

Experimental*Crystal data*[Ru(C₅H₅N)₄Cl₂] $M_r = 488.38$

Tetragonal

 $I4_1/acd$ (origin at $\bar{1}$) $a = 15.701$ (2) Å $c = 16.987$ (2) Å $V = 4187.7$ (8) Å³ $Z = 8$ $D_x = 1.550$ Mg m⁻³Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

 $\theta = 10\text{--}13^\circ$ $\mu = 1.0$ mm⁻¹ $T = 293$ K

Block

 $0.22 \times 0.12 \times 0.12$ mm

Orange-red

Data collection

Enraf-Nonius CAD-4 diffractometer

 ω -2 θ scans

Absorption correction:

empirical

 $T_{\min} = 0.810$, $T_{\max} = 0.999$

2293 measured reflections

1208 independent reflections

650 observed reflections

 $[F_o > 3\sigma(F_o)]$ $R_{\text{int}} = 0.022$ $\theta_{\max} = 26^\circ$ $h = 0 \rightarrow 19$ $k = 0 \rightarrow 19$ $l = 0 \rightarrow 20$

3 standard reflections

frequency: 120 min

intensity variation: $\pm 2\%$ *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 (Å²)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B_{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.

References

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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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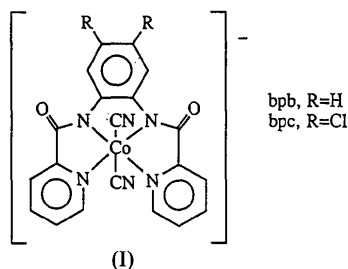
(Received 27 September 1993; accepted 16 February 1994)

Abstract

The Co atom in the [Co^{III}(CN)₂(C₁₈H₁₂N₄O₂)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



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.

Experimental

Crystal data

Na[Co(CN)₂(C₁₈H₁₂N₄O₂)]

M_r = 450.28

Monoclinic

*P*2₁/*n*

a = 8.987 (1) Å

b = 14.090 (3) Å

c = 15.543 (2) Å

β = 101.67 (1)°

V = 1927.8 (8) Å³

Z = 4

D_x = 1.55 Mg m^{−3}

D_m = 1.55 Mg m^{−3}

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 10–22°

μ = 0.94 mm^{−1}

T = 292 K

Parallelepiped

0.22 × 0.21 × 0.14 mm

Violet

Data collection

Enraf–Nonius CAD-4 diffractometer

ω–2θ scans

Absorption correction: empirical

T_{min} = 0.956, *T_{max}* = 0.999

7878 measured reflections

3952 independent reflections

2995 observed reflections

[*F_o* > 3σ(*F_o*)]

R_{int} = 0.034

θ_{max} = 26°

h = −11 → 11

k = 0 → 17

l = −18 → 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 = 4*F_o*²/[σ²(*F_o*²) + (0.04*F_o*²)²]

(Δ/σ)_{max} = 0.03

Δρ_{max} = 0.38 e Å^{−3}

Δρ_{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 (Å²)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
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)

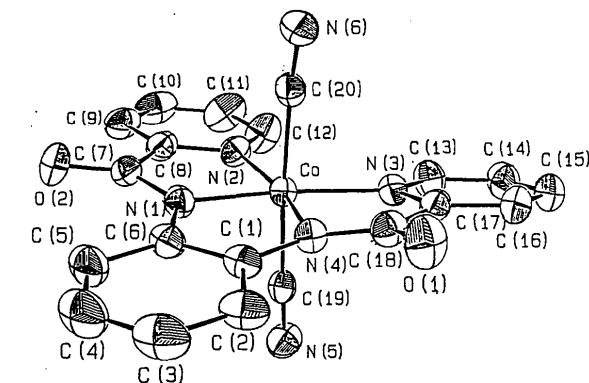


Fig. 1. ORTEP (Johnson, 1976) drawing of the anion with atomic numbering scheme. 50% probability ellipsoids are displayed.

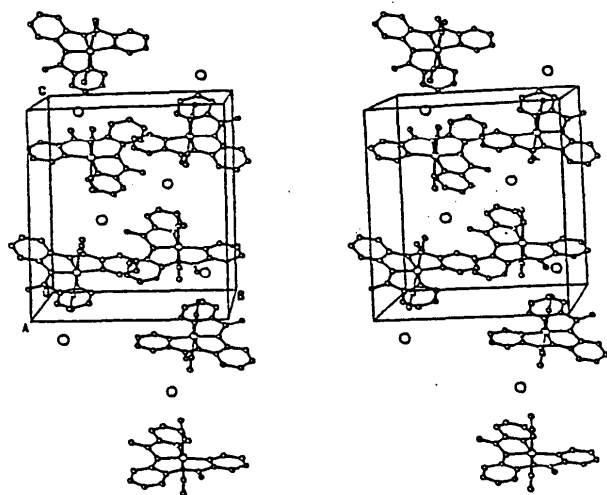


Fig. 2. Stereoview of the packing of the molecules in the unit cell.

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)

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trans-Carbonylchlorobis(tricyclohexylphosphine)iridium(I)

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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...O(1 ⁱ)	2.263 (2)	Na...N(5 ⁱⁱⁱ)	2.355 (2)
Na...O(2 ⁱⁱ)	2.234 (2)	Na...N(6)	2.379 (2)
O(1 ⁱ)...Na...O(2 ⁱⁱ)	115.8 (1)	O(2 ⁱⁱ)...Na...N(5 ⁱⁱⁱ)	115.5 (1)
O(1 ⁱ)...Na...N(5 ⁱⁱⁱ)	91.1 (1)	O(2 ⁱⁱ)...Na...N(6)	102.4 (2)
O(1 ⁱ)...Na...N(6)	122.4 (1)	N(5 ⁱⁱⁱ)...Na...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.

References

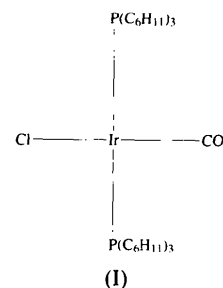
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- Johnson, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Mak, S.-T., Wong, W.-T., Yam, V. W.-W., Lai, T.-F. & Che, C.-M. (1991). *J. Chem. Soc. Dalton Trans.* pp. 1915–1922.

Abstract

The structure of *trans*-carbonylchlorobis(tricyclohexylphosphine)iridium(I), *trans*-[IrCl(CO){P(C₆H₁₁)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)(PR₃)₂] has been well studied (Dickson, 1983). Here we report the structure of the title complex, (I), which contains tricyclohexylphosphine ligands.



The unit cell of the title complex contains one *trans*-[IrCl(CO){P(C₆H₁₁)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)(PPh₃)₂] (Churchill, Fetting, Buttrey, Barkan & Thompson, 1988) and *trans*-[IrCl(CO){P(*o*-tolyl)₃}₂] (Brady *et al.*, 1975). It should also be mentioned that *trans*-[IrCl(CO){P(C₆H₁₁)₃}₂] is isomorphous and nearly isostructural with [OsClH(CO){P(C₆H₁₁)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)(PR₃)₂] in which the aryl groups R = phenyl [2.330 (1) Å] and R = *o*-tolyl [2.338 (5) Å] are present.