

Table 2. Bond distances (Å) and angles (°)

Cu—N(1)	1.991 (7)	N(2)—C(5)	1.499 (10)
Cu—N(3)	1.991 (9)	C(3)—N(3)	1.260 (11)
Cu—O(1)	1.972 (7)	N(3)—C(4)	1.498 (12)
Cu—O(2)	2.531 (8)	C(4)—C(18)	1.519 (13)
Cu—O(3)	1.960 (6)	C(5)—C(12)	1.503 (12)
Cu—O(4)	2.483 (7)	C(25)—C(24)	1.532 (18)
C(1)—N(1)	1.482 (13)	C(24)—O(1)	1.294 (15)
C(1)—C(6)	1.503 (14)	C(24)—O(2)	1.218 (15)
N(1)—C(2)	1.262 (12)	C(27)—C(26)	1.505 (13)
C(2)—N(2)	1.361 (15)	C(26)—O(3)	1.291 (14)
N(2)—C(3)	1.371 (14)	C(26)—O(4)	1.230 (12)
N(1)—Cu—N(3)	90.1 (3)	C(12)—C(5)—N(2)	112.7 (8)
N(1)—Cu—O(1)	93.5 (3)	N(2)—C(3)—N(3)	126.0 (10)
N(3)—Cu—O(3)	91.9 (3)	C(3)—N(3)—C(4)	115.4 (9)
O(1)—Cu—O(3)	91.7 (3)	N(3)—C(4)—C(18)	110.9 (7)
C(6)—C(1)—N(1)	113.2 (7)	C(25)—C(24)—O(1)	113.6 (10)
C(1)—N(1)—C(2)	114.8 (8)	C(25)—C(24)—O(2)	120.8 (12)
N(1)—C(2)—N(2)	124.9 (9)	O(1)—C(24)—O(2)	125.5 (13)
C(2)—N(2)—C(5)	116.0 (8)	C(27)—C(26)—O(3)	117.1 (9)
C(2)—N(2)—C(3)	126.3 (7)	C(27)—C(26)—O(4)	121.6 (10)
C(5)—N(2)—C(3)	117.2 (9)	O(3)—C(26)—O(4)	121.2 (10)

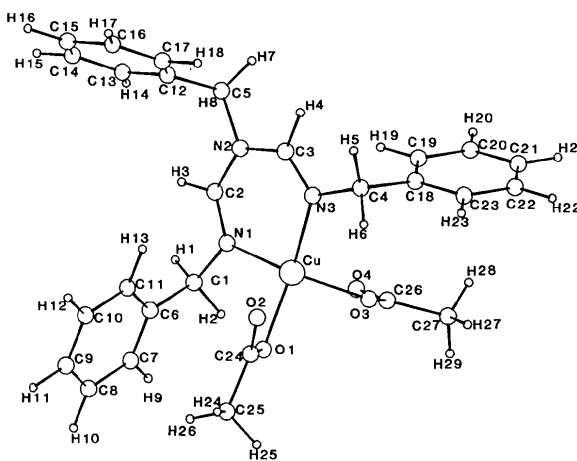


Fig. 1. Molecule of title compound with atom labelling.

Atomic scattering factors: for Cu, Cromer & Mann (1968), other atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic parameters in Table 1, bond distances and angles in Table 2, the molecule with atomic labelling is shown in Fig. 1.

Related literature. No other complexes of copper(II) acetate with a bidentate N-donor ligand have been reported. Some square planar Cu^{II} complexes in which the four coordination involves a bidentate N-donor ligand and two O atoms from other groups have been reported. Amongst these are the following: dinitrato(*N,N,N',N'*-tetramethylethylenediamine)-copper(II) (Pavkovic, Miller & Brown, 1977); bis(*N*-acetyl-glycinato)(1,10-phenanthroline)copper(II) (Battaglia, Bonamartini, Corradi & Marcotrigiano,

1977); bis(nitrito)(2,2'-bipyridyl)copper(II) (Stephens, 1969); (2,2'-bipyridyl)bis(*N*-acetyl-*dl*-leucinato-*O*)copper(II) (Antolini, Menabue, Saladini, Battaglia & Corradi, 1984).

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Structure of Di(quinoxalinium) Tetrachlorocobaltate(II) Monohydrate

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Abstract. (C₈H₇N₂)₂[CoCl₄].H₂O, *M*_r = 481.06, triclinic, *P* $\bar{1}$, *a* = 7.439 (2), *b* = 7.561 (2), *c* = 20.104 (5) Å, α = 94.11 (2), β = 90.95 (2), γ = 117.57 (2)°, *V* = 998.3 (5) Å³, *Z* = 2, *D*_m = 1.604 (8), *D*_x = 1.60 g cm⁻³, λ (Mo *K*α) = 0.71069 Å, μ = 14.3 cm⁻¹, *F*(000) = 486, *T* = 296 K, 4587 unique

reflections measured, final *R* = 0.067 over 3054 reflections having *F*_o > 6σ(*F*_o). The structure consists of discrete CoCl₄²⁻ anions and two quinoxalinium cations. The tetrachlorocobaltate anion is a nearly regular tetrahedron, with average Co—Cl 2.266 (19) Å and Cl—Co—Cl 109.4 (3.5)°. Both quinoxalinium cations are planar. One of them is disordered, having two orientations with a dihedral

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Table 1. Fractional atomic coordinates and equivalent isotropic thermal factors (\AA^2) with e.s.d.'s in parentheses
$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Co(1)	0.0395 (1)	0.1286 (1)	0.2507 (1)	0.041 (1)
Cl(1)	-0.1198 (3)	0.2773 (3)	0.1959 (1)	0.053 (1)
Cl(2)	0.1705 (3)	0.3241 (3)	0.3478 (1)	0.065 (1)
Cl(3)	0.2787 (03)	0.1310 (3)	0.1825 (1)	0.061 (1)
Cl(4)	-0.1829 (4)	-0.1873 (3)	0.2685 (1)	0.089 (2)
O(1)	0.2341 (11)	0.7209 (7)	0.2852 (3)	0.096 (5)
N(1)	-0.2439 (9)	0.4606 (8)	-0.1113 (2)	0.048 (4)
N(2)	-0.4973 (10)	0.1995 (9)	-0.0256 (4)	0.065 (5)
C(1)	-0.3845 (13)	0.2775 (11)	-0.1348 (4)	0.063 (5)
C(2)	-0.5066 (12)	0.1555 (10)	-0.0884 (5)	0.071 (6)
C(3)	-0.3489 (11)	0.3858 (10)	-0.0022 (3)	0.050 (5)
C(4)	-0.3218 (13)	0.4512 (13)	0.0679 (3)	0.071 (6)
C(5)	-0.1711 (13)	0.6324 (12)	0.0877 (4)	0.062 (5)
C(6)	-0.0445 (14)	0.7620 (12)	0.0459 (4)	0.074 (6)
C(7)	-0.0646 (12)	0.7094 (10)	-0.0211 (4)	0.056 (5)
C(8)	-0.2177 (10)	0.5223 (9)	-0.0447 (3)	0.040 (4)
N(3)	0.4145 (6)	0.0478 (5)	0.3585 (2)	0.038 (6)
N(4)	0.6265 (19)	0.4106 (15)	0.4369 (6)	0.055 (7)
C(9)	0.5817 (12)	0.2130 (10)	0.3324 (3)	0.054 (5)
C(10)	0.6905 (15)	0.3943 (11)	0.3716 (4)	0.074 (5)
C(11)	0.4684	0.2421	0.4566	0.045 (8)
C(12)	0.4262	0.2733	0.5225	0.05
C(13)	0.2778	0.1155	0.5537	0.05
C(14)	0.1716	-0.0735	0.5190	0.05
C(15)	0.2138	-0.1046	0.4531	0.05
C(16)	0.3622	0.0532	0.4219	0.039 (7)
N(3')	0.5737 (18)	0.0603 (15)	0.3557 (5)	0.047 (7)
N(4')	0.8070 (18)	0.4170 (16)	0.4284 (6)	0.057 (8)
C(11')	0.7754	0.2380	0.4511	0.039 (7)
C(12')	0.8603	0.2539	0.5151	0.05
C(13')	0.8338	0.0819	0.5441	0.05
C(14')	0.7224	-0.1060	0.5991	0.05
C(15')	0.6375	-0.1219	0.4452	0.05
C(16')	0.6640	0.0501	0.4162	0.037 (7)

angle of $52.3 (1)^\circ$. The water molecule has hydrogen bonds to two Cl atoms and to one of the quinoxalinium cations. The other quinoxalinium cation is hydrogen bonded to one of the Cl atoms. The cations are stacked, so that the two orientations of the disordered quinoxalinium cation are ordered in different stacks. The anions form separate columns in the **a** direction.

Experimental. The title compound was prepared by adding quinoxaline (0.005 mol) to $\text{CoCl}_4 \cdot 6\text{H}_2\text{O}$ (0.005 mol) in ethanol. The light blue precipitate was recrystallized from an ethanol/water/HCl mixture. Dark-blue crystals were isolated, filtered and washed with ethanol and ether. The density was determined by flotation. A crystal of $0.2 \times 0.2 \times 0.25$ mm was used for intensity data collection. Cell parameters were determined from 25 centred reflections ($10.4 < 2\theta < 24.3^\circ$) measured with a Nicolet P3F diffractometer using graphite-monochromated Mo $K\alpha$ radiation. The intensity data were collected with ω - 2θ scans ($2\theta = 3$ - 55° ; h : $0 \rightarrow 10$, k : $-10 \rightarrow 10$, l : $-27 \rightarrow 27$). 4587 unique reflections were measured, 3054 of which where $F_o > 6\sigma(F_o)$ were used for structure solution. Three strong reflections (020, 311, $2\bar{1}\bar{8}$) showed strong decay (*ca* 42%). The intensity data were corrected for the decay and Lorentz and polarization effects but not for absorption. The position of

Table 2. Selected bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

Co(1)—Cl(1)	2.292 (2)	Co(1)—Cl(3)	2.258 (2)
Co(1)—Cl(2)	2.266 (2)	Co(1)—Cl(4)	2.248 (2)
N(1)—C(1)	1.336 (9)	C(4)—C(5)	1.333 (10)
C(1)—C(2)	1.387 (11)	C(5)—C(6)	1.365 (12)
C(2)—N(2)	1.275 (10)	C(6)—C(7)	1.362 (10)
N(2)—C(3)	1.368 (9)	C(7)—C(8)	1.385 (9)
C(3)—C(8)	1.399 (9)	C(8)—N(1)	1.366 (7)
C(3)—C(4)	1.439 (9)	N(3')—C(9)	1.253 (12)
N(3)—C(9)	1.435 (8)	C(10)—N(4')	1.374 (14)
C(9)—C(10)	1.397 (10)	N(4')—C(11')	1.376 (12)
C(10)—N(4)	1.420 (14)	N(3')—C(16')	1.404 (11)
N(4)—C(11)	1.366 (11)		
N(3)—C(16)	1.345 (10)		
Cl(1)—Co(1)—Cl(2)	104.8 (1)	Cl(2)—Co(1)—Cl(3)	113.4 (1)
Cl(1)—Co(1)—Cl(3)	105.4 (1)	Cl(2)—Co(1)—Cl(4)	111.8 (1)
Cl(1)—Co(1)—Cl(4)	110.6 (1)	Cl(3)—Co(1)—Cl(4)	110.6 (1)
N(2)—C(2)—C(1)	126.8 (7)	C(7)—C(6)—C(5)	120.2 (8)
C(3)—N(2)—C(2)	115.8 (6)	C(8)—C(7)—C(6)	118.1 (7)
C(8)—C(3)—N(2)	122.3 (6)	C(3)—C(8)—C(7)	122.5 (6)
C(8)—C(3)—C(4)	116.8 (7)	C(3)—C(8)—N(1)	116.8 (6)
C(4)—C(3)—N(2)	120.9 (7)	C(7)—C(8)—N(1)	120.7 (6)
C(5)—C(4)—C(3)	117.9 (7)	C(1)—N(1)—C(8)	121.7 (6)
C(6)—C(5)—C(4)	124.4 (7)	C(2)—C(1)—N(1)	116.5 (6)
C(9)—N(3)—C(16)	122.7 (3)	C(10)—C(9)—N(3')	116.4 (7)
C(10)—C(9)—N(3)	120.7 (6)	N(4')—C(10)—C(9)	124.0 (8)
N(4)—C(10)—C(9)	117.8 (8)	C(11')—N(4')—C(10)	113.4 (8)
C(11)—N(4)—C(10)	116.2 (9)	C(16')—C(11')—N(4')	125.1 (5)
C(15)—C(16)—N(3)	126.8 (4)	C(12')—C(11')—N(4')	114.8 (8)
C(16)—C(11)—N(4)	128.9 (5)	C(11')—C(16')—N(3')	112.4 (4)
C(12)—C(11)—N(4)	110.9 (5)	C(15')—C(16')—N(3')	127.4 (5)
C(11)—C(16)—N(3)	113.2 (4)	N(3)—C(9)—N(3')	49.9 (6)
C(9')—N(3')—C(16')	127.6 (8)	N(4)—C(10)—N(4')	57.1 (7)

the Co atom was determined from a Patterson map with *SHELXS86* (Sheldrick, 1990). The Cl atoms and one of the quinoxalinium cations were located in successive difference Fourier syntheses. The $\Delta\rho$ map indicated that the other quinoxalinium cation was severely disordered. The heterocyclic ring was refined over two sites (each with occupancies 0.5) except for C(9) and C(10) which act as a hinge between the two orientations of the molecule. The aromatic ring of the disordered quinoxalinium cation was refined with calculated atomic positions over two sites and with isotropic fixed (0.05\AA^2) temperature factors.

The H atoms were located from the difference Fourier map or calculated [H(4), H(6) and H(7)]. All H atoms were refined with fixed coordinates and temperature factors (0.05\AA^2). Refinement on F of 221 parameters by full-matrix least squares with *SHELX76* (Sheldrick, 1976) converged to $R = 0.067$, $wR = 0.069$, $w = 5.5313/[\sigma^2(F_o) + 0.000001(F_o)^2]$, $(\Delta/\sigma)_{\text{max}} = 0.003$, $S = 0.757$. The max. and min. residues in the final difference Fourier map were $+0.95$ and -0.96 e \AA^{-3} , respectively. The atomic scattering factors were as supplied in *SHELX76*, except for Co which were taken from Cromer & Mann (1968). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* with selected bond

* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53630 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

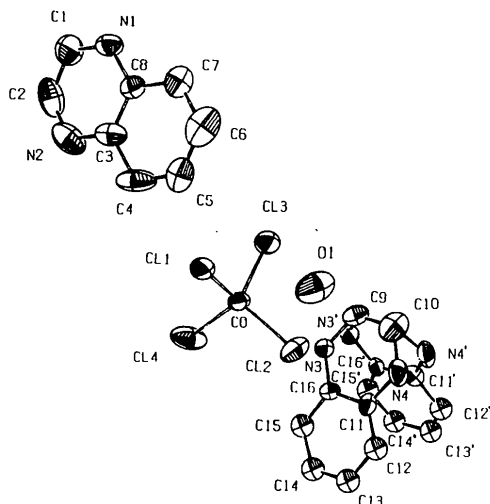


Fig. 1. View of the asymmetric unit showing atom numbering. The second orientation of the disordered quinoxalium cation is marked with primes. Thermal ellipsoids are drawn at the 50% probability level.

lengths and angles in Table 2. The atomic numbering scheme for the asymmetric unit is shown in Fig. 1 (Johnson, 1976) and the packing in Fig. 2 (Motherwell & Clegg, 1978).

Related literature. The $[\text{CoCl}_4]^{2-}$ tetrahedron is described, for example, by Søtøfte & Nielsen (1981)

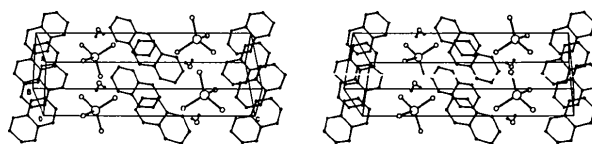


Fig. 2. Stereoview of the structure of $(\text{C}_8\text{H}_7\text{N}_2)_2[\text{CoCl}_4]\cdot\text{H}_2\text{O}$. Only one orientation of the disordered quinoxalium cation is shown. **A** is vertical, **B** is into plane of paper and **C** is horizontal.

and Campayo, Navarro, Cana & Foces-Foces (1987) and the quinoxaline molecule by Lipkowski, Andreotti & Sgarabotto (1977).

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Chloro(tri-*tert*-butylphosphino-*C,P*)(tricyclohexylphosphine)platinum(II)

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Abstract. (*SP-4-4*)-Chloro[2-(di-*tert*-butylphosphino)-2-methylpropyl-*C,P*](tricyclohexylphosphine)platinum(II), $\text{C}_{30}\text{H}_{59}\text{ClP}_2\text{Pt}$, $M_r = 712.27$, triclinic, $P\bar{1}$, $a = 9.986(2)$, $b = 12.998(4)$, $c = 14.170(5)$ Å, $\alpha = 106.18(3)$, $\beta = 94.18(2)$, $\gamma = 91.59(2)^\circ$, $V = 1760.95$ Å³, $Z = 2$, $D_x = 1.343$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 4.21$ mm⁻¹, $F(000) = 728$, $T = 294$ K, final $R = 0.055$ for 4109 unique observed reflections with $I > 3\sigma(I)$. The geometry of the central atom (Pt) is square planar, mean deviation from planarity is 0.011(9) Å. The phosphine ligands are oriented *trans* across the square-planar geometry. The bond angles centered about the Pt atom are distorted owing to the steric bulk of the ligands and

the formation of the four-membered ring, Pt—P(1)—C(2)—C(1), which is severely distorted. The cyclohexyl substituent rings associated with P(2) are in chair conformations and in a staggered orientation. A ligand bulk calculation for the PCy₃ group yields a cone angle, θ , of 136°. Selected geometrical details are: Pt—Cl = 2.409(2), Pt—P = 2.283(14), Pt—C(1) = 2.043(9), P(1)—C = 1.881(10), P(2)—Cy = 1.856(10), C—C = 1.55(3), and C—C (cyclohexyl) = 1.52(2) Å.

Experimental. Crystals of the title compound obtained from Dr H. C. Clark of Dalhousie University, Halifax, Canada; off-white, 0.35 × 0.20 ×