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Structure of Pentacoordinated *b*-Carbonyl-*cd*-(*N*-hydroxy-*N*-nitrosobenzenaminato-*O*,*O*′)-*ae*-bis(triphenylphosphine)rhodium(I)

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Abstract. $[Rh(C_6H_5N_2O_2)(CO)\{P(C_6H_5)_3\}_2]$, M_r = 792·6, triclinic, $P\bar{1}$, a = 12·298 (9), b = 12·658 (2), c = 13·488 (3) Å, α = 90·72 (2), β = 106·38 (3), γ = 112·76 (3)°, V = 1840·1 (2) Å^3, Z = 2, D_m = 1·42 (1), D_x = 1·43 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 5·8 cm^{-1}, $F(000)$ = 812. Final R = 0·070 for 3406 observed reflections. The Rh atom has trigonal bipyramidal coordination with distortion in the trigonal plane [C—Rh—O angles of 159·6 (5) and 130·8 (5)° for amine and nitroso oxygens of the cupferron ligand respectively]. Bond distances: Rh—C = 1·77 (1), Rh—O(nitroso) = 2·339 (9), Rh—O(amine) = 2·147 (8), Rh—P = 2·323 (4) and 2·342 (4) Å.

Experimental. Yellow crystals of the title complex crystallized after 30 min from 0·6 cm^3 acetone solution containing 23 mg (0·043 mmol) $[Rh(C_6H_5N_2O_2)(CO)(PPh_3)]$ and 100 mg (0·38 mmol) PPh_3. Density determined by flotation in sodium iodide solution. Crystal size 0·09 × 0·15 × 0·2 mm, Enraf–Nonius CAD-4F diffractometer, graphite monochromator, Mo $K\alpha$ radiation, $\omega/2\theta$-scan technique, variable scan width $\Delta\omega = (0{\cdot}77 + 0{\cdot}35\tan\theta)°$, scan speed maximum 3·3° min^{-1} in ω and minimum corresponding to 50 s per reflection, unit-cell parameters from least-squares refinement of 25 reflections with $7 < \theta < 15°$, measuring range $3 < \theta < 25°$, no absorption corrections, data corrected for Lorentz and polarization effects, mean intensity of three standard reflections measured every 3600 s of X-ray exposure varied from initial value by −0·8%, $0 < h < 14$, $-15 < k < 13$, $-16 < l < 15$. 6415 unique reflections of which 3406 observed reflections with $I > 3{\cdot}0\sigma(I)$ were used for all calculations (*XRAY*72 system, Stewart, Kruger, Ammon,

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
Rh	0·0977 (1)	0·2097 (1)	0·2624 (1)	0·034 (1)
N(1)	0·1774 (9)	0·3744 (9)	0·4682 (8)	0·051 (4)
N(2)	0·1310 (9)	0·2687 (9)	0·4869 (7)	0·042 (4)
O(1)	0·0699 (10)	0·1720 (11)	0·0402 (8)	0·084 (5)
O(2)	0·0868 (7)	0·1764 (7)	0·4158 (6)	0·043 (3)
O(3)	0·1805 (8)	0·3835 (7)	0·3743 (7)	0·053 (3)
P(1)	−0·1001 (3)	0·2068 (3)	0·2215 (2)	0·039 (1)
P(2)	0·2932 (3)	0·2033 (3)	0·3074 (2)	0·038 (1)
C(1)	0·0788 (11)	0·1830 (13)	0·1278 (11)	0·058 (6)
C(11)	−0·1431 (10)	0·2357 (11)	0·3344 (9)	0·044 (4)
C(12)	−0·1378 (12)	0·3449 (11)	0·3656 (10)	0·055 (5)
C(13)	−0·1675 (14)	0·3621 (12)	0·4563 (11)	0·070 (6)
C(14)	−0·2091 (14)	0·2704 (14)	0·5105 (11)	0·075 (7)
C(15)	−0·2091 (13)	0·1651 (13)	0·4836 (11)	0·066 (6)
C(16)	−0·1797 (11)	0·1448 (12)	0·3938 (10)	0·057 (5)
C(21)	−0·2315 (11)	0·0754 (10)	0·1530 (9)	0·046 (5)
C(22)	−0·3502 (11)	0·0490 (12)	0·1601 (12)	0·063 (6)
C(23)	−0·4482 (13)	−0·0561 (14)	0·1085 (12)	0·071 (6)
C(24)	−0·4292 (14)	−0·1344 (14)	0·0509 (12)	0·074 (7)
C(25)	−0·3117 (13)	−0·1067 (14)	0·0395 (12)	0·074 (7)
C(26)	−0·2118 (13)	−0·0026 (12)	0·0921 (11)	0·061 (6)
C(31)	−0·1151 (11)	0·3195 (10)	0·1419 (8)	0·046 (5)
C(32)	−0·2167 (12)	0·2946 (13)	0·0504 (9)	0·062 (6)
C(33)	−0·2271 (16)	0·3859 (16)	−0·0061 (11)	0·080 (8)
C(34)	−0·1372 (16)	0·4982 (15)	0·0301 (12)	0·080 (7)
C(35)	−0·0347 (15)	0·5207 (13)	0·1173 (13)	0·080 (7)
C(36)	−0·0226 (12)	0·4326 (11)	0·1758 (12)	0·062 (6)
C(41)	0·1214 (10)	0·2490 (10)	0·5886 (8)	0·043 (4)
C(42)	0·0650 (13)	0·1373 (12)	0·6065 (10)	0·061 (6)
C(43)	0·0528 (14)	0·1149 (12)	0·7047 (10)	0·066 (6)
C(44)	0·0985 (13)	0·2052 (13)	0·7843 (10)	0·065 (6)
C(45)	0·1584 (14)	0·3195 (13)	0·7657 (10)	0·070 (6)
C(46)	0·1690 (12)	0·3430 (12)	0·6657 (10)	0·061 (6)
C(51)	0·3828 (10)	0·2505 (10)	0·4453 (8)	0·043 (4)
C(52)	0·4903 (12)	0·3532 (11)	0·4806 (10)	0·058 (5)
C(53)	0·5493 (13)	0·3844 (13)	0·5881 (11)	0·064 (6)
C(54)	0·5034 (13)	0·3159 (14)	0·6598 (10)	0·063 (6)
C(55)	0·3961 (14)	0·2138 (13)	0·6233 (10)	0·063 (6)
C(56)	0·3348 (10)	0·1796 (10)	0·5168 (9)	0·046 (4)
C(61)	0·2951 (10)	0·0627 (10)	0·2813 (9)	0·043 (4)
C(62)	0·2072 (12)	−0·0149 (11)	0·1989 (10)	0·054 (5)
C(63)	0·2061 (14)	−0·1240 (12)	0·1740 (13)	0·069 (6)
C(64)	0·2995 (15)	−0·1519 (12)	0·2382 (12)	0·070 (6)
C(65)	0·3911 (14)	−0·0719 (14)	0·3263 (11)	0·074 (7)
C(66)	0·3929 (12)	0·0371 (12)	0·3484 (11)	0·064 (6)
C(71)	0·3940 (11)	0·2955 (11)	0·2408 (9)	0·043 (4)
C(72)	0·3625 (11)	0·3787 (11)	0·1884 (9)	0·050 (5)
C(73)	0·4388 (14)	0·4529 (13)	0·1350 (11)	0·068 (6)
C(74)	0·5474 (13)	0·4385 (15)	0·1323 (11)	0·071 (7)
C(75)	0·5782 (13)	0·3576 (14)	0·1837 (12)	0·068 (6)
C(76)	0·5035 (12)	0·2830 (12)	0·2378 (10)	0·059 (6)

Table 2. *Bond lengths* (Å) *and angles* (°)

Rh—C(1)	1·77 (1)	N(1)—N(2)	1·30 (1)
Rh—O(2)	2·147 (8)	N(1)—O(3)	1·28 (1)
Rh—O(3)	2·339 (9)	P(1)—C(11)	1·82 (1)
Rh—P(1)	2·323 (4)	P(1)—C(21)	1·81 (1)
Rh—P(2)	2·342 (4)	P(1)—C(31)	1·83 (1)
C(1)—O(1)	1·16 (2)	P(2)—C(51)	1·83 (1)
N(2)—O(2)	1·33 (1)	P(2)—C(61)	1·82 (1)
N(2)—C(41)	1·43 (2)	P(2)—C(71)	1·80 (1)

Rh—C(1)—O(1)	176 (1)	O(2)—Rh—O(3)	69·6 (3)
Rh—O(2)—N(2)	116·3 (6)	P(1)—Rh—P(2)	176·9 (1)
Rh—O(3)—N(1)	116·0 (7)	Rh—P(1)—C(11)	114·2 (4)
O(2)—N(2)—N(1)	124 (1)	Rh—P(1)—C(21)	118·7 (5)
N(2)—N(1)—O(3)	114 (1)	Rh—P(1)—C(31)	111·7 (4)
O(2)—N(2)—C(41)	117·4 (9)	Rh—P(2)—C(51)	114·2 (5)
C(1)—Rh—O(2)	159·6 (5)	Rh—P(2)—C(61)	116·3 (4)
C(1)—Rh—O(3)	130·8 (5)	Rh—P(2)—C(71)	113·5 (5)

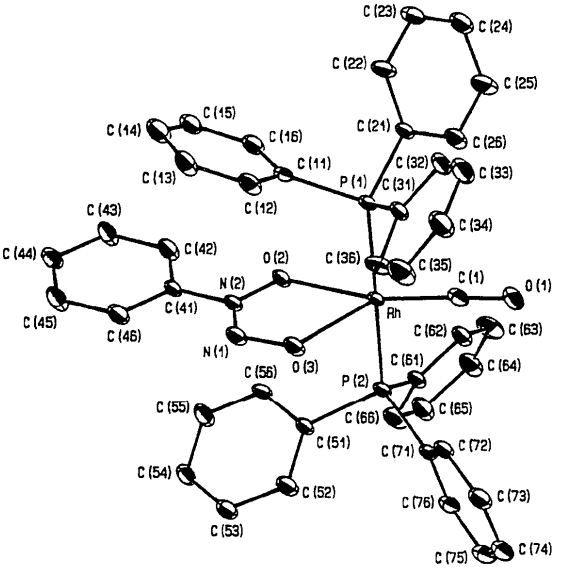

Fig. 1. Perspective view and atomic labelling of the molecule.

Dickinson & Hall, 1972). The structure was solved by the heavy-atom method and subjected to anisotropic full-matrix least-squares refinement on F (461 variables). H atoms were not placed. Neutral-atom scattering factors (Cromer & Mann, 1968) and anomalous-dispersion corrections for rhodium from *International Tables for X-ray Crystallography* (1962). Final $R = 0·070$ and $wR = 0·076$ with unit weights taken [a more stringent criterion of $I > 4·0\sigma(I)$ gave 3206 observed reflections with $R = 0·065$ and $wR = 0·072$], $(\Delta\rho)_{max} = 0·71$ and $(\Delta\rho)_{min} = -0·56$ e Å^{-3} within 1 Å from rhodium, $(\Delta/\sigma)_{max} = 0·0872$. Final atomic coordinates are given in Table 1,* main geometrical parameters in Table 2 and a perspective view (Johnson, 1976) with atomic labels in Fig. 1.

Related literature. The title compound forms part of a family of pentacoordinated complexes of general formula $[Rh(cupf)(CO)(PX_3)_2]$ (cupf = 'cupferron ion' or *N*-phenyl-*N*-nitrosohydroxylamido ligand, *X*

* A least-squares plane and deviations, lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52667 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

= phenyl, *p*-chlorophenyl, *p*-methoxyphenyl or cyclohexyl) which was recently prepared in our laboratory. A related series of 2-quinaldinate (quin) complexes, $[Rh(quin)(CO)\{P(X\text{-}C_6H_4)_3\}_2]$ (X = 4-CH_3O, 4-CH_3, 3-CH_3, 4-F and 4-Cl), crystallizing in two isomeric forms was recently reported (Heras, Cano, Lobo & Pinilla, 1989). This structure determination established the stereochemistry of the addition product between $[Rh(cupf)(CO)(PX_3)]$ and an additional phosphine molecule and will help us to elucide the mechanism of this reaction. Structural details are comparable to those of $[Rh(cupf)(CO)(PPh_3)]$ (Basson, Leipoldt, Roodt & Venter, 1986), $[Rh(cupf)(CO)(CH_3)(I)(PPh_3)]$ (Basson, Leipoldt, Roodt & Venter, 1987) and $[Rh(Cl)_2\{ONN(C_6H_4Me\text{-}p)O\}(H_2O)(PPh_3)].0{\cdot}5Me_2CO$ (Ahmed, Edwards, Jones, McCleverty, Rothin & Tate, 1988).

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cis-Chloro(1-methylcytosine-N^3)(N,N,N',N'-tetramethylethylenediamine)platinum(II) Perchlorate Hemihydrate, *cis*-$[PtCl(C_5H_7N_3O)(C_6H_{16}N_2)]ClO_4.0{\cdot}5H_2O$

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Abstract. $C_{11}H_{24}N_5O_{5\cdot5}Cl_2Pt$, $M_r = 580{\cdot}34$, monoclinic, $C2/c$, $a = 12{\cdot}329$ (3), $b = 11{\cdot}078$ (3), $c = 27{\cdot}417$ (7) Å, $\beta = 93{\cdot}26$ (2)°, $V = 3739$ (2) Å^3, $Z = 8$, $D_x = 2{\cdot}062$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7{\cdot}91$ mm^{-1}, $F(000) = 2248$, $T = 291$ (1) K, final $R = 0{\cdot}029$ for 3138 unique observed [$F \geq 3{\cdot}0\sigma(F)$] diffractometer data. The cation contains N,N,N',N'-tetramethylethylenediamine chelated to platinum, coordinated chloride and a 1-methylcytosine nucleobase bound through the N(3) position. The angles of the coordination sphere of platinum display some deviation from ideal square planarity which is caused by the bite of the ethylenediamine [N(11)—Pt(1)—N(12) 85·8 (2)°]. Pt—N and Pt—Cl distances as well as geometry of the methylcytosine ring [Pesch, Preut & Lippert (1989). *Inorg. Chim. Acta*. In the press; Orbell, Marzilli & Kistenmacher (1981). *J. Am. Chem. Soc.* **103**, 5126–5133; Britten, Lippert, Lock & Pilon (1982). *Inorg. Chem.* **21**, 1936–1941] and of the diamine ligand [Orbell, Taylor, Birch, Lawton, Vilkins & Keefe (1988). *Inorg. Chim. Acta*, **152**, 125–134] are normal. The puckering of the diamine ligands is such that C(11) and C(12) are on opposite sides of the platinum coordination plane. The 1-methylcytosine ring is almost at right angles to the plane [86·2 (1)°]. The only hydrogen bond of less than 3 Å involves H_2O protons and oxygen sites of the 1-methylcytosine in such a way that H_2O links pairs of cations.

Experimental. The title compound was prepared by reaction of $Pt(C_6H_{16}N_2)Cl_2$, obtained according to a modified Dhara method (Dhara, 1970) from K_2PtCl_4 and $C_6H_{16}N_2$ with $AgClO_4$ (1 equiv.) and 1-methylcytosine (1 equiv.) at pH 5–6, 80 h, 303 K. On slow evaporation, pale yellow crystals were isolated in 35% yield. According to ^{1}H NMR spectroscopy, the filtrate contained more of the title compound together with the bis(1-methylcytosine) complex and free ligand. Crystal size ~0·12 × 0·12 × 0·33 mm, D_m

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