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$[Ir(dppm)_2(O_2)](C_4N_3)$

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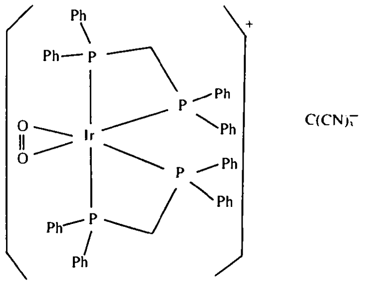
Abstract

The compound bis[bis(diphenylphosphino)methane]-peroxoiridium(I) tricyanomethanide crystallized in the triclinic system. There are two independent molecules per asymmetic unit. The geometry of the uncoordinated tricyanomethanide (TCM) is planar. The average C—C distance is 1.38 (4) Å and the average C—N distance is 1.16 (5) Å in the TCM group. The coordination about the Ir centre is distorted trigonal bipyramidal (TBP). The Ir—P distances range from 2.307 (4) to 2.343 (5) Å and the Ir—O distances range from 2.00 (1) to 2.07 (1) Å. The O—O distances are 1.39 (2) and 1.43 (1) Å.

Comment

The tricyanomethanide anion [C(CN)$_3^-$, TCM] displays various bonding modes with transition-metal ions. It can form a polymeric structure (Konnert & Britton, 1966; Biondi, Bonamico, Torelli & Vaciage, 1965; Chow & Britton, 1975; Summerville, Cohen, Hatano & Scheidt, 1978; Britton & Chow, 1983), coordinate to transition metals through its N atom (Wang, Shih, Chen, Wang, Fronczek & Watkins, 1993), form weak interactions with metal ions (Wang & Wang, 1993) or can serve as an uncoordinated counter ion (Baukova *et al.*, 1989). The title compound, Ir(dppm)$_2$(O)$_2$(TCM) (I), is the second example of the counter-ion type TCM–transition metal complex.



(I)

There are two independent molecules per asymmetric unit with almost identical molecular geometry. The geometry of uncoordinated TCM is, as expected, planar. The largest deviation from the least-squares plane formed by the four C atoms and three N atoms in TCM is 0.07 Å. The coordination

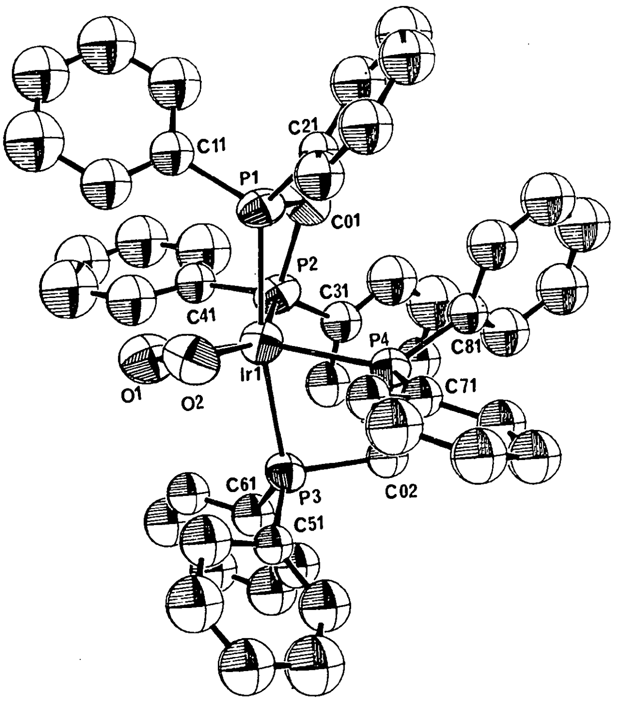


Fig. 1. *ORTEP* drawing of the title compound with 50% probability ellipsoids for non-H atoms.

about the Ir centre is distorted TBP with two P atoms in axial positions and the other two P atoms in equatorial positions. The two O atoms coordinate to Ir through their π bond and occupy the remaining equatorial position of the trigonal bipyramid. Fig. 1 shows an *ORTEP* drawing (Johnson, 1970; B. A. Frenz & Associates, Inc., 1989) of the molecule and atomic labelling scheme.

Experimental

Crystals were obtained by dissolving the compound in CH_2Cl_2 and letting Et_2O diffuse into the solution.

Crystal data

$[Ir(C_{25}H_{22}P_2)_2(O_2)](C_4N_3)$
$M_r = 1083.07$
Triclinic
$P\bar{1}$
$a = 11.045$ (3) Å
$b = 21.031$ (3) Å
$c = 23.151$ (3) Å
$\alpha = 102.99$ (2)°
$\beta = 102.47$ (2)°
$\gamma = 103.06$ (2)°
$V = 4902.4$ (29) Å^3
$Z = 4$

$D_x = 1.467$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 22 reflections
$\theta = 9.5$–15.5°
$\mu = 2.88$ mm^{-1}
$T = 298$ K
Irregular
0.26 × 0.25 × 0.19 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical ψ scan
$T_{min} = 0.847$, $T_{max} = 1.00$
9784 measured reflections
9141 independent reflections
5126 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.028$
$\theta_{max} = 20°$
$h = 0 \rightarrow 10$
$k = -20 \rightarrow 20$
$l = -22 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity variation: −7.0%

Refinement

Refinement on F
$R = 0.057$
$wR = 0.070$
$S = 1.816$
5126 reflections
598 parameters
H-atom parameters not refined

$w = 4F_o^2/[\sigma^2(I) + 0.04F_o^2]$
$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 1.331$ e Å^{-3}
$\Delta\rho_{min} = -1.457$ e Å^{-3}
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)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Ir1	0.18182 (8)	0.17403 (4)	0.42458 (4)	4.28 (2)
Ir2	0.59348 (8)	0.32020 (4)	0.06763 (4)	4.48 (2)
P1	0.2516 (5)	0.1978 (3)	0.5322 (2)	4.6 (2)
P2	0.3523 (5)	0.2726 (3)	0.4648 (2)	4.6 (2)
P3	0.1566 (5)	0.1518 (3)	0.3180 (2)	4.0 (1)
P4	0.3045 (5)	0.1038 (3)	0.3957 (2)	3.7 (1)
P5	0.5471 (5)	0.3016 (3)	−0.0397 (3)	4.8 (2)
P6	0.6407 (5)	0.2221 (3)	0.0234 (3)	4.7 (2)
P7	0.8041 (5)	0.3895 (3)	0.0992 (2)	4.3 (1)
P8	0.6827 (5)	0.3385 (3)	0.1734 (2)	4.4 (1)
O1	0.006 (1)	0.1916 (7)	0.4196 (7)	6.4 (4)
O2	−0.006 (1)	0.1213 (7)	0.4159 (6)	5.9 (4)
O3	0.405 (1)	0.3039 (7)	0.0729 (6)	6.0 (4)
O4	0.464 (1)	0.3715 (8)	0.0779 (7)	7.5 (5)
N1*	0.183 (2)	0.481 (1)	0.244 (1)	10.1 (7)
N2*	0.305 (3)	0.695 (2)	0.346 (2)	17.2 (12)
N3*	0.062 (3)	0.539 (2)	0.408 (2)	20.1 (14)
N5*	0.483 (2)	1.005 (1)	0.254 (1)	9.7 (6)
N6*	0.191 (3)	0.818 (1)	0.154 (1)	14.5 (10)
N7*	0.199 (3)	1.006 (2)	0.107 (2)	18.2 (12)
C1*	0.192 (2)	0.524 (1)	0.287 (1)	8.4 (7)
C01	0.410 (2)	0.256 (1)	0.5397 (9)	5.1 (6)
C02*	0.305 (2)	0.1258 (9)	0.3231 (9)	4.5 (5)
C2*	0.260 (4)	0.633 (2)	0.337 (2)	16.6 (14)
C3*	0.132 (3)	0.564 (2)	0.385 (2)	13.8 (12)
C03	0.647 (2)	0.243 (1)	−0.0495 (9)	5.7 (6)
C04	0.851 (2)	0.3645 (9)	0.1709 (8)	4.3 (5)
C4*	0.199 (3)	0.576 (1)	0.336 (1)	10.2 (9)
C5*	0.398 (2)	0.972 (1)	0.213 (1)	8.6 (7)
C6*	0.228 (3)	0.879 (2)	0.157 (1)	11.3 (10)
C7*	0.201 (4)	0.958 (2)	0.114 (2)	16.3 (14)
C8*	0.290 (2)	0.943 (1)	0.166 (1)	8.2 (7)
C11*	0.175 (2)	0.245 (1)	0.5827 (9)	4.8 (5)
C12*	0.249 (2)	0.283 (1)	0.641 (1)	7.3 (6)
C13*	0.185 (2)	0.317 (1)	0.677 (1)	8.7 (7)
C14*	0.059 (2)	0.311 (1)	0.655 (1)	8.1 (7)
C15*	−0.015 (2)	0.275 (1)	0.599 (1)	9.0 (8)
C16*	0.048 (2)	0.239 (1)	0.560 (1)	6.4 (6)
C21*	0.279 (2)	0.1331 (9)	0.5685 (8)	3.6 (4)
C22*	0.189 (2)	0.071 (1)	0.5450 (9)	5.5 (5)
C23*	0.203 (2)	0.019 (1)	0.574 (1)	6.2 (6)
C24*	0.307 (2)	0.033 (1)	0.624 (1)	6.6 (6)
C25*	0.397 (2)	0.094 (1)	0.649 (1)	7.2 (6)
C26*	0.384 (2)	0.147 (1)	0.621 (1)	7.3 (6)
C31*	0.496 (2)	0.297 (1)	0.4379 (9)	5.1 (5)
C32*	0.477 (2)	0.316 (1)	0.384 (1)	6.2 (6)
C33*	0.588 (2)	0.340 (1)	0.364 (1)	6.2 (6)
C34*	0.702 (2)	0.345 (1)	0.398 (1)	6.5 (6)
C35*	0.725 (3)	0.326 (1)	0.448 (1)	9.6 (8)
C36*	0.616 (2)	0.304 (1)	0.471 (1)	7.5 (7)
C41*	0.302 (2)	0.349 (1)	0.4821 (9)	4.8 (5)
C42*	0.180 (2)	0.348 (1)	0.471 (1)	7.2 (6)
C43*	0.139 (3)	0.407 (1)	0.487 (1)	10.2 (8)
C44*	0.238 (3)	0.466 (1)	0.511 (1)	9.5 (8)
C45*	0.363 (2)	0.471 (1)	0.528 (1)	8.9 (8)
C46*	0.400 (2)	0.408 (1)	0.512 (1)	9.4 (8)
C51*	0.024 (2)	0.0813 (9)	0.2654 (8)	3.8 (4)
C52*	0.037 (2)	0.038 (1)	0.216 (1)	6.4 (6)
C53*	−0.066 (2)	−0.015 (1)	0.177 (1)	8.2 (7)
C54*	−0.186 (2)	−0.025 (1)	0.185 (1)	7.0 (6)
C55*	−0.197 (2)	0.017 (1)	0.234 (1)	7.2 (6)
C56*	−0.096 (2)	0.073 (1)	0.276 (1)	6.3 (6)
C61*	0.171 (2)	0.220 (1)	0.2813 (9)	5.08 (5)
C62*	0.228 (2)	0.215 (1)	0.233 (1)	5.9 (5)
C63*	0.244 (2)	0.270 (1)	0.207 (1)	7.5 (6)
C64*	0.205 (2)	0.322 (1)	0.228 (1)	6.8 (6)
C65*	0.146 (2)	0.328 (1)	0.276 (1)	7.3 (6)
C66*	0.126 (2)	0.272 (1)	0.3026 (9)	5.1 (5)
C71*	0.222 (2)	0.0140 (9)	0.3724 (8)	3.8 (4)
C72*	0.282 (2)	−0.031 (1)	0.3472 (9)	5.3 (5)
C73*	0.221 (2)	−0.102 (1)	0.323 (1)	6.0 (6)
C74*	0.099 (2)	−0.123 (1)	0.330 (1)	6.2 (6)
C75*	0.037 (2)	−0.079 (1)	0.356 (1)	6.5 (6)
C76*	0.101 (2)	−0.0099 (9)	0.3762 (9)	4.3 (4)
C81*	0.466 (2)	0.1112 (9)	0.4332 (8)	4.2 (4)
C82*	0.490 (2)	0.087 (1)	0.486 (1)	6.4 (6)
C83*	0.615 (2)	0.095 (1)	0.520 (1)	7.3 (6)
C84*	0.713 (2)	0.120 (1)	0.499 (1)	8.0 (7)
C85*	0.697 (2)	0.142 (1)	0.448 (1)	7.3 (6)
C86*	0.573 (2)	0.139 (1)	0.416 (1)	6.5 (6)
C91*	0.393 (2)	0.259 (1)	−0.0886 (9)	4.6 (5)
C92*	0.285 (2)	0.255 (1)	−0.069 (1)	6.1 (6)
C93*	0.158 (2)	0.222 (1)	−0.108 (1)	8.5 (7)
C94*	0.142 (2)	0.193 (1)	−0.172 (1)	7.1 (6)
C95*	0.244 (2)	0.194 (1)	−0.193 (1)	9.1 (8)

C96*	0.368 (2)	0.229 (1)	−0.152 (1)	7.9 (7)
C101*	0.601 (2)	0.370 (1)	−0.0724 (9)	4.8 (5)
C102*	0.588 (2)	0.432 (1)	−0.0477 (9)	5.4 (5)
C103*	0.626 (2)	0.488 (1)	−0.072 (1)	8.1 (7)
C104*	0.674 (2)	0.474 (1)	−0.121 (1)	8.2 (7)
C105*	0.690 (2)	0.415 (1)	−0.147 (1)	8.1 (7)
C106*	0.655 (2)	0.359 (1)	−0.121 (1)	7.5 (7)
C111*	0.781 (2)	0.194 (1)	0.0512 (9)	4.8 (5)
C112*	0.796 (2)	0.176 (1)	0.105 (1)	6.2 (6)
C113*	0.900 (2)	0.149 (1)	0.123 (1)	6.8 (6)
C114*	0.981 (2)	0.142 (1)	0.090 (1)	6.8 (6)
C115*	0.969 (2)	0.160 (1)	0.036 (1)	8.9 (8)
C116*	0.868 (2)	0.186 (1)	0.016 (1)	7.0 (6)
C121*	0.509 (2)	0.144 (1)	−0.0003 (9)	5.6 (5)
C122*	0.402 (2)	0.144 (1)	0.019 (1)	6.1 (6)
C123*	0.298 (2)	0.082 (1)	0.004 (1)	8.8 (8)
C124*	0.315 (2)	0.027 (1)	−0.033 (1)	8.7 (8)
C125*	0.420 (2)	0.026 (1)	−0.051 (1)	8.8 (7)
C126*	0.517 (2)	0.086 (1)	−0.038 (1)	7.2 (6)
C131*	0.928 (2)	0.3823 (9)	0.0591 (9)	4.4 (5)
C132*	0.924 (2)	0.412 (1)	0.010 (1)	6.1 (6)
C133*	1.020 (2)	0.401 (1)	−0.021 (1)	7.4 (6)
C134*	1.103 (2)	0.370 (1)	−0.006 (1)	8.3 (7)
C135*	1.116 (2)	0.344 (1)	0.047 (1)	9.4 (8)
C136*	1.019 (2)	0.349 (1)	0.076 (1)	6.3 (6)
C141*	0.824 (2)	0.4804 (9)	0.1254 (8)	3.5 (4)
C142*	0.947 (2)	0.523 (1)	0.1523 (9)	5.5 (5)
C143*	0.967 (2)	0.593 (1)	0.176 (1)	6.1 (6)
C144*	0.864 (2)	0.617 (1)	0.169 (1)	6.8 (6)
C145*	0.744 (2)	0.576 (1)	0.144 (1)	6.9 (6)
C146*	0.721 (2)	0.506 (1)	0.1216 (9)	4.9 (5)
C151*	0.667 (2)	0.269 (1)	0.2100 (9)	5.0 (5)
C152*	0.551 (2)	0.217 (1)	0.187 (1)	6.8 (6)
C153*	0.547 (2)	0.162 (1)	0.216 (1)	7.2 (6)
C154*	0.647 (2)	0.165 (1)	0.260 (1)	6.6 (6)
C155*	0.759 (2)	0.213 (1)	0.285 (1)	7.4 (6)
C156*	0.768 (2)	0.270 (1)	0.256 (1)	6.0 (6)
C161*	0.662 (2)	0.4070 (9)	0.2276 (8)	4.1 (4)
C162*	0.544 (2)	0.416 (1)	0.221 (1)	6.3 (6)
C163*	0.519 (2)	0.467 (1)	0.261 (1)	7.6 (7)
C164*	0.616 (2)	0.511 (1)	0.310 (1)	7.6 (7)
C165*	0.738 (2)	0.503 (1)	0.320 (1)	8.0 (7)
C166*	0.758 (2)	0.450 (1)	0.2762 (9)	5.4 (5)

* Refined isotropically.

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

Ir1—P1	2.343 (5)	P7—C04	1.86 (1)
Ir1—P2	2.308 (4)	P7—C131	1.82 (2)
Ir1—P3	2.347 (4)	P7—C141	1.82 (1)
Ir1—P4	2.307 (4)	P8—C04	1.84 (1)
Ir1—O1	2.04 (1)	P8—C151	1.85 (2)
Ir1—O2	2.06 (1)	P8—C161	1.78 (2)
Ir2—P5	2.348 (5)	O1—O2	1.43 (1)
Ir2—P6	2.313 (5)	O3—O4	1.39 (2)
Ir2—P7	2.308 (5)	N1—C1	1.14 (2)
Ir2—P8	2.341 (5)	N2—C2	1.23 (4)
Ir2—O3	2.07 (1)	N3—C3	1.13 (4)
Ir2—O4	2.00 (1)	N5—C5	1.14 (2)
P1—C01	1.85 (1)	N6—C6	1.25 (3)
P1—C11	1.83 (2)	N7—C7	1.07 (5)
P1—C21	1.80 (2)	C1—C4	1.36 (3)
P2—C01	1.85 (2)	P3—C61	1.82 (2)
P2—C31	1.84 (2)	P4—C02	1.84 (2)
P2—C41	1.81 (2)	P4—C71	1.80 (1)
P3—C02	1.83 (1)	P4—C81	1.76 (1)
P3—C51	1.81 (1)	P5—C03	1.85 (2)
P5—C91	1.73 (2)	C2—C4	1.23 (4)
P5—C101	1.81 (2)	C3—C4	1.52 (4)
P6—C03	1.85 (2)	C5—C8	1.34 (3)
P6—C111	1.81 (2)	C6—C8	1.30 (3)
P6—C121	1.81 (2)	C7—C8	1.53 (4)
P1—Ir1—P2	70.6 (2)	Ir2—P5—C91	121.8 (6)
P1—Ir1—P3	168.2 (2)	Ir2—P5—C101	121.3 (6)
P1—Ir1—P4	100.4 (2)	C03—P5—C91	107.2 (8)
P1—Ir1—O1	96.9 (3)	C03—P5—C101	108.6 (8)
P1—Ir1—O2	95.2 (3)	C91—P5—C101	102.5 (8)
P2—Ir1—P3	102.3 (2)	Ir2—P6—C03	95.1 (6)
P2—Ir1—P4	95.6 (2)	Ir2—P6—C111	127.8 (6)
P2—Ir1—O1	112.9 (4)	Ir2—P6—C121	115.6 (6)
P2—Ir1—O2	150.1 (4)	C03—P6—C111	109.2 (8)
P3—Ir1—P4	70.5 (1)	C03—P6—C121	103.9 (8)
P3—Ir1—O1	94.5 (3)	C02—P3—C51	107.7 (7)
P3—Ir1—O2	95.3 (3)	C02—P3—C61	107.1 (7)
P4—Ir1—O1	150.3 (4)	C51—P3—C61	106.2 (7)
P4—Ir1—O2	113.0 (4)	Ir1—P4—C02	94.2 (5)
O1—Ir1—O2	41.0 (4)	Ir1—P4—C71	115.0 (5)
P5—Ir2—P6	70.5 (2)	C04—P8—C151	107.4 (7)
P5—Ir2—P7	100.6 (2)	C04—P8—C161	106.6 (7)
P5—Ir2—P8	168.2 (2)	C151—P8—C161	104.1 (7)
P5—Ir2—O3	97.2 (3)	Ir1—O1—O2	70.4 (7)
P5—Ir2—O4	94.9 (4)	Ir1—O2—O1	68.6 (6)
P7—Ir2—O3	149.4 (4)	Ir2—O3—O4	67.3 (8)
P7—Ir2—O4	113.2 (4)	Ir2—O4—O3	72.6 (8)
P8—Ir2—O3	94.4 (3)	N1—C1—C4	177 (3)
P8—Ir2—O4	95.4 (4)	P1—C01—P2	93.2 (7)
O3—Ir2—O4	40.1 (4)	P3—C02—P4	94.0 (7)
Ir1—P1—C01	94.1 (5)	N2—C2—C4	167 (5)
Ir1—P1—C11	121.7 (6)	N3—C3—C4	160 (4)
Ir1—P1—C21	121.6 (5)	P5—C03—P6	93.5 (8)
C01—P1—C11	106.7 (8)	P7—C04—P8	92.2 (7)
C01—P1—C21	108.5 (7)	C1—C4—C2	116 (3)
C11—P1—C21	102.6 (7)	C111—P6—C121	102.6 (8)
Ir1—P2—C01	95.2 (5)	Ir2—P7—C04	95.2 (5)
Ir1—P2—C31	128.5 (6)	Ir2—P7—C131	126.2 (5)
Ir1—P2—C41	113.5 (6)	Ir2—P7—C141	115.7 (5)
C01—P2—C31	106.4 (7)	C04—P7—C131	107.3 (7)
C01—P2—C41	106.0 (8)	C04—P7—C141	105.0 (7)
C31—P2—C41	104.6 (8)	C131—P7—C141	104.8 (7)
Ir1—P3—C02	93.2 (5)	Ir2—P8—C04	94.6 (5)
Ir1—P3—C51	119.0 (5)	Ir2—P8—C151	122.5 (6)
Ir1—P3—C61	121.4 (6)	Ir2—P8—C161	119.5 (5)
P6—Ir2—P7	95.6 (2)	C1—C4—C3	122 (3)
P6—Ir2—P8	102.9 (2)	C2—C4—C3	122 (3)
P6—Ir2—O3	113.8 (4)	N5—C5—C8	169 (3)
P6—Ir2—O4	149.9 (4)	N6—C6—C8	168 (3)
P7—Ir2—P8	69.8 (2)	N7—C7—C8	129 (4)
Ir1—P4—C81	127.3 (5)	C5—C8—C6	119 (3)
C02—P4—C71	105.1 (7)	C5—C8—C7	142 (3)
C02—P4—C81	107.1 (7)	C6—C8—C7	99 (3)
C71—P4—C81	105.2 (7)	P1—C11—C12	118 (1)
Ir2—P5—C03	93.9 (5)	P1—C11—C16	118 (1)

Computations were performed using *SDP* software (B. A. Frenz & Associates, Inc., 1989).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71717 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1055]

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

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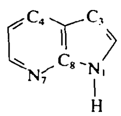
Abstract

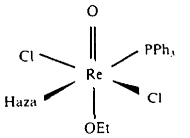
The compound consists of $[ReCl_2O(C_2H_5O)(C_7H_6N_2)\{P(C_6H_5)_3\}]$ monomers in which the Re^V centre has slightly distorted octahedral coordination. The azaindole (1*H*-pyrrolo[2,3-*b*]pyridine) and the phosphine ligands occupy *trans* positions, whereas the perpendicular $ReCl_2O(OR)$ plane contains 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)

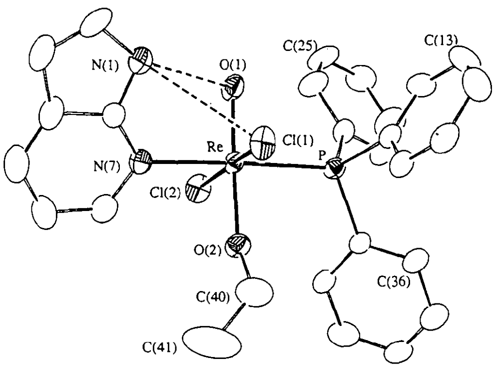


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title molecule. The atoms in the phenyl rings are assigned C_{ij} symbols, where *i* represents the ring number and *j* the position in the ring. Ellipsoids correspond to 50% probability and H atoms are omitted for simplicity. Dashed lines represent hydrogen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