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(*OC*-6-14)-(7-Azaindole)dichloro(ethoxo)-oxo(triphenylphosphine)rhenium(V)

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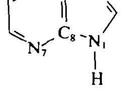
Abstract

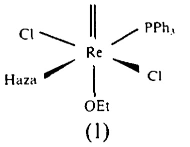
The compound consists of $[ReCl_2O(C_2H_5O)(C_7H_6N_2)\{P(C_6H_5)_3\}]$ monomers in which the Re^V centre has slightly distorted octahedral coordination. The azaindole (1*H*-pyrrolo[2,3-*b*]pyridine) and the phosphine ligands occupy *trans* positions, whereas the perpendicular $ReCl_2O(OR)$ plane contains a *trans* O=Re—O*R* unit. Azaindole forms a bifurcated intramolecular hydrogen bond with the oxo group and a Cl atom.

Comment

Several types of Re^V complexes with 7-azaindole (Haza) have been prepared recently (Lebuis & Beauchamp, 1993*b*). The structure of the title compound (1) was determined to ascertain the stereochemistry proposed from NMR data. The Haza and $P(C_6H_5)_3$ ligands are found to occupy *trans* positions in the slightly distorted octahedron (Fig. 1), whereas the ethoxo ligand is *trans* to the oxo group in the $ReCl_2O(C_2H_5O)$ plane, as is generally the case.



Haza



(1)

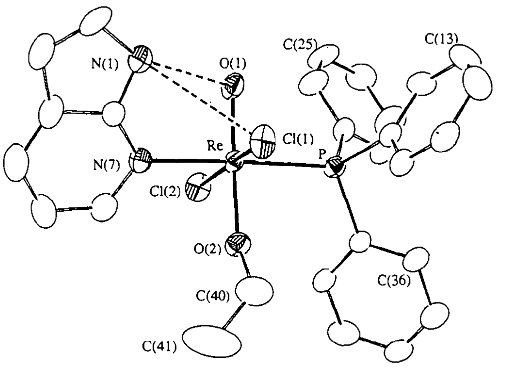


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title molecule. The atoms in the phenyl rings are assigned C_{ij} symbols, where *i* represents the ring number and *j* the position in the ring. Ellipsoids correspond to 50% probability and H atoms are omitted for simplicity. Dashed lines represent hydrogen bonds.

Few structures are known for Re^V–oxo compounds with an N atom donor *trans* to a phosphine. The $[ReOBr_2L\{P(C_6H_5)_3\}]$ complex, where *L* = *N*-phenylsalicylideneiminato, exists as two polymorphs [form (I) (Bertolasi, Sacerdoti, Gilli & Mazzi, 1982) and form (II) (Sacerdoti, Bertolasi, Gilli & Duatti, 1984)]. Our Re—P distance [2.463 (2) Å] is close to those found in both forms of the compound [2.465 (2) for (I), 2.482 (3) Å for (II)]. This is definitely short when compared to those of bis(triphenylphosphine)-Re^V complexes [2.52–2.55 Å (Lebuis & Beauchamp, 1993*a*; Lebuis, Roux & Beauchamp, 1993; Ciani, D'Alfonso, Romiti, Sironi & Freni, 1983; Graziani, Casellato, Rossi & Marchi, 1985)]. On the other hand, the Re—N distance [2.185 (7) Å] is significantly longer than those found for $[ReCl_2O(C_2H_5O)L_2]$ with *L* = py [2.144 (7) Å (Lock & Turner, 1977)] and *L* = Haza [2.136 (8) Å (Lebuis & Beauchamp, 1993*b*)]. The Re—Cl distances are unequal [Re—Cl(1) = 2.427 (2), Re—Cl(2) = 2.385 (2) Å], but within the range (2.37–2.44 Å) observed for a series of Re^V–mono-oxo complexes (Ehrlich & Owston, 1963; Sergienko & Porai-Koshits, 1982; Lebuis & Beauchamp, 1993*a*). As for the Re—O(1) distance [1.691 (6) Å], it corresponds to the mean value reported by Mayer (1988) for a large sample of Re^V–mono-oxo compounds. The Re—O(2) bond [1.885 (6) Å] is also normal and the Re—O(2)—C(40) angle [148.8 (6)°] is in the range found for $[ReX_2O(C_2H_5O)L_2]$ complexes (Ciani *et al.*, 1983; Lebuis & Beauchamp, 1993*a*,*b*; Graziani *et al.*, 1985; Lock & Turner, 1977). The terminal atom C(41) in the ethyl group has large thermal parameters, but no resolved disorder was detected.

In molecules of this type (Nugent & Mayer, 1988), the Re=O unit is found to repel the adjacent bonds, making the *cis* angles > 90°. This is observed here only for O(1)—Re—Cl(2) [95.5 (2)°]. A more impor-

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tant steric effect seems to result from the conformation of the ethoxy and phosphine ligands. Indeed, the Re—Cl(1) bond is nearly eclipsed by both O(2)—C(40) and P—C(11) [torsion angles of −10.6 (6) and −17.3 (4)°, respectively], so that Cl(1) is displaced towards the Haza ligand [P—Re—Cl(1) = 94.30 (8), N(7)—Re—Cl(1) = 85.8 (2)°]. Cl(1) makes a short contact (2.76 Å) to H(40*A*) of the ethoxo group and the orientation of phenyl ring 1 brings the *ortho* atom C(16) close to Cl(1) (3.17 Å). Similar short contacts were noted in $[ReBr_2O(C_2H_5O)\{P(C_6H_5)_3\}_2]$ (Lebuis *et al.*, 1993), $[ReOCl_2(acac)\{P(C_6H_5)_3\}]$ (Lock & Wan, 1975) and the monoclinic polymorph of $[ReCl_3O\{P(C_6H_5)_3\}_2]$ (Lebuis & Beauchamp, 1993). These various effects result in a bend in the Cl(1)—Re—Cl(2) unit [172.3 (1)°] and probably explain the discrepancy between our Re—Cl distances, since the Re—Cl(2) bond is oriented between phenyl rings 2 and 3 and avoids such short contacts. The orientation of ring 2 leads to another short intramolecular contact of 2.32 Å between H(26) and O(1), which lies almost exactly along the C(26)—H(26) direction.

Details of the geometry of the coordinated phosphine are provided in the supplementary material. The triphenylphosphine ligand adopts a conformation close to the classic 60/60/40° pattern of Re—P—C—C torsion angles (Horrocks & Greenberg, 1971). The P—C bond lengths are normal (mean 1.821 Å, Table 2). The phenyl rings are planar, the mean and maximum atom-to-plane distances being 0.006 and 0.014 Å ($\sigma \simeq 0.014$ Å), respectively. The P atom does not lie in the plane of any ring: the distances from the three ring planes are 0.065 (15), 0.076 (16) and 0.177 (15) Å, respectively. As usual, coordination makes the C—P—C angles (mean 104.6°) slightly greater than those of the uncoordinated phosphine (mean 102.6°) (Daly, 1964; Jones, 1980; Fenske, Basoglu, Hachgenei & Rogel, 1984; Albinati, Anklin, Ganazzoli, Ruegg & Pregosin, 1987).

Coordination does not appreciably affect the geometry of Haza (Dufour, Dartiguenave, Dartiguenave, Dufour, Lebuis, Bélanger-Gariépy & Beauchamp, 1990). The ligand is planar to within 0.8σ (0.012 Å). Haza makes a bifurcated intramolecular hydrogen bond *via* N(1)—H(1) to O(1) and Cl(1) (Table 3) as in the bis(azaindole) complex $[ReCl_2O(C_2H_5O)(Haza)_2]$ (Lebuis & Beauchamp, 1993*b*). The plane of the Haza ligand is roughly a bisector of the O(1)—Re—Cl(1) angle [O(1)—Re—N(7)—C(8) = −34.8 (6)°].

Molecules in the unit cell interact *via* normal contacts (Fig. S1, supplementary material), including a stacking interaction between azaindole rings of adjacent molecules along the *bc* diagonal (mean ring separation = 3.54 Å).

Experimental

Crystal data

$[ReCl_2O(C_2H_5O)(C_7H_6N_2)(C_{18}H_{15}P)]$
M_r = 698.59
Triclinic
$P\bar{1}$
a = 10.075 (2) Å
b = 10.170 (3) Å
c = 15.015 (4) Å
α = 71.27 (3)°
β = 74.01 (2)°
γ = 64.28 (2)°
V = 1295.8 (6) Å^3
Z = 2

D_x = 1.790 Mg m^{-3}
Mo *K*α radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 20–21°
μ = 5.040 mm^{-1}
T = 295 K
Elongated prism
0.32 × 0.14 × 0.10 mm
Blue

Data collection

Enraf-Nonius diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical
T_{min} = 0.48, T_{max} = 0.64
9087 measured reflections
4545 independent reflections
3485 observed reflections [$I \geq 3\sigma(I)$]

R_{int} = 0.034
θ_{max} = 25°
h = −10 → 11
k = 0 → 12
l = −17 → 17
7 standard reflections
frequency: 60 min
intensity variation: 1%

Refinement

Refinement on *F*
R = 0.038
wR = 0.039
S = 1.30
3485 reflections
337 parameters
Only H-atom *U*'s refined
$w = 1/[\sigma^2(F) + 0.0002F^2]$

$(\Delta/\sigma)_{max}$ = 0.231
$\Delta\rho_{max}$ = 1.68 e Å^{-3}
$\Delta\rho_{min}$ = −1.09 e Å^{-3}
Extinction correction: none
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)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Re	0.32754 (4)	0.40237 (4)	0.27570 (3)	2.59 (2)
Cl(1)	0.5742 (3)	0.2481 (3)	0.31832 (18)	4.11 (14)
Cl(2)	0.0741 (3)	0.5539 (3)	0.25598 (18)	4.01 (13)
P	0.3523 (3)	0.2565 (3)	0.16487 (16)	2.72 (12)
O(1)	0.4008 (7)	0.5154 (7)	0.1866 (4)	3.7 (4)
O(2)	0.2658 (7)	0.2642 (6)	0.3774 (4)	3.5 (3)
N(1)	0.5204 (8)	0.5849 (9)	0.3147 (5)	3.9 (4)
N(7)	0.3012 (8)	0.5259 (8)	0.3787 (5)	3.1 (4)
C(2)	0.5847 (12)	0.6614 (11)	0.3392 (8)	4.8 (6)
C(3)	0.5015 (12)	0.7132 (12)	0.4145 (8)	4.9 (6)
C(4)	0.2580 (12)	0.6839 (12)	0.5138 (8)	5.0 (6)
C(5)	0.1599 (12)	0.6216 (12)	0.5178 (7)	4.9 (7)
C(6)	0.1843 (11)	0.5463 (11)	0.4493 (7)	4.0 (6)
C(8)	0.3950 (10)	0.5878 (9)	0.3774 (6)	3.2 (5)
C(9)	0.3784 (11)	0.6688 (10)	0.4429 (7)	4.0 (5)
C(11)	0.5442 (10)	0.1383 (10)	0.1286 (6)	3.1 (5)
C(12)	0.6178 (11)	0.1604 (11)	0.0365 (7)	4.1 (6)
C(13)	0.7655 (11)	0.0675 (14)	0.0147 (8)	5.3 (7)
C(14)	0.8400 (10)	−0.0425 (13)	0.0829 (9)	5.3 (7)
C(15)	0.7673 (11)	−0.0661 (11)	0.1746 (8)	4.4 (6)
C(16)	0.6207 (10)	0.0214 (10)	0.1986 (7)	3.6 (5)
C(21)	0.2812 (10)	0.3702 (9)	0.0537 (6)	3.1 (5)
C(22)	0.2261 (13)	0.3115 (11)	0.0064 (7)	4.8 (6)
C(23)	0.1800 (14)	0.3946 (13)	−0.0797 (8)	5.9 (8)

C(24)	0.1840 (12)	0.5328 (12)	−0.1181 (7)	4.8 (6)
C(25)	0.2379 (13)	0.5915 (11)	−0.0727 (8)	5.1 (7)
C(26)	0.2868 (12)	0.5092 (11)	0.0140 (7)	4.6 (6)
C(31)	0.2548 (9)	0.1279 (9)	0.2129 (6)	2.8 (4)
C(32)	0.1104 (11)	0.1763 (10)	0.2615 (7)	3.9 (5)
C(33)	0.0281 (12)	0.0888 (13)	0.2896 (8)	5.6 (7)
C(34)	0.0903 (12)	−0.0521 (11)	0.2713 (7)	4.5 (6)
C(35)	0.2312 (11)	−0.1038 (11)	0.2266 (7)	4.2 (6)
C(36)	0.3143 (10)	−0.0128 (10)	0.1955 (7)	3.7 (5)
C(40)	0.3034 (13)	0.1292 (13)	0.4444 (8)	6.0 (7)
C(41)	0.2174 (15)	0.1328 (17)	0.5354 (11)	11.1 (10)

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

Re—O(1)	1.691 (6)	Re—O(2)	1.885 (6)
Re—Cl(1)	2.427 (2)	Re—Cl(2)	2.385 (2)
Re—P	2.463 (2)	Re—N(7)	2.185 (7)
P—C(11)	1.817 (9)	P—C(21)	1.826 (9)
P—C(31)	1.821 (8)	O(2)—C(40)	1.383 (12)
C(40)—C(41)	1.40 (2)		
Cl(1)—Re—P	94.30 (8)	Cl(1)—Re—O(1)	89.5 (2)
Cl(1)—Re—O(2)	84.7 (2)	Cl(1)—Re—N(7)	85.8 (2)
Cl(2)—Re—P	91.49 (8)	Cl(2)—Re—O(1)	95.5 (2)
Cl(2)—Re—O(2)	90.3 (2)	Cl(2)—Re—N(7)	88.2 (2)
P—Re—O(1)	90.7 (2)	P—Re—O(2)	89.4 (2)
O(1)—Re—N(7)	91.7 (3)	O(2)—Re—N(7)	88.3 (3)
Cl(1)—Re—Cl(2)	172.30 (9)	P—Re—N(7)	177.6 (2)
O(1)—Re—O(2)	174.2 (3)	Re—O(2)—C(40)	148.8 (6)
O(2)—C(40)—C(41)	115.3 (10)	C(11)—P—C(21)	105.1 (4)
Re—P—C(11)	113.4 (3)	C(11)—P—C(31)	104.4 (4)
Re—P—C(21)	114.4 (3)	C(21)—P—C(31)	104.3 (4)
Re—P—C(31)	114.2 (3)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	H···*A*	*D*···*A*	*D*—H···*A*
N(1)	H(1)	O(1)	2.305	2.922 (9)	120.5
N(1)	H(1)	Cl(1)	2.737	3.212 (8)	111.3

The intensities were corrected for Lp effects. No symmetry higher than triclinic was indicated by axial photographs and Niggli matrix. The structure was solved in space group $P\bar{1}$ by direct methods (Sheldrick, 1985). The *NRCVAX* package (Gabe, Le Page, Charland, Lee & White, 1989) was used for all other calculations. The non-H atoms were refined anisotropically by full-matrix least-squares methods. H-atom positions were fixed at idealized geometries (C—H = 0.95 Å). Isotropic temperature factors were refined for ring H atoms, but were fixed at $U_H = (U_C + 0.01)$ Å^2 for those on the ethyl group.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, and bond distances and angles of the phosphine and 7-azaindole ligands, as well as a stereoview of the unit cell, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71769 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1050]

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Tris(1,10-phenanthroline)iron(II) Diperchlorate Hemihydrate

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Abstract

Crystals of $[Fe(phen)_3](ClO_4)_2.0.5H_2O$ (phen = 1,10-phenanthroline) are nearly isostructural with those of the Co^{II} and Cu^{II} analogues, with similar disorder of the perchlorate groups. The Fe^{II} complex ion is less distorted than the Co^{II} and Cu^{II} complex ions. The average Fe—N distance is 1.978 (3) Å and the average N—Fe—N bidentate angle is 82.6 (4)°. The phenanthroline ligands are planar.

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