Experimental

Crystal data

$[Ru(C_5H_5N)_4Cl_2]$
$M_r = 488.38$
Tetragonal
$I4_1/acd$ (origin at $\bar{1}$)
$a = 15.701$ (2) Å
$c = 16.987$ (2) Å
$V = 4187.7$ (8) Å^3
$Z = 8$
$D_x = 1.550$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–$13°$
$\mu = 1.0$ mm^{-1}
$T = 293$ K
Block
0.22 × 0.12 × 0.12 mm
Orange–red

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical
$T_{\min} = 0.810$, $T_{\max} = 0.999$
2293 measured reflections
1208 independent reflections

650 observed reflections $[F_o > 3\sigma(F_o)]$
$R_{\rm int} = 0.022$
$\theta_{\max} = 26°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity variation: ±2%

Refinement

Refinement on F
$R = 0.029$
$wR = 0.036$
$S = 1.119$
650 reflections
63 parameters
H-atom parameters not refined

$w = 4F_o^2/[\sigma^2(F_o^2) + (0.04F_o^2)^2]$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ 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)

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

	x	y	z	$B_{\rm eq}$
Ru	0	1/4	1/8	2.249 (6)
Cl1	0.10837 (5)	x+1/4	1/8	3.50 (2)
N1	0.0659 (2)	0.1833 (2)	0.2115 (1)	2.70 (5)
C1	0.0251 (2)	0.1456 (2)	0.2720 (2)	3.45 (7)
C2	0.0676 (2)	0.1013 (3)	0.3299 (2)	4.40 (8)
C3	0.1541 (3)	0.0942 (3)	0.3267 (2)	4.86 (9)
C4	0.1964 (2)	0.1316 (2)	0.2653 (2)	3.91 (8)
C5	0.1507 (2)	0.1753 (2)	0.2093 (2)	3.18 (7)

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

Ru—Cl1	2.4054 (6)	C1—C2	1.376 (5)
Ru—N1	2.079 (2)	C2—C3	1.363 (5)
N1—C1	1.347 (5)	C3—C4	1.369 (5)
N1—C5	1.337 (4)	C4—C5	1.373 (5)
Cl1—Ru—N1	90.23 (8)	C1—C2—C3	119.7 (3)
Ru—N1—C1	121.5 (2)	C2—C3—C4	118.6 (3)
Ru—N1—C5	121.5 (3)	C3—C4—C5	119.2 (3)
C1—N1—C5	116.9 (3)	N1—C5—C4	123.2 (3)
N1—C1—C2	122.5 (3)		

The space group $I4_1/acd$ was chosen from the systematic absences and led to a successful refinement. The structure was solved by the Patterson method and refined by full-matrix least squares. All calculations were performed using the *SDP* system (Enraf–Nonius, 1985) on a MicroVAX II computer.

We thank the Hong Kong Research Grant Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1092). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 1407–1409

Sodium *trans*-Dicyano[*N*,*N*'-1,2-phenylenebis(2-pyridinecarboxamido)]cobaltate(III)

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Abstract

The Co atom in the $[Co^{III}(CN)_2(C_{18}H_{12}N_4O_2)]^-$ complex anion is hexacoordinate with a distorted octahedral geometry. The four N atoms in the amide ligand occupy equatorial positions and are coplanar to within 0.007 Å. The two CN groups are coordinated to Co in a *trans* configuration. The Na cation interacts with four complex anions through their cyanide-N and amide-O atoms, thereby adopting a distorted tetrahedral coordination.

Comment

We recently reported some σ-alkylcobalt(III) complexes bearing an organic amide ligand (bpb or bpc; see scheme below) (Mak, Wong, Yam, Lai & Che, 1991). We are also interested in the structural chemistry of coordination compounds containing these amide ligands. This study indicates that the bpb ligand in the $[Co(bpb)(CN)_2]^-$ anion (I) deviates from planarity; the interplanar angles between the

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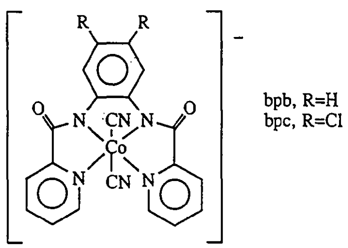

(I)

central C(1)–C(6) benzene plane and the two pyridyl rings are 10.7 (3) and 17.5 (2)°. This observation can be attributed to the close intramolecular contact that occurs between the two H atoms in the 6-positions of the pyridyl rings. The H(12)···H(13) distance of 1.99 Å is significantly less than the sum of the van der Waals radii (2.40 Å). The effect would be expected to be more pronounced if the ligand was perfectly planar.

Another interesting structural feature is the coordination of the Na cation which can be described as being tetracoordinated by two carbonyl O atoms and two cyanide N atoms from four distinct adjacent $[Co(bpb)(CN)_2]^-$ anions in a distorted tetrahedral environment.

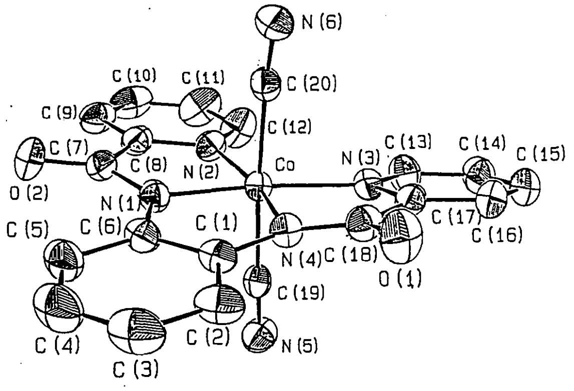

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the anion with atomic numbering scheme. 50% probability ellipsoids are displayed.

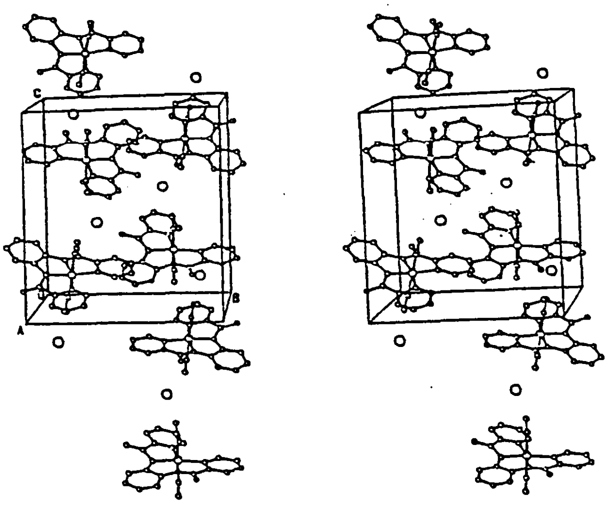

Fig. 2. Stereoview of the packing of the molecules in the unit cell.

Experimental

Crystal data

$Na[Co(CN)_2(C_{18}H_{12}N_4O_2)]$
$M_r = 450.28$
Monoclinic
$P2_1/n$
$a = 8.987\ (1)$ Å
$b = 14.090\ (3)$ Å
$c = 15.543\ (2)$ Å
$\beta = 101.67\ (1)°$
$V = 1927.8\ (8)$ Å^3
$Z = 4$
$D_x = 1.55$ Mg m^{-3}
$D_m = 1.55$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–22°$
$\mu = 0.94$ mm^{-1}
$T = 292$ K
Parallelepiped
0.22 × 0.21 × 0.14 mm
Violet

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: empirical
$T_{min} = 0.956$, $T_{max} = 0.999$
7878 measured reflections
3952 independent reflections

2995 observed reflections $[F_o > 3\sigma(F_o)]$
$R_{int} = 0.034$
$\theta_{max} = 26°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 17$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity variation: 2%

Refinement

Refinement on F
$R = 0.024$
$wR = 0.036$
$S = 0.956$
2995 reflections
272 parameters
H-atom parameters not refined

$w = 4F_o^2/[\sigma^2(F_o^2) + (0.04F_o^2)^2]$
$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ 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)

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

	x	y	z	B_{eq}
Co	−0.05782 (2)	0.23048 (2)	0.21749 (1)	2.042 (4)
Na	0.07221 (8)	0.17037 (5)	−0.07731 (5)	2.73 (1)
O(1)	−0.1471 (2)	−0.0324 (1)	0.1297 (1)	4.34 (4)
O(2)	0.3238 (2)	0.2981 (1)	0.3865 (1)	4.16 (3)
N(1)	0.1163 (2)	0.2168 (1)	0.30812 (9)	2.50 (3)
N(2)	−0.0106 (2)	0.3669 (1)	0.2353 (1)	2.63 (3)
N(3)	−0.2396 (2)	0.2134 (1)	0.12331 (9)	2.48 (3)
N(4)	−0.0620 (2)	0.0963 (1)	0.21929 (9)	2.42 (3)
N(5)	−0.2730 (2)	0.2421 (1)	0.3460 (1)	3.46 (4)
N(6)	0.1312 (2)	0.2099 (1)	0.0746 (1)	3.79 (4)
C(1)	0.0486 (2)	0.0554 (1)	0.2861 (1)	2.66 (4)
C(2)	0.0624 (2)	−0.0408 (2)	0.3067 (1)	3.57 (4)
C(3)	0.1761 (3)	−0.0694 (2)	0.3764 (2)	4.46 (5)
C(4)	0.2730 (3)	−0.0042 (2)	0.4246 (2)	4.77 (5)
C(5)	0.2605 (2)	0.0919 (2)	0.4053 (1)	3.85 (5)
C(6)	0.1472 (2)	0.1228 (1)	0.3352 (1)	2.74 (4)

C(7)	0.1989 (2)	0.2934 (2)	0.3346 (1)	2.85 (4)
C(8)	0.1202 (2)	0.3808 (1)	0.2951 (1)	2.77 (4)
C(9)	0.1734 (3)	0.4713 (2)	0.3198 (1)	3.72 (4)
C(10)	0.0916 (3)	0.5486 (2)	0.2837 (2)	4.40 (5)
C(11)	−0.0404 (3)	0.5353 (2)	0.2229 (2)	4.73 (6)
C(12)	−0.0878 (3)	0.4435 (2)	0.1999 (2)	4.04 (5)
C(13)	−0.3447 (2)	0.2754 (2)	0.0835 (1)	3.31 (4)
C(14)	−0.4655 (2)	0.2492 (2)	0.0178 (1)	3.71 (5)
C(15)	−0.4786 (2)	0.1566 (2)	−0.0093 (1)	3.71 (5)
C(16)	−0.3714 (2)	0.0914 (2)	0.0312 (1)	3.37 (4)
C(17)	−0.2562 (2)	0.1212 (1)	0.0983 (1)	2.58 (3)
C(18)	−0.1468 (2)	0.0527 (1)	0.1508 (1)	2.79 (4)
C(19)	−0.1900 (2)	0.2376 (1)	0.2999 (1)	2.41 (3)
C(20)	0.0648 (2)	0.2222 (1)	0.1289 (1)	2.56 (4)

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

Co—N(1)	1.892 (1)	N(1)—C(7)	1.328 (2)
Co—N(2)	1.975 (2)	N(2)—C(8)	1.357 (2)
Co—N(3)	1.976 (1)	N(2)—C(12)	1.340 (3)
Co—N(4)	1.891 (2)	N(3)—C(13)	1.343 (2)
Co—C(19)	1.917 (2)	N(3)—C(17)	1.355 (2)
Co—C(20)	1.934 (2)	N(4)—C(1)	1.407 (3)
O(1)—C(18)	1.243 (2)	N(4)—C(18)	1.328 (2)
O(2)—C(7)	1.245 (2)	N(5)—C(19)	1.137 (3)
N(1)—C(6)	1.401 (3)	N(6)—C(20)	1.142 (3)
N(1)—Co—N(2)	82.71 (6)	N(3)—Co—N(4)	82.71 (6)
N(1)—Co—N(3)	167.12 (7)	N(3)—Co—C(19)	88.29 (7)
N(1)—Co—N(4)	84.42 (6)	N(3)—Co—C(20)	88.26 (7)
N(1)—Co—C(19)	92.23 (7)	N(4)—Co—C(19)	91.48 (7)
N(1)—Co—C(20)	91.13 (7)	N(4)—Co—C(20)	88.06 (7)
N(2)—Co—N(3)	110.17 (7)	C(19)—Co—C(20)	176.56 (7)
N(2)—Co—N(4)	167.09 (6)	Co—C(19)—N(5)	177.4 (1)
N(2)—Co—C(19)	89.99 (7)	Co—C(20)—N(6)	174.1 (2)
N(2)—Co—C(20)	91.22 (7)		

Table 3. *Coordination environment* (Å, °) *of the sodium cation*

$Na\cdots O(1^{i})$	2.263 (2)	$Na\cdots N(5^{iii})$	2.355 (2)
$Na\cdots O(2^{ii})$	2.234 (2)	$Na\cdots N(6)$	2.379 (2)
$O(1^{i})\cdots Na\cdots O(2^{ii})$	115.8 (1)	$O(2^{ii})\cdots Na\cdots N(5^{iii})$	115.5 (1)
$O(1^{i})\cdots Na\cdots N(5^{iii})$	91.1 (1)	$O(2^{ii})\cdots Na\cdots N(6)$	102.4 (2)
$O(1^{i})\cdots Na\cdots N(6)$	122.4 (1)	$N(5^{iii})\cdots Na\cdots N(6)$	110.1 (1)

Symmetry codes: (i) $-x, -y, -z$; (ii) $x+\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$.

The structure was solved by the Patterson method and refined by full-matrix least squares. All calculations were performed using the *SDP* system (Enraf–Nonius, 1985) on a MicroVAX II computer.

We thank Dr S.-T. Mak for providing the crystals and the Hong Kong Research Grant Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: MU1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Carbonylchlorobis(tricyclohexylphosphine)iridium(I)

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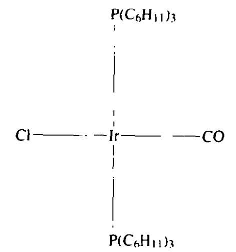
Abstract

The structure of *trans*-carbonylchlorobis(tricyclohexylphosphine)iridium(I), *trans*-$[IrCl(CO)\{P(C_6H_{11})_3\}_2]$, has been determined by single-crystal X-ray diffraction. The iridium(I) atom, situated on an inversion center, has a square-planar environment in which Ir(1)—P(1) = 2.345 (2), Ir(1)—Cl(1) = 2.398 (7), Ir(1)—CO = 1.78 (2) and C(1)—O(1) = 1.10 (2) Å.

Comment

The chemistry of complexes with the formula *trans*-$[IrCl(CO)(PR_3)_2]$ has been well studied (Dickson, 1983). Here we report the structure of the title complex, (I), which contains tricyclohexylphosphine ligands.



$$\begin{array}{c} P(C_6H_{11})_3 \\ | \\ Cl-Ir-CO \\ | \\ P(C_6H_{11})_3 \end{array}$$

(I)

The unit cell of the title complex contains one *trans*-$[IrCl(CO)\{P(C_6H_{11})_3\}_2]$ molecule, as the Ir atom is situated on the inversion center at (0, 0, 0). The chloride and carbonyl ligands are necessarily disordered such that these ligands occupy mutually *trans* sites with 50% probability. This Cl/CO disorder is also present in the related crystal structures of *trans*-$[IrCl(CO)(PPh_3)_2]$ (Churchill, Fettinger, Buttrey, Barkan & Thompson, 1988) and *trans*-$[IrCl(CO)\{P(o\text{-tolyl})_3\}_2]$ (Brady *et al.*, 1975). It should also be mentioned that *trans*-$[IrCl(CO)\{P(C_6H_{11})_3\}_2]$ is isomorphous and nearly isostructural with $[OsClH(CO)\{P(C_6H_{11})_3\}_2]$ (Moers, Noordik & Beurskens, 1981).

The structure of (I) contains no abnormal distances. The Ir—P distance is 2.345 (2) Å, slightly longer than those found in the complexes *trans*-$[IrCl(CO)(PR_3)_2]$ in which the aryl groups *R* = phenyl [2.330 (1) Å] and *R* = *o*-tolyl [2.338 (5) Å] are present.

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