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Pentachloro-1,3,6-tris(diethylphenylphosphino)dirhenium(II,III)

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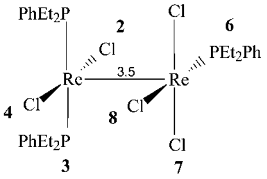
The title complex [systematic name: pentachloro-1κ^3*Cl*,2κ^2*Cl*-tris(diethylphenylphosphino)-1κ*P*,2κ^2*P*-dirhenium(II,III)-(*Re*—*Re*)], 1,3,6-$Re_2Cl_5(PEt_2Ph)_3$ or $[Re_2Cl_5(C_{10}H_{15}P)_3]$, consists of dirhenium molecules with eclipsed structures similar to those of previously characterized 1,3,6-$Re_2Cl_5(PR_3)_3$ compounds. The Re—Re bond distance is 2.2262 (3) Å and the metal–metal bond order is 3.5.

Comment

Although many dirhenium compounds with multiple metal–metal bonds and tertiary phosphine ligands (PR_3) of the type $Re_2Cl_5(PR_3)_3$ are known (Cotton & Walton, 1993), none containing diethylphenylphosphine ligands have been reported previously. Structures have been determined for 1,2,7- and 1,3,6-Re_2Cl_5(trimethylphosphine)$_3$ and 1,3,6-Re_2Cl_5(triethylphosphine)$_3$ (Cotton *et al.*, 1990), 1,2,7- and 1,3,6-Re_2Cl_5(dimethylphenylphosphine)$_3$ (Cotton *et al.*, 1998), 1,3,6-Re_2Cl_5(tri-*n*-propylphosphine)$_3$ (Cotton *et al.*, 1999) and 1,3,6-Re_2Cl_5(dicyclohexylphosphine)$_3$ (Angaridis *et al.*, 2002). The title compound, (I), was prepared from the quadruply bonded $[Re_2Cl_8]^{2-}$ ion in a solvent mixture containing a room-temperature ionic liquid.



(I)

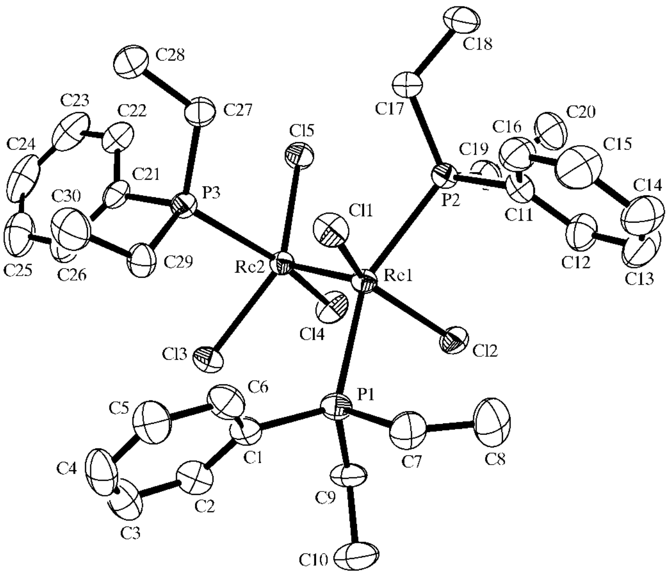


Figure 1
The molecular structure of the title compound shown with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

As expected, the molecular structure of (I) (Fig. 1) is similar to that of the five previously characterized 1,3,6-$Re_2Cl_5(PR_3)_3$ compounds. In each molecule, one Re atom is bound to two *trans*-phosphine ligands, while the other Re atom is bound to one phosphine ligand. The two Re atoms are also bound to one another, with a metal–metal bond order of 3.5. The rotational conformation is essentially eclipsed, as opposed to the somewhat staggered conformations adopted by 1,3,6-Re_2Cl_5(dicyclohexylphosphine)$_3$ and the two known 1,2,7-$Re_2Cl_5(PR_3)_3$ complexes. The Re—Re bond distance of 2.2262 (3) Å is slightly longer than those found in the other 1,3,6-$Re_2Cl_5(PR_3)_3$ compounds. This is presumably due to some additional steric crowding introduced by the larger PEt_2Ph ligands. The average Re—P and Re—Cl bond lengths [2.459 (6) and 2.345 (4) Å, respectively; Table 1] are well within the ranges [2.425 (4)–2.478 (5) Å for Re—P and 2.313 (4)–2.416 (8) Å for Re—Cl] observed previously in these complexes. A threefold disordering of the dirhenium unit is a common feature of multiply bonded dirhenium complexes of the types $Re_2Cl_4(PR_3)_3$ and $Re_2Cl_5(PR_3)_3$ (Cotton *et al.*, 1999), but no such disorder exists in 1,3,6-$Re_2Cl_5(PEt_2Ph)_3$.

Experimental

For the preparation of the title compound, tetra-*n*-butylammonium octachlorodirhenate(III) (508 mg, 0.445 mmol) was combined with 1-butyl-3-methylimidazolium hexafluorophosphate (7 ml) and ethanol (7 ml) in a round-bottomed flask under a nitrogen atmosphere. Two drops of concentrated hydrochloric acid were added followed by diethylphenylphosphine (1.00 g, 6.02 mmol). The mixture was stirred for 30 min at 298 K. Tetrahydrofuran (15 ml) was added and the mixture was stirred for 1 h. Diethyl ether (60 ml) was added next and the mixture was stirred for 2 h, then allowed to sit for 14 h. This resulted in two distinct liquid layers, *i.e.* a larger brown–green top layer and a small dark-brown bottom layer. The layers were separated in air using a separatory funnel and the top layer was placed in a large Schlenk flask. Dark crystals of the title compound formed as this solution was allowed to evaporate slowly.

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Crystal data

$[Re_2Cl_5(C_{10}H_{15}P)_3]$
$M_r = 1048.22$
Orthorhombic, *Pbca*
$a = 15.5374\ (7)$ Å
$b = 20.7751\ (9)$ Å
$c = 22.3924\ (10)$ Å
$V = 7228.1\ (6)$ Å^3
$Z = 8$
$D_x = 1.927$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4402 reflections
$\theta = 2.4$–$27.4°$
$\mu = 7.22$ mm^{-1}
$T = 213\ (2)$ K
Plate, red
0.12 × 0.11 × 0.08 mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
$T_{min} = 0.410$, $T_{max} = 0.561$
34 336 measured reflections
8235 independent reflections
5875 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 27.6°$
$h = -20 \rightarrow 18$
$k = -21 \rightarrow 27$
$l = -24 \rightarrow 28$
50 standard reflections
frequency: 560 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.048$
$S = 1.01$
8235 reflections
367 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.009$
$\Delta\rho_{max} = 1.64$ e Å^{-3}
$\Delta\rho_{min} = -1.11$ e Å^{-3}

H atoms were treated as riding, with C—H distances in the range 0.94–0.98 Å, and U_{iso} values of $1.2U_{eq}$(C) and $1.5U_{eq}$(C) for phenyl and ethyl C atoms, respectively.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL*97.

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Table 1
Selected geometric parameters (Å, °).

Re1—Re2	2.2262 (3)	Re2—Cl3	2.3314 (12)
Re1—Cl2	2.3453 (12)	Re2—Cl5	2.3436 (12)
Re1—Cl1	2.3556 (12)	Re2—Cl4	2.3469 (13)
Re1—P2	2.4525 (12)	Re2—P3	2.4551 (13)
Re1—P1	2.4703 (13)		
Re2—Re1—Cl2	103.86 (3)	Re1—Re2—Cl3	108.23 (4)
Re2—Re1—Cl1	112.83 (3)	Re1—Re2—Cl5	109.51 (3)
Cl2—Re1—Cl1	143.29 (5)	Cl3—Re2—Cl5	142.19 (5)
Re2—Re1—P2	103.76 (4)	Re1—Re2—Cl4	104.17 (3)
Cl2—Re1—P2	83.86 (4)	Cl3—Re2—Cl4	86.39 (5)
Cl1—Re1—P2	86.25 (4)	Cl5—Re2—Cl4	86.55 (5)
Re2—Re1—P1	104.88 (3)	Re1—Re2—P3	101.04 (3)
Cl2—Re1—P1	84.40 (4)	Cl3—Re2—P3	84.93 (4)
Cl1—Re1—P1	87.42 (4)	Cl5—Re2—P3	85.91 (4)
P2—Re1—P1	150.89 (5)	Cl4—Re2—P3	154.77 (5)
Cl1—Re1—Re2—P3	−4.10 (5)	Cl2—Re1—Re2—Cl4	−1.88 (5)
P2—Re1—Re2—Cl5	−6.28 (5)	P1—Re1—Re2—Cl3	1.16 (5)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1365). Services for accessing these data are described at the back of the journal.

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