
C(10)	7162 (11)	7058 (7)	3471 (7)	51 (6)	3386 (6)	8923 (7)	3942 (7)	50 (5)
C(11)	7123 (13)	6523 (9)	2788 (7)	66 (7)	2519 (7)	9348 (7)	3954 (8)	59 (5)
C(12)	6492 (12)	5747 (8)	2838 (7)	57 (6)	1816 (6)	8740 (8)	3629 (7)	63 (6)
C(13)	5839 (11)	5539 (7)	3559 (8)	52 (6)	1951 (6)	7676 (8)	3348 (9)	66 (6)
C(14)	5864 (10)	6098 (7)	4258 (7)	46 (6)	2825 (5)	7241 (7)	3376 (7)	47 (5)
C(15)	8184 (9)	8036 (6)	5220 (6)	36 (5)	5051 (5)	7875 (6)	2410 (6)	40 (4)
C(16)	9341 (11)	7569 (8)	5018 (7)	51 (6)	5757 (6)	8617 (8)	2298 (7)	55 (5)
C(17)	10617 (11)	7917 (9)	5204 (8)	67 (8)	5981 (7)	9019 (9)	1392 (8)	64 (6)
C(18)	10784 (12)	8687 (9)	5581 (8)	72 (8)	5497 (7)	8645 (9)	599 (7)	69 (6)
C(19)	9670 (13)	9133 (9)	5794 (9)	75 (8)	4799 (9)	7934 (8)	691 (7)	73 (7)
C(20)	8417 (12)	8823 (7)	5619 (8)	58 (7)	4575 (7)	7567 (9)	1605 (7)	59 (5)
C(21)	5537 (10)	8477 (7)	4543 (6)	41 (5)	4624 (5)	5923 (6)	3530 (6)	32 (4)
C(22)	6153 (12)	9129 (7)	4112 (8)	57 (7)	4958 (5)	5328 (7)	2762 (6)	48 (4)
C(23)	5388 (14)	9711 (7)	3660 (8)	67 (8)	4863 (6)	4210 (7)	2760 (6)	48 (5)
C(24)	4041 (13)	9643 (8)	3589 (8)	68 (7)	4431 (6)	3711 (7)	3501 (7)	56 (5)
C(25)	3407 (11)	9016 (7)	4023 (8)	68 (8)	4084 (6)	4302 (8)	4280 (8)	57 (5)
C(26)	4162 (11)	8439 (8)	4504 (7)	58 (6)	4195 (6)	5387 (7)	4294 (6)	46 (4)

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

	CIS	TRANS		CIS	TRANS		CIS	TRANS
Re—Cl(1)	2·397 (3)	2·430 (2)	C(2)—C(3)	1·407 (18)	1·374 (12)	C(15)—C(16)	1·417 (14)	1·400 (12)
Re—Cl(2)	2·355 (3)	2·375 (2)	C(2)—C(7)	1·351 (16)	1·407 (11)	C(15)—C(20)	1·410 (15)	1·370 (12)
Re—P	2·485 (3)	2·471 (2)	C(3)—C(4)	1·335 (29)	1·358 (15)	C(16)—C(17)	1·396 (16)	1·386 (13)
Re—O(1)	1·660 (8)	1·701 (5)	C(4)—C(5)	1·346 (29)	1·388 (14)	C(17)—C(18)	1·362 (19)	1·387 (15)
Re—O(2)	1·955 (6)	1·949 (5)	C(5)—C(6)	1·365 (21)	1·380 (11)	C(18)—C(19)	1·371 (20)	1·363 (15)
Re—N	2·150 (10)	2·125 (6)	C(6)—C(7)	1·410 (16)	1·393 (11)	C(19)—C(20)	1·350 (17)	1·381 (13)
P—C(9)	1·823 (11)	1·825 (8)	C(9)—C(10)	1·416 (14)	1·380 (11)	C(21)—C(22)	1·396 (14)	1·382 (10)
P—C(15)	1·792 (10)	1·816 (8)	C(9)—C(14)	1·360 (14)	1·380 (11)	C(21)—C(26)	1·365 (14)	1·397 (11)
P—C(21)	1·827 (10)	1·826 (7)	C(10)—C(11)	1·364 (16)	1·387 (12)	C(22)—C(23)	1·366 (17)	1·390 (12)
O(2)—C(7)	1·347 (11)	1·319 (8)	C(11)—C(12)	1·387 (17)	1·359 (13)	C(23)—C(24)	1·340 (19)	1·355 (14)
N—C(1)	1·275 (17)	1·267 (11)	C(12)—C(13)	1·374 (16)	1·387 (14)	C(24)—C(25)	1·378 (18)	1·398 (14)
N—C(8)	1·454 (17)	1·471 (11)	C(13)—C(14)	1·408 (15)	1·402 (11)	C(25)—C(26)	1·378 (16)	1·353 (12)
C(1)—C(2)	1·462 (19)	1·463 (13)						

	CIS	TRANS		CIS	TRANS		CIS	TRANS
Cl(1)—Re—Cl(2)	86·9 (1)	169·72 (8)	C(15)—P—C(21)	104·6 (4)	107·5 (4)	C(10)—C(11)—C(12)	120 (1)	119·7 (8)
Cl(1)—Re—P	173·2 (1)	94·62 (7)	Re—O(2)—C(7)	137·2 (7)	134·0 (5)	C(11)—C(12)—C(13)	120 (1)	120·5 (9)
Cl(1)—Re—O(1)	100·7 (3)	91·4 (2)	Re—N—C(1)	128 (1)	126·2 (6)	C(12)—C(13)—C(14)	120 (1)	119·3 (9)
Cl(1)—Re—O(2)	88·5 (2)	82·1 (1)	Re—N—C(8)	116 (1)	114·9 (6)	C(9)—C(14)—C(13)	121 (1)	120·4 (8)
Cl(1)—Re—N	86·0 (3)	86·4 (2)	C(1)—N—C(8)	116 (1)	118·9 (7)	P—C(15)—C(16)	123·1 (8)	121·4 (6)
Cl(2)—Re—P	93·9 (1)	89·97 (7)	N—C(1)—C(2)	126 (1)	127·4 (8)	P—C(15)—C(20)	120·1 (8)	119·9 (7)
Cl(2)—Re—O(1)	100·1 (4)	97·6 (2)	C(1)—C(2)—C(3)	119 (1)	119·9 (8)	C(16)—C(15)—C(20)	117 (1)	118·6 (8)
Cl(2)—Re—O(2)	89·5 (2)	88·9 (1)	C(1)—C(2)—C(7)	123 (1)	120·8 (8)	C(15)—C(16)—C(17)	119 (1)	120·8 (9)
Cl(2)—Re—N	169·1 (3)	87·5 (2)	C(3)—C(2)—C(7)	118 (1)	119·2 (9)	C(16)—C(17)—C(18)	122 (1)	118·0 (9)
P—Re—O(1)	85·8 (3)	92·4 (2)	C(2)—C(3)—C(4)	121 (2)	121·4 (9)	C(17)—C(18)—C(19)	119 (1)	122·2 (9)
P—Re—O(2)	84·7 (2)	87·6 (1)	C(3)—C(4)—C(5)	119 (2)	119·9 (9)	C(18)—C(19)—C(20)	121 (1)	119 (1)
P—Re—N	92·2 (3)	170·0 (2)	C(4)—C(5)—C(6)	123 (2)	120·1 (9)	C(15)—C(20)—C(19)	123 (1)	121·7 (9)
O(1)—Re—O(2)	167·0 (4)	173·4 (2)	C(5)—C(6)—C(7)	117 (2)	119·9 (9)	P—C(21)—C(22)	122·3 (8)	123·0 (6)
O(1)—Re—N	89·4 (5)	97·5 (3)	O(2)—C(7)—C(2)	121 (1)	120·0 (8)	P—C(21)—C(26)	119·0 (8)	117·8 (6)
O(2)—Re—N	82·0 (4)	82·8 (2)	O(2)—C(7)—C(6)	118 (1)	120·7 (8)	C(22)—C(21)—C(26)	118 (1)	119·2 (7)
Re—P—C(9)	117·8 (3)	114·4 (3)	C(2)—C(7)—C(6)	121 (1)	119·2 (7)	C(21)—C(22)—C(23)	120 (1)	119·8 (9)
Re—P—C(15)	111·1 (3)	117·6 (3)	P—C(9)—C(10)	118·5 (8)	118·9 (6)	C(22)—C(23)—C(24)	121 (1)	119·9 (9)
Re—P—C(21)	114·5 (3)	110·5 (3)	P—C(9)—C(14)	122·9 (8)	122·5 (6)	C(23)—C(24)—C(25)	120 (1)	121·0 (8)
C(9)—P—C(15)	106·6 (5)	100·8 (4)	C(10)—C(9)—C(14)	118 (1)	118·6 (8)	C(24)—C(25)—C(26)	120 (1)	119·0 (9)
C(9)—P—C(21)	101·0 (5)	105·0 (4)	C(9)—C(10)—C(11)	121 (1)	121·4 (8)	C(21)—C(26)—C(25)	120 (1)	121·0 (9)

Table 3. *Selected bond distances* (Å) *and angles* (°) *in oxo complexes of* Re^{V} *with bidentate oxygenated ligands with e.s.d.'s in parentheses*

	Re–O$_{oxo}$	Re–O	O *trans* to	Re–N	N *trans* to	Re–P	P *trans* to	Re–Cl	Cl *trans* to	Re–Br	Br *trans* to	Mean angle X_{cis}–Re–O$_{oxo}$	Reference
$ReOCl_2(PPh_3)(acac)$	1·69 (1)	2·10 (1)*	O$_{oxo}$			2·431 (4)	O	2·376 (7)	Cl			96·9 (3)‡	(*a*)
		1·99 (1)	P					2·339 (7)	Cl				
$Re_2O_2Cl_4(PPh_3)(salen)$	1·68 (1)	1·92 (1)	O$_{oxo}$	2·15 (1)	Cl	2·466 (4)	Cl	2·367 (3)	N			93·8 (2)	(*b*)
	1·68 (1)	1·93 (1)	O$_{oxo}$	2·15 (1)	Cl	2·472 (4)	Cl	2·365 (3)	N			93·3 (2)	
								2·416 (4)	P				
								2·404 (4)	P				
$ReOCl_2(PPh_3)\{Ph(O)$-$CNNCMe_3\}$	1·685 (8)	2·013 (7)	O$_{oxo}$	2·13 (1)	Cl	2·472 (2)	Cl	2·344 (4)	N			94·5 (2)	(*c*)
								2·388 (3)	P				
ReOCl (Me-sal)$_2$	1·680 (4)	1·979 (4)	O$_{oxo}$	2·120 (5)	O			2·399 (2)	N			95·1 (1)	(*d*)
		1·995 (4)	N	2·103 (5)	Cl								
trans-$ReOBr_2(PPh_3)$(Ph-sal) monoclinic form	1·683 (5)	1·937 (5)	O$_{oxo}$	2·173 (6)	P	2·465 (2)	N			2·550 (1)	Br	93·9 (1)	(*e*)
										2·562 (1)	Br		
trans-$ReOBr_2(PPh_3)$(Ph-sal) triclinic form	1·680 (4)	1·972 (4)	O$_{oxo}$	2·141 (9)	P	2·482 (3)	N			2·545 (1)	Br	94·9 (1)	(*f*)
										2·547 (1)	Br		
trans-$ReOCl_2(PPh_3)$(Me-sal)	1·701 (5)	1·949 (5)	O$_{oxo}$	2·125 (6)	P	2·471 (2)	N	2·375 (2)	Cl			94·3 (1)	(*g*)
								2·430 (2)	Cl				
cis-$ReOCl_2(PPh_3)$(Me-sal)	1·660 (8)	1·955 (6)	O$_{oxo}$	2·15 (1)	Cl	2·485 (3)	Cl	2·355 (3)	N			94·1 (2)	(*h*)
								2·397 (3)	P				
Weighted mean [σ_m]†	1·683 [3]	1·970 [8]		2·132 [8]		2·470 [4]		2·389 [7]		2·551 [4]		94·5 [3]	

Abbreviations: acac = acetylacetonato; salen = *N,N'*-ethylenebis(salicylideneaminato); Me-sal and Ph-sal = *N*-methyl- and *N*-phenylsalicylideneaminato.

References: (*a*) Lock & Che'ng Wan (1975); (*b*) Bombieri *et al.* (1978); (*c*) Hursthouse *et al.* (1979); (*d*) Gilli *et al.* (1982); (*e*) Bertolasi *et al.* (1982); (*f*) Sacerdoti *et al.* (1984); (*g, h*) present work.

* Not included in the average.

† Averages and their standard errors are calculated by $x_m = \sum f_i x_i / \sum f_i$ and $\sigma_m = \{\sum f_i (x_i - x_m)^2 / [(N-1)\sum f_i]\}^{1/2}$, where $f_i = 1/\sigma_i^2$ and the summation is from 1 to N.

‡ Standard errors of the single angles are calculated by $\sigma'_m = 1/\sum f_i$(average as above).

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The Structure of *cis*-Dichlorobis[dimethyl(phenyl)phosphine]-(*N*-phenylsalicylideneaminato)rhenium(III), [$Re(C_{13}H_{10}NO)Cl_2\{P(CH_3)_2(C_6H_5)\}_2$]

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Abstract. $M_r = 729{\cdot}6$, monoclinic, $P2_1/c$, $a = 17{\cdot}411$ (3), $b = 10{\cdot}710$ (3), $c = 20{\cdot}967$ (4) Å, $\beta = 129{\cdot}58$ (4)°, $U = 3013$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}61$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 4{\cdot}13$ mm^{-1}, $F(000) = 1440$, $T = 298$ K, $R = 0{\cdot}036$ for 3895 reflections having $I > 3\sigma(I)$. The coordination is nearly octahedral with the two Cl in *cis* and the two P atoms in *trans* positions. Re^{III}–X (X = O, N, Cl, P) distances

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