C14	0.4256 (5)	0.5757 (3)	0.6769 (2)	0.037 (1)
C15	0.3156 (4)	0.5434 (3)	0.7494 (2)	0.033 (1)
C16	0.2308 (4)	0.4032 (3)	0.7605 (2)	0.035 (1)
C17	0.1519 (5)	0.3848 (3)	0.8480 (3)	0.043 (1)
C18	0.0723 (5)	0.2536 (3)	0.8594 (3)	0.055 (1)
C19	0.0697 (5)	0.1422 (3)	0.7847 (3)	0.061 (1)
C20	0.1435 (6)	0.1585 (4)	0.6979 (4)	0.061 (1)
C21	0.2246 (5)	0.2882 (3)	0.6852 (3)	0.049 (1)
C22	0.7167 (6)	0.8171 (4)	0.9210 (4)	0.066 (1)

Table 2. *Selected geometric parameters (Å, °)*

Mo—O1	1.968 (2)	N1—N2	1.410 (3)
Mo—O2	1.930 (2)	N1—C11	1.284 (5)
Mo—O3	2.415 (3)	N2—C13	1.297 (5)
Mo—O4	1.714 (2)	C1—C11	1.484 (5)
Mo—O5	1.689 (3)	C12—C13	1.498 (5)
Mo—N2	2.201 (3)	C13—C14	1.437 (4)
O1—C11	1.333 (4)	C14—C15	1.352 (5)
O2—C15	1.329 (4)	C15—C16	1.481 (4)
O3—C22	1.399 (6)		
O1—Mo—O2	148.5 (2)	N2—N1—C11	109.4 (2)
O1—Mo—O3	77.41 (9)	Mo—N2—N1	115.0 (2)
O1—Mo—O4	94.0 (1)	Mo—N2—C13	129.3 (3)
O1—Mo—O5	100.0 (1)	N1—N2—C13	115.7 (3)
O1—Mo—N2	72.74 (9)	C2—C1—C11	116.8 (3)
O2—Mo—O3	78.9 (1)	C10—C1—C11	123.0 (2)
O2—Mo—O4	103.1 (1)	O1—C11—C1	114.3 (3)
O2—Mo—O5	100.5 (1)	N1—C11—C1	123.2 (3)
O2—Mo—N2	81.6 (1)	O1—C11—N1	122.5 (3)
O3—Mo—O4	82.2 (2)	N2—C13—C12	121.6 (3)
O3—Mo—O5	171.9 (1)	N2—C13—C14	120.6 (3)
O3—Mo—N2	75.65 (9)	C12—C13—C14	117.9 (3)
O4—Mo—O5	105.7 (1)	C13—C14—C15	126.2 (3)
O4—Mo—N2	156.12 (9)	O2—C15—C14	122.0 (3)
O5—Mo—N2	96.2 (1)	O2—C15—C16	113.4 (3)
Mo—O1—C11	120.1 (2)	C14—C15—C16	124.4 (3)
Mo—O2—C15	134.0 (2)	C15—C16—C17	119.7 (3)
Mo—O3—C22	133.0 (2)	C15—C16—C21	121.7 (4)

The structure was solved by Patterson and difference Fourier methods and refined by full-matrix least-squares methods. All H atoms were found in the difference Fourier map, but they were placed at calculated positions (C—H and N—H 0.95 Å, O—H 0.92 Å). H atoms were allowed to ride on their attached atom with $U(H) = 1.2U(\text{carrier atom})$.

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SDP* (Frenz, 1982). Program(s) used to refine structure: *SDP*. Molecular graphics: *ORTEX3* (McArdle, 1994). Software used to prepare material for publication: *CSU* (Vicković, 1988).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Tetrachlorobis(triphenylphosphine-*P*)-rhenium(IV)–*mer-trans*-Trichlorooxobis(triphenylphosphine-*P*)rhenium(V) (0.86/0.14)

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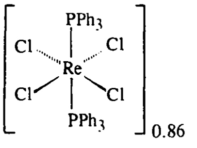
Abstract

trans-Tetrachlorobis(triphenylphosphine)rhenium(IV), (1), is isomorphous with the triclinic polymorph of *mer-trans*-trichlorooxobis(triphenylphosphine)rhenium(V), (2) [Lebuis & Beauchamp (1993). *Can. J. Chem.* **71**, 441–449]. The sample of (1) has been partially oxidized (14–25%) to (2) by aerial oxidation, resulting in the title compound, $[ReCl_4(C_{18}H_{15}P)_2]_{0.86}$.$[ReCl_3O(C_{18}H_{15}P)_2]_{0.14}$. In the crystal lattice, the oxo ligand is located primarily at one of six possible sites. The molecules have *trans*-octahedral coordination geometry, with Re—P distances in the range 2.564 (1)–2.580 (1) Å, having a mean value of 2.571 (7) Å, and Re—Cl distances in the range 2.291 (3)–2.339 (1) Å.

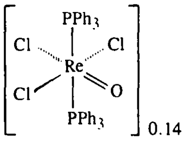
Comment

There are 1.5 molecules in the asymmetric unit of the title compound [(1):(2) 0.86:0.14], with one molecule situated at a centre of symmetry [molecule (1); Fig. 1] and one in a general position [molecule (2); Fig. 2]. The red crystals are isomorphous with the yellow triclinic crystal form of the oxidation product *mer-trans*-trichlorooxobis(triphenylphosphine)rhenium(V), (2) (Lebuis & Beauchamp, 1993). The structure of the monoclinic polymorph of (2) was also reported by Lebuis &

Beauchamp (1993). In triclinic (2), the molecule lying at the centre of symmetry is (necessarily) disordered, while that in the general position is ordered. In the case of partially oxidized (1), the reverse is true. The location of the partial oxo ligand in (1) corresponds to that for the molecule of triclinic (2) in the general position. The refined occupancy for Cl(3) yields a composition of 86% (1) and 14% (2) for this solid solution.



(1)



(2)

A similar situation involving solid solutions of Mo complexes was reported as the solution to the long-standing question of bond-stretch isomerism (Yoon, Parkin & Rheingold, 1992). A key point of this work was the sensitivity of metal–halide bond lengths to minor disordering with oxo ligands resulting from partial oxidation. In view of these findings, there is probably a small amount of oxide at other Cl sites in the structure, namely the Cl(6) and Cl(1) sites on the basis of the slightly shorter Re—Cl distances and marginally higher displacement parameters. The Cl(6) site of (1) corresponds to the disordered Cl/O site of the centrosymmetric molecule of triclinic (2), while the Cl(1) site of (1), *trans* to the partial oxo ligand, also corresponds to a site in (2) that probably contains a trace of oxo ligand. The presence of additional oxide is further supported by elemental analysis of a crystal from the same batch as that used for data collection (see *Experimental*) which gives values consistent with 25% conversion to (2). Some additional oxidation of the sample may have taken place during preparation of the sample for elemental analysis, so that the amount of Re^V oxide (2) present in the crystal studied lies between these limiting values of 14 and 25%. It is noteworthy that most of the oxide in the sample is located at a single site.

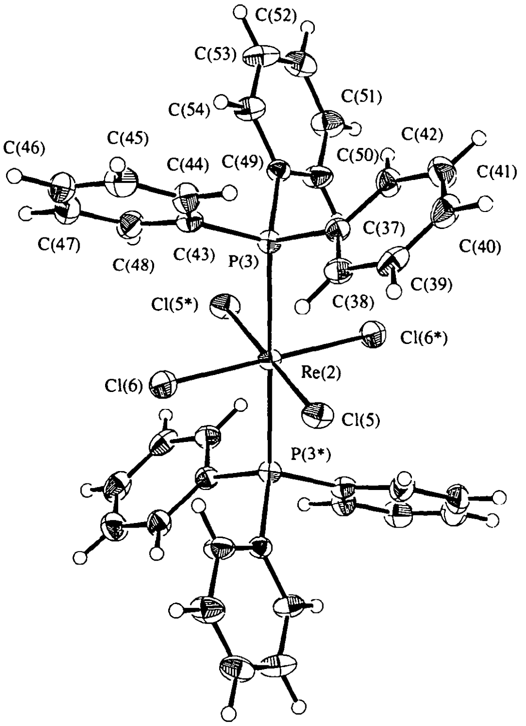


Fig. 1. Perspective view of molecule (1) (centrosymmetric) with 33% probability ellipsoids shown for non-H atoms.

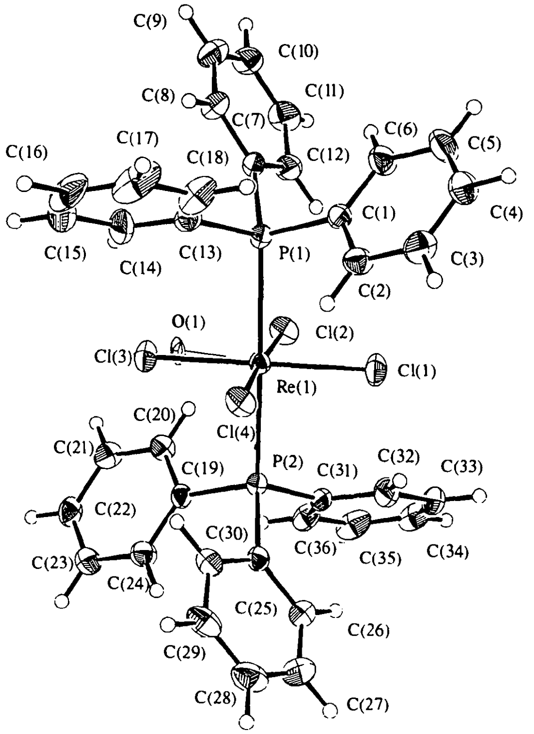


Fig. 2. Perspective view of molecule (2) (general position) with 33% probability ellipsoids shown for non-H atoms.

The two independent molecules (Figs. 1 and 2) differ primarily in the orientation of the triphenylphosphine ligands with respect to the $ReCl_4$ equatorial plane. The Re—Cl distances in the Re^{IV} complex (1) (mean 2.322 Å) are shorter than those reported for the Re^V complex (2) [mean 2.390 Å for the monoclinic form and 2.360 Å for the ordered molecule of triclinic (2)]. This is probably a result of a general weakening of the Re—Cl bonds by the very strong interaction of the metal with the oxo ligand in (2). The Re—O distance of 1.71 (3) Å in the present structure is not significantly different from the values of 1.73 (2) Å observed for the ordered molecule of triclinic (2) and 1.663 (5) Å observed for monoclinic (2). The mean Re—P distance of 2.571 Å for (1) is longer than the average of 2.529 Å for the two polymorphs of (2), although it is about the same as the value of 2.562 (9) Å for the ordered molecule of triclinic (2). Intramolecular C—H···Cl interactions [C···Cl 3.166 (6)–3.518 (5) Å] probably play a role in stabilizing the phenyl-ring orientations, while intermolecular C—H···Cl interactions [C···Cl 3.402 (5)–3.797 (7) Å] represent the most significant intermolecular contacts.

Experimental

In an attempt to prepare an Re^{IV} complex of the potentially hexadentate ligand *P,P,P′,P′*-tetrakis(*o*-hydroxyphenyl)diphosphinoethane (abbreviated $H_4P_2O_4$) (Luo & Orvig, 1996), a suspension of the hydrochloride adduct of the ligand (abbreviated $H_4P_2O_4.2HCl$) (54 mg, 0.1 mmol) and $[ReCl_4(PPh_3)_2]$ (Rouchias & Wilkinson, 1966) (84 mg, 0.1 mmol) in EtOH (25 ml) was refluxed under an N_2 atmosphere overnight. The brown–red mixture turned to a clear red solution and a coral-red precipitate formed after about 4 h. The red precipitate was recrystallized from CH_2Cl_2, resulting in red crystals suitable for X-ray crystallographic analysis. Complete details of the synthesis of the $H_4P_2O_4.2HCl$ adduct will be published elsewhere (Luo & Orvig, 1996).

Crystal data

$[ReCl_4(C_{18}H_{15}P)_2]_{0.86}$.-$[ReCl_3O(C_{18}H_{15}P)_2]_{0.14}$
$M_r = 849.80$
Triclinic
$P\overline{1}$
$a = 14.204\ (2)$ Å
$b = 16.381\ (3)$ Å
$c = 11.878\ (2)$ Å
$\alpha = 96.13\ (1)°$
$\beta = 92.42\ (1)°$
$\gamma = 64.94\ (1)°$
$V = 2489.2\ (7)$ Å^3
$Z = 3$
$D_x = 1.701$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12.6$–$13.5°$
$\mu = 4.10$ mm^{-1}
$T = 294.2$ K
Prism
0.35 × 0.20 × 0.12 mm
Red

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.853$, $T_{max} = 1.000$
18 631 measured reflections
18 002 independent reflections

8572 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.037$
$\theta_{max} = 32.5°$
$h = 0 \rightarrow 21$
$k = -21 \rightarrow 24$
$l = -17 \rightarrow 17$
3 standard reflections monitored every 200 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.032$
$wR = 0.025$
$S = 1.240$
8572 reflections
594 parameters
H atoms riding with C—H 0.98 Å
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.005$

$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -1.07$ e Å^{-3}
Extinction correction: Zachariasen (1967) type 2, Gaussian isotropic
Extinction coefficient: $1.0\ (2) \times 10^{-8}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Re(1)	0.33731 (2)	0.33707 (1)	0.15430 (2)	0.02931 (6)
Re(2)	0	0	1/2	0.03017 (9)
Cl(1)	0.2208 (1)	0.48893 (9)	0.1555 (1)	0.0497 (4)
Cl(2)	0.4694 (1)	0.38419 (9)	0.2007 (1)	0.0467 (4)
Cl(3)†	0.4466 (2)	0.1862 (2)	0.1505 (2)	0.0467 (6)
Cl(4)	0.1981 (1)	0.29823 (9)	0.1135 (1)	0.0440 (4)
Cl(5)	0.1252 (1)	−0.14968 (9)	0.4856 (1)	0.0483 (4)
Cl(6)	−0.1321 (1)	−0.04651 (10)	0.4790 (1)	0.0503 (4)
P(1)	0.3517 (1)	0.33039 (9)	−0.0626 (1)	0.0316 (4)
P(2)	0.3217 (1)	0.34039 (8)	0.36950 (10)	0.0310 (4)
P(3)	−0.0070 (1)	0.01202 (9)	0.71677 (10)	0.0317 (4)
O(1)†	0.432 (2)	0.229 (2)	0.149 (2)	0.035 (5)
C(1)	0.2450 (4)	0.4255 (3)	−0.1218 (3)	0.035 (1)
C(2)	0.1422 (4)	0.4406 (4)	−0.1021 (4)	0.046 (2)
C(3)	0.0605 (4)	0.5112 (4)	−0.1462 (4)	0.058 (2)
C(4)	0.0798 (5)	0.5677 (4)	−0.2108 (5)	0.060 (2)
C(5)	0.1810 (5)	0.5543 (4)	−0.2286 (5)	0.067 (2)
C(6)	0.2638 (4)	0.4835 (4)	−0.1845 (4)	0.051 (2)
C(7)	0.4705 (4)	0.3276 (3)	−0.1209 (4)	0.034 (1)
C(8)	0.5152 (4)	0.2741 (3)	−0.2199 (4)	0.047 (2)
C(9)	0.6041 (4)	0.2752 (4)	−0.2651 (4)	0.058 (2)
C(10)	0.6462 (4)	0.3290 (4)	−0.2129 (5)	0.054 (2)
C(11)	0.6040 (5)	0.3827 (4)	−0.1141 (5)	0.060 (2)
C(12)	0.5149 (4)	0.3831 (3)	−0.0696 (4)	0.048 (2)
C(13)	0.3443 (4)	0.2292 (4)	−0.1357 (4)	0.042 (2)
C(14)	0.4170 (5)	0.1455 (4)	−0.1120 (5)	0.064 (2)
C(15)	0.4122 (7)	0.0677 (5)	−0.1661 (6)	0.094 (3)
C(16)	0.3372 (8)	0.0735 (6)	−0.2435 (7)	0.095 (3)
C(17)	0.2664 (6)	0.1556 (6)	−0.2694 (6)	0.093 (3)
C(18)	0.2699 (5)	0.2351 (4)	−0.2161 (5)	0.064 (2)
C(19)	0.4271 (4)	0.2420 (3)	0.4233 (3)	0.034 (1)
C(20)	0.5295 (4)	0.2197 (3)	0.3924 (4)	0.042 (1)
C(21)	0.6110 (4)	0.1459 (4)	0.4319 (4)	0.047 (2)
C(22)	0.5906 (4)	0.0927 (3)	0.5031 (4)	0.050 (2)
C(23)	0.4903 (5)	0.1158 (4)	0.5352 (4)	0.054 (2)
C(24)	0.4090 (4)	0.1900 (3)	0.4957 (4)	0.045 (2)
C(25)	0.2048 (4)	0.3347 (3)	0.4198 (4)	0.034 (1)
C(26)	0.1444 (4)	0.3949 (3)	0.5067 (4)	0.049 (2)
C(27)	0.0609 (5)	0.3840 (4)	0.5475 (5)	0.066 (2)
C(28)	0.0375 (5)	0.3135 (5)	0.5023 (5)	0.064 (2)
C(29)	0.0972 (5)	0.2536 (4)	0.4149 (5)	0.056 (2)
C(30)	0.1806 (4)	0.2645 (3)	0.3744 (4)	0.043 (2)
C(31)	0.3305 (4)	0.4378 (3)	0.4506 (4)	0.034 (1)
C(32)	0.2620 (5)	0.5250 (4)	0.4279 (4)	0.055 (2)
C(33)	0.2723 (5)	0.5998 (4)	0.4872 (5)	0.060 (2)
C(34)	0.3495 (6)	0.5868 (5)	0.5672 (5)	0.065 (2)
C(35)	0.4137 (5)	0.5023 (5)	0.5910 (5)	0.066 (2)
C(36)	0.4060 (4)	0.4267 (4)	0.5340 (4)	0.047 (2)
C(37)	0.1156 (4)	−0.0621 (3)	0.7787 (3)	0.035 (1)
C(38)	0.1519 (4)	−0.1556 (3)	0.7561 (4)	0.042 (1)
C(39)	0.2437 (4)	−0.2124 (3)	0.8053 (4)	0.050 (2)
C(40)	0.2998 (4)	−0.1775 (4)	0.8749 (4)	0.056 (2)
C(41)	0.2642 (4)	−0.0847 (4)	0.8954 (4)	0.058 (2)
C(42)	0.1726 (4)	−0.0278 (3)	0.8484 (4)	0.044 (1)
C(43)	−0.1052 (4)	−0.0185 (3)	0.7697 (4)	0.036 (1)
C(44)	−0.0834 (4)	−0.0832 (3)	0.8451 (4)	0.044 (2)
C(45)	−0.1612 (5)	−0.1032 (4)	0.8832 (4)	0.055 (2)
C(46)	−0.2609 (5)	−0.0576 (4)	0.8487 (4)	0.059 (2)
C(47)	−0.2847 (4)	0.0078 (4)	0.7759 (4)	0.062 (2)
C(48)	−0.2080 (4)	0.0284 (4)	0.7375 (4)	0.049 (2)
C(49)	−0.0378 (4)	0.1245 (3)	0.7907 (4)	0.033 (1)
C(50)	−0.0032 (4)	0.1831 (3)	0.7496 (4)	0.050 (2)

C(51)	−0.0190 (5)	0.2647 (4)	0.8117 (5)	0.059 (2)
C(52)	−0.0714 (5)	0.2895 (3)	0.9133 (4)	0.054 (2)
C(53)	−0.1062 (5)	0.2321 (4)	0.9534 (4)	0.058 (2)
C(54)	−0.0898 (4)	0.1495 (3)	0.8935 (4)	0.046 (1)

† Partial occupancy (see below).

Table 2. *Selected geometric parameters (Å, °)*

Re(1)—Cl(1)	2.331 (1)	P(1)—C(1)	1.834 (4)
Re(1)—Cl(2)	2.339 (1)	P(1)—C(7)	1.832 (5)
Re(1)—Cl(3)	2.291 (3)	P(1)—C(13)	1.829 (5)
Re(1)—Cl(4)	2.338 (1)	P(2)—C(19)	1.828 (5)
Re(1)—P(1)	2.580 (1)	P(2)—C(25)	1.830 (5)
Re(1)—P(2)	2.568 (1)	P(2)—C(31)	1.822 (5)
Re(1)—O(1)	1.71 (3)	P(3)—C(37)	1.828 (5)
Re(2)—Cl(5)	2.334 (1)	P(3)—C(43)	1.822 (5)
Re(2)—Cl(6)	2.301 (1)	P(3)—C(49)	1.833 (5)
Re(2)—P(3)	2.564 (1)		
Cl(1)—Re(1)—Cl(2)	88.43 (5)	Cl(5)—Re(2)—P(3^{i})	86.50 (4)
Cl(1)—Re(1)—Cl(3)	177.69 (8)	Cl(6)—Re(2)—Cl(6^{i})	180.0
Cl(1)—Re(1)—Cl(4)	88.51 (5)	Cl(6)—Re(2)—P(3)	93.02 (4)
Cl(1)—Re(1)—P(1)	88.59 (4)	Cl(6)—Re(2)—P(3^{i})	86.98 (4)
Cl(1)—Re(1)—P(2)	92.22 (4)	P(3)—Re(2)—P(3^{i})	180.0
Cl(1)—Re(1)—O(1)	174.1 (7)	Re(1)—P(1)—C(1)	112.4 (1)
Cl(2)—Re(1)—Cl(3)	93.86 (7)	Re(1)—P(1)—C(7)	118.0 (2)
Cl(2)—Re(1)—Cl(4)	176.54 (5)	Re(1)—P(1)—C(13)	112.3 (1)
Cl(2)—Re(1)—P(1)	96.88 (4)	C(1)—P(1)—C(7)	105.2 (2)
Cl(2)—Re(1)—P(2)	84.33 (4)	C(1)—P(1)—C(13)	104.8 (2)
Cl(2)—Re(1)—O(1)	86.5 (7)	C(7)—P(1)—C(13)	103.0 (2)
Cl(3)—Re(1)—Cl(4)	89.20 (7)	Re(1)—P(2)—C(19)	111.0 (1)
Cl(3)—Re(1)—P(1)	90.90 (6)	Re(1)—P(2)—C(25)	116.1 (2)
Cl(3)—Re(1)—P(2)	88.24 (6)	Re(1)—P(2)—C(31)	114.4 (1)
Cl(4)—Re(1)—P(1)	84.67 (4)	C(19)—P(2)—C(25)	103.2 (2)
Cl(4)—Re(1)—P(2)	94.17 (4)	C(19)—P(2)—C(31)	104.7 (2)
Cl(4)—Re(1)—O(1)	96.7 (7)	C(25)—P(2)—C(31)	106.3 (2)
P(1)—Re(1)—P(2)	178.56 (4)	Re(2)—P(3)—C(37)	112.7 (1)
P(1)—Re(1)—O(1)	89.2 (6)	Re(2)—P(3)—C(43)	113.2 (2)
P(2)—Re(1)—O(1)	90.1 (6)	Re(2)—P(3)—C(49)	116.2 (1)
Cl(5)—Re(2)—Cl(5^{i})	180.0	C(37)—P(3)—C(43)	105.6 (2)
Cl(5)—Re(2)—Cl(6)	91.34 (5)	C(37)—P(3)—C(49)	104.7 (2)
Cl(5)—Re(2)—Cl(6^{i})	88.66 (5)	C(43)—P(3)—C(49)	103.3 (2)
Cl(5)—Re(2)—P(3)	93.50 (4)		

Symmetry code: (i) $-x, -y, 1 - z$.

There are 1.5 molecules in the asymmetric unit, one in a general position and one at a centre of symmetry. The oxo ligand was detectable only at one Cl site of the molecule in a general position. An anomalous displacement parameter and a short Re—Cl distance for Cl(3) indicated the presence of an oxo ligand at this site. A partial O atom, O(1), was included in the model. The population parameter of atom Cl(3) was refined with the constraint that the Cl(3) and O(1) populations summed to unity. The final values of the population parameters were 0.797 (8) for Cl(3) and 0.203 for O(1). The largest peaks and troughs on the final difference map were near the Re atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *DIRDIF* (Beurskens *et al.* 1994). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including hydrogen-bonding parameters, have been deposited with the IUCr (Reference: FG1149). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Z)-2,3-Bis(4′-[2.2.2]paracyclophanyl)but-2-ene and μ-η^6:η^6-[(Z)-2,3-Bis(4′-[2.2.2]paracyclophanyl)but-2-ene]-bis[(hexafluoroantimonato-*F*)silver(I)] Toluene Dichloromethane (1/1/1)

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Abstract

The free ligand, $C_{52}H_{52}$, displays approximate twofold symmetry, with a C═C bond of 1.337 (4) Å and essentially planar olefin geometry. The (methyl C)–(olefin

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