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[1,2-Bis(diphenylphosphino)ethane]dichloro-(methoxo)oxorhenium(V) dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.013 Å
Some non-H atoms missing
R factor = 0.047
wR factor = 0.122
Data-to-parameter ratio = 19.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Re(CH_3O)Cl_2O(C_{26}H_{24}P_2)]\cdot CH_2Cl_2$, contains slightly distorted octahedrally coordinated molecules in which the methoxo group is *trans* to the oxo ligand. A *cis* arrangement of the Cl atoms is imposed in the equatorial plane by the bidentate diphosphine ligand. The methoxo group is approximately end-on coordinated [Re—O—CH_3 = 168.1 (5)°].

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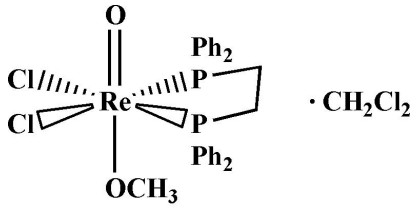
Comment

The title compound, (I), was prepared as part of a spectroscopic study on a series of oxo-rhenium(V) complexes with various diphosphines and various ligands *trans* to the Re=O bond (Baril-Robert & Beauchamp, 2003).



(I)

The stereochemistry about the Re atom is the same as observed in related complexes with different alkoxo groups. The Re atom is displaced by 0.0631 (8) Å from the P_2Cl_2 plane on the oxo side. The reduced steric requirement of the approximately end-on coordinated methoxo group removes some of the distortions noted for compounds with bulkier alkoxo groups.

Experimental

$ReOCl_3(OPPh_3)(Me_2S)$ (0.16 mmol) was stirred in a refluxing solution of 1,2-bis(diphenylphosphino)ethane (0.16 mmol) in methanol (10 ml). The initially green suspension turned purple and, after 3 days, a purple solid was collected by filtration, washed with methanol and diethyl ether, and dried *in vacuo*. Crystals were obtained by vapor diffusion of $CHCl_3$ into a solution in CH_2Cl_2.

Crystal data

$[Re(CH_3O)Cl_2O(C_{26}H_{24}P_2)]\cdot CH_2Cl_2$
M_r = 787.45
Monoclinic, $P2_1/c$
a = 11.3352 (1) Å
b = 20.4411 (1) Å
c = 13.9594 (1) Å
β = 108.303 (1)°
V = 3070.81 (4) Å^3
Z = 4
D_x = 1.703 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 27775 reflections
θ = 3.3–72.8°
μ = 11.96 mm^{-1}
T = 223 (2) K
Platelet, purple
0.42 × 0.12 × 0.06 mm

Data collection

Bruker SMART 2K/Platform diffractometer
ω scans
Absorption correction: Gaussian (*ABSORP* in *NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.080, T_{max} = 0.530
36674 measured reflections
6069 independent reflections
5369 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 72.9°
$h = -14 \rightarrow 14$
$k = -24 \rightarrow 25$
$l = -15 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.122
S = 1.11
6069 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 12.2591P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 2.34 e Å^{-3}
$\Delta\rho_{min}$ = −1.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re—O1	1.714 (5)	Re—P1	2.4348 (16)
Re—O2	1.872 (4)	Re—Cl12	2.4380 (15)
Re—P2	2.4241 (15)	Re—Cl11	2.4495 (15)
O1—Re—O2	172.18 (19)	P2—Re—Cl12	176.17 (6)
O1—Re—P2	88.15 (15)	P1—Re—Cl12	95.41 (5)
O2—Re—P2	85.83 (14)	O1—Re—Cl11	94.95 (15)
O1—Re—P1	87.33 (15)	O2—Re—Cl11	90.52 (14)
O2—Re—P1	86.98 (14)	P2—Re—Cl11	94.59 (5)
P2—Re—P1	83.02 (5)	P1—Re—Cl11	176.66 (5)
O1—Re—Cl12	95.27 (15)	Cl12—Re—Cl11	86.82 (6)
O2—Re—Cl12	90.60 (14)	C61—O2—Re	168.1 (5)
Re—P1—C11—C12	56.6 (6)	C21—P1—C51—C52	169.4 (5)
Re—P1—C21—C26	−6.2 (7)	C11—P1—C51—C52	−79.5 (5)
Re—P2—C31—C36	−4.0 (8)	C31—P2—C52—C51	169.6 (5)
Re—P2—C41—C42	61.8 (7)	C41—P2—C52—C51	−81.1 (5)

The H atoms were positioned geometrically (C—H 0.93–0.98) and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $1.2U_{eq}(C)$ for others. An electron density map showed two regions centered at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) and ($\frac{1}{2}$, 0, 0), containing peaks due to severely disordered solvent. No consistent models for dichloromethane molecules could be assembled from these peaks. This part of the structure was modeled by using the SQUEEZE procedure of *PLATON* (Spek, 2003), which indicated the presence of two cavities of 280 Å^3, each occupied by 106 electrons, which is consistent with the presence of two CH_2Cl_2 molecules per cavity (one per asymmetric unit). The contribution of the disordered solvent was calculated with *BYPASS* (van der Sluis & Spek, 1990) and a new data set without the solvent contribution was generated. The final model consisting of the ordered part only was refined. The solvent molecules were taken into account for the calculation of the crystal data. The highest peak and deepest hole are within 1 Å of the Re atom.

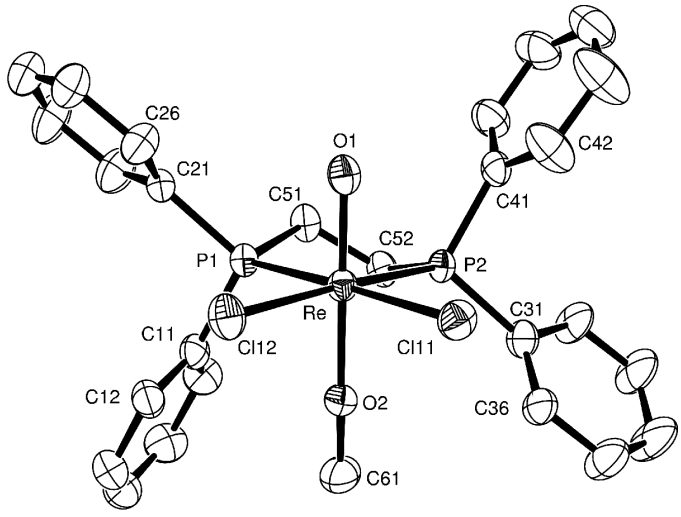


Figure 1
ORTEPII (Johnson, 1976) view of the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms are omitted for simplicity. In the phenyl-ring numbering scheme, the last digit corresponds to the position around the ring, starting with 1 for the *ipso* position.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *UdMX* (local program).

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References

Baril-Robert, F. & Beauchamp, A. L. (2003). *Can. J. Chem.* **81**, 1326–1340.
Bruker (1999). *SMART* (Version 5.059) and *SAINT* (Version 6.06). Bruker AXS Inc., Madison, USA.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Johnson, C. K. (1976). *ORTEPII*. Technical Report ORNL-5138. Oak Ridge National Laboratory, Tenessee, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sluis, P. van der & Spek, A. L. (1990). *Acta Cryst.* A**46**, 194–201.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.