

Fig. 1. The molecule of the title compound in the crystal, showing the atom-labelling scheme. Radii arbitrary, H atoms omitted.

Final atomic coordinates are given in Table 1,\* and selected geometry in Table 2. A perspective view of the title compound is shown in Fig. 1.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44849 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of (3,5-Dimethylpyridine)bis(1,10-phenanthroline)copper(II) Diperchlorate

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**Abstract.**  $[\text{Cu}(\text{C}_7\text{H}_9\text{N})(\text{C}_{12}\text{H}_8\text{N}_2)](\text{ClO}_4)_2$ ,  $M_r = 730.0$ , monoclinic,  $P2_1/n$ ,  $a = 18.430$  (3),  $b = 9.122$  (2),  $c = 18.744$  (8) Å,  $\beta = 97.92$  (3)°,  $V = 3121$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.56$  (1),  $D_x = 1.55$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 9.3$  cm<sup>-1</sup>,  $F(000) = 1492$ ,  $T = 293$  K,  $R = 0.081$  for 1312 unique observed reflections. The Cu-atom environment is approximately square pyramidal, with the apical position occupied by an N atom of a 1,10-phenanthroline (phen) molecule. The Cu atom is 0.256 (3) Å above the basal

plane; the four basal N atoms are planar within 0.10 (2) Å. The Cu–N distances to the phen ligand in the basal plane are 2.01 (1), to the pyridine 2.04 (1) and to the phen which spans the basal-apical positions 2.12 (1) Å. The latter phen is folded [deviations from planarity of up to 0.09 (2) Å] while the basal phen is planar within 0.03 (2) Å.

**Experimental.** Complex (I) prepared by reaction of bis(1,10-phenanthroline)copper(II) perchlorate with

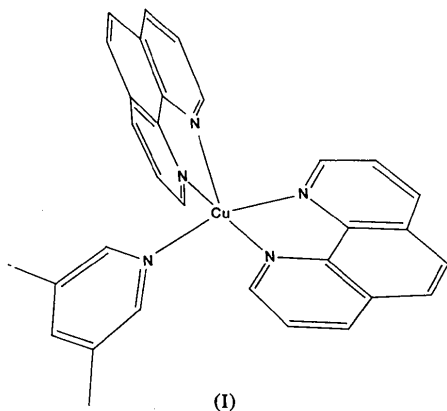
**Related literature.** Preparation of the ligand: Osborne, Whiteley & Meads (1980). Summary of the crystallographic literature on gold compounds: Jones (1981, 1983, 1986). Structure of a gold–ferrocenyl derivative with Au–Fe bonding: Adrianov, Struchkov & Rossinskaya (1974). Use of ferrocenyl–gold compounds in asymmetric synthesis: Ito, Sawamura & Hayashi (1986).

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3,5-dimethylpyridine. Crystals obtained from aqueous solutions. Density by suspension. Blue needle-shaped data crystal 0.10 × 0.10 × 0.58 mm mounted on a glass fiber. Intensities measured with an Enraf-Nonius CAD-4 diffractometer using  $\omega$ -2 $\theta$  scans of 4–16° min<sup>-1</sup> in  $\theta$ . Unit cell determined from least-squares



analysis of angle data for 25 reflections with  $16 < 2\theta < 21^\circ$ . Analytical absorption correction based on crystal-face measurements varied from 0.92 to 1.00. Data collected to  $(\sin\theta)/\lambda$  of  $0.48 \text{ \AA}^{-1}$ ,  $-17 \leq h \leq 17$ ,  $0 \leq k \leq 8$ ,  $-18 \leq l \leq 0$ . Three standard reflections ( $60\bar{6}$ ,  $33\bar{3}$ ,  $53\bar{3}$ ) decreased 0.2% over 25.1 h of data collection. 3014 reflections measured, 2897 unique ( $R_{\text{int}} = 0.02$ ), 1585 reflections with  $I < 3\sigma(I)$  considered unobserved. Solved by Patterson and Fourier methods. Full-matrix least squares minimized  $\sum w(\Delta F)^2$ . H atoms constrained to idealized positions with C–H = 0.95 Å and isotropic  $B$  value of 1.2 times that of the C atom to which it is bonded, except for the methyl H atoms which were not located. The Cu, N and Cl atoms were refined anisotropically, as were the O atoms of one perchlorate ion. The other perchlorate ion was disordered and was modelled with seven O atoms, three of which had an occupancy factor of 0.25 and were not refined. The other four O atoms of the latter perchlorate and all C atoms were refined isotropically, for a total of 249 variables.  $R = 0.081$ ,  $wR = 0.097$ ,  $S = 2.0$ , where non-Poisson  $w^{-1} = [\sigma^2(I) + 0.0036I^2]/4F^2$ . Final  $(\Delta/\sigma)_{\text{max}} < 0.2$ ,  $\Delta\rho_{\text{max}} = 0.6 (1)$  and  $\Delta\rho_{\text{min}} = -0.5 (1) \text{ e \AA}^{-3}$  on final difference map. Atomic scattering factors and anomalous-dispersion correction from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf-Nonius (1982) SDP.\* Tables 1 and 2 give the atom coordinates and selected intramolecular distances and

\* Lists of structure factors, distances and angles involving C atoms, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44832 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates and isotropic or equivalent isotropic thermal parameters

	x	y	z	B/B <sub>eq</sub> (Å <sup>2</sup> )
Cu	0.4831 (1)	0.3650 (3)	0.2606 (1)	4.06 (5)
Cl(1)	0.5593 (3)	0.9422 (7)	0.2039 (3)	7.6 (2)
Cl(2)	0.2290 (3)	0.1066 (8)	0.4274 (3)	7.6 (2)
O(1)	0.538 (1)	0.980 (3)	0.2636 (9)	20.9 (7)
O(2)	0.495 (1)	0.920 (3)	0.155 (1)	14.9 (8)
O(3)	0.6022 (8)	1.046 (2)	0.176 (1)	11.1 (5)
O(4)	0.587 (1)	0.806 (2)	0.200 (1)	17.4 (8)
O(5)	0.272 (1)	0.030 (3)	0.480 (1)	18.8 (9)*
O(6)	0.268 (2)	0.133 (4)	0.377 (2)	17 (1)*
O(7)	0.158 (1)	0.056 (3)	0.413 (1)	11.1 (7)*
O(8)	0.227 (2)	0.240 (4)	0.470 (2)	16 (1)*
O(9)	0.2227	0.0000	0.3711	12*
O(10)	0.2617	0.2324	0.4023	12*
O(11)	0.1649	0.1616	0.4518	12*
N(1)	0.5057 (7)	0.361 (2)	0.1588 (7)	4.4 (4)
N(2)	0.3936 (7)	0.257 (2)	0.2145 (7)	3.3 (4)
N(3)	0.4584 (8)	0.330 (2)	0.3663 (8)	5.2 (4)
N(4)	0.4350 (7)	0.568 (2)	0.2855 (8)	5.8 (5)
N(5)	0.5894 (7)	0.403 (2)	0.3017 (7)	4.3 (4)
C(1)	0.563 (1)	0.414 (2)	0.134 (1)	5.5 (5)*
C(2)	0.578 (1)	0.386 (2)	0.062 (1)	6.3 (5)*
C(3)	0.531 (1)	0.296 (2)	0.020 (1)	5.9 (5)*
C(4)	0.470 (1)	0.233 (2)	0.045 (1)	4.8 (5)*
C(5)	0.4597 (9)	0.273 (2)	0.1162 (9)	3.5 (4)*
C(6)	0.417 (1)	0.140 (2)	0.008 (1)	6.0 (5)*
C(7)	0.361 (1)	0.084 (2)	0.034 (1)	6.9 (6)*
C(8)	0.3486 (9)	0.117 (2)	0.1060 (9)	4.2 (4)*
C(9)	0.3988 (9)	0.212 (2)	0.1447 (9)	3.2 (4)*
C(10)	0.291 (1)	0.068 (2)	0.139 (1)	5.4 (5)*
C(11)	0.2844 (9)	0.114 (2)	0.2080 (9)	4.8 (5)*
C(12)	0.3361 (9)	0.209 (2)	0.242 (1)	4.5 (5)*
C(13)	0.467 (1)	0.200 (2)	0.397 (1)	6.2 (6)*
C(14)	0.447 (1)	0.182 (3)	0.469 (1)	8.3 (7)*
C(15)	0.422 (1)	0.299 (3)	0.498 (1)	6.8 (6)*
C(16)	0.413 (1)	0.427 (2)	0.467 (1)	5.8 (5)*
C(17)	0.4314 (9)	0.447 (2)	0.3934 (9)	4.2 (4)*
C(18)	0.385 (1)	0.557 (3)	0.491 (1)	6.8 (6)*
C(19)	0.372 (1)	0.679 (3)	0.457 (1)	6.8 (6)*
C(20)	0.389 (1)	0.689 (3)	0.384 (1)	6.8 (6)*
C(21)	0.4197 (9)	0.571 (2)	0.3546 (9)	4.1 (4)*
C(22)	0.377 (1)	0.817 (3)	0.342 (1)	7.5 (6)*
C(23)	0.391 (1)	0.816 (3)	0.271 (1)	9.2 (7)*
C(24)	0.422 (1)	0.680 (3)	0.242 (1)	7.7 (6)*
C(25)	0.6311 (9)	0.287 (2)	0.3196 (9)	4.1 (4)*
C(26)	0.706 (1)	0.295 (2)	0.345 (1)	4.8 (5)*
C(27)	0.736 (1)	0.432 (2)	0.351 (1)	5.0 (5)*
C(28)	0.6948 (9)	0.555 (2)	0.3363 (9)	3.9 (4)*
C(29)	0.6196 (9)	0.534 (2)	0.3099 (9)	4.2 (4)*
C(30)	0.751 (1)	0.157 (3)	0.362 (1)	7.0 (6)*
C(31)	0.727 (1)	0.712 (3)	0.343 (1)	6.9 (6)*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as:  $\frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$ .

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Cu–N(1)	2.009 (10)	Cu–N(4)	2.134 (13)
Cu–N(2)	2.010 (10)	Cu–N(5)	2.036 (10)
Cu–N(3)	2.119 (13)		
N(1)–Cu–N(2)	81.3 (5)	N(2)–Cu–N(4)	100.3 (4)
N(1)–Cu–N(3)	170.3 (5)	N(2)–Cu–N(5)	159.6 (5)
N(1)–Cu–N(4)	111.7 (5)	N(3)–Cu–N(4)	77.1 (5)
N(1)–Cu–N(5)	92.7 (4)	N(3)–Cu–N(5)	89.5 (4)
N(2)–Cu–N(3)	93.4 (4)	N(4)–Cu–N(5)	100.1 (5)

angles; Fig. 1 shows the molecule with the numbering scheme used.

**Related literature.** Five-coordinate square-pyramidal complexes of Cu<sup>II</sup> have been reviewed by O'Brien

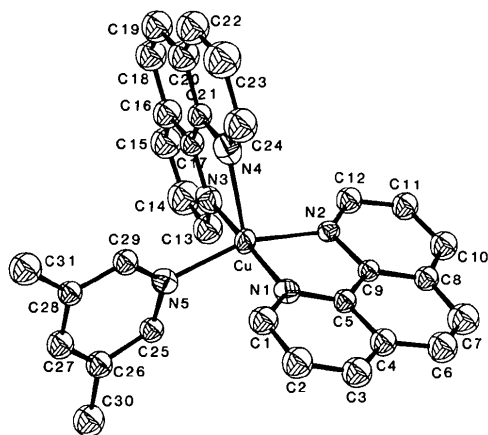


Fig. 1. ORTEP diagram (Johnson, 1976) showing the atom-numbering scheme of the cation. Thermal ellipsoids at 30% probability level.

(1984) and Hathaway (1982, 1983). Structures previously reported for  $\text{Cu}(\text{phen})_2\text{X}$  cations are those with small  $\text{X}$  ligands which have trigonal bipyramidal geometries; these include structures in which  $\text{X}$  is

chloride (Boys, Escobar & Martinez-Carrera, 1981), cyanide (Anderson, 1975) and water (Nakai & Deguchi, 1975; Nakai & Noda, 1978).

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## Bis[(*N,N*-diisopropyldithiocarbamato)(diphenylphosphinito)(diphenylthiophosphinito)-platinato-*O,S*]cobalt(II)

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**Abstract.**  $[\text{CoPt}_2(\text{C}_7\text{H}_{14}\text{NS}_2)_2(\text{C}_{12}\text{H}_{10}\text{OP})_2(\text{C}_{12}\text{H}_{10}\text{PS})_2]$ ,  $M_r = 1638.6$ , monoclinic,  $C2/c$ ,  $a = 29.168$  (23),  $b = 9.693$  (15),  $c = 24.467$  (15) Å,  $\beta = 101.13$  (4)°,  $V = 6788$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.603$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 4.673$  mm<sup>-1</sup>,  $F(000) = 3236$ ,  $T = 293$  K,  $R = 0.0648$  for 3755 unique observed reflections. The Co lies on a crystallographic twofold axis and is tetrahedrally coordinated by one S and one O donor atom from each Pt-containing ligand. At 1.884 (11) Å, the Co–O bond length lies significantly below the range of 1.95–1.97 Å found in the corresponding complex where both donor atoms in each ligand are oxygen. The Co–S distance is 2.319 (4) Å, angles at Co range from 99.7 (5) to

119.2 (3)° and the coordination about the Pt is distorted square-planar.

**Experimental.** Synthesis of title compound (Anderson, Stephenson & Walkinshaw, 1988) by reaction of  $[\text{Pt}(\text{S}_2\text{CN}^i\text{Pr}_2)(\text{Ph}_2\text{PO})(\text{Ph}_2\text{PS})\text{H}]$  (Anderson, Ebsworth, Stephenson & Walkinshaw, 1981, 1982) with  $\text{Co}(\text{acac})_2$ . Blue lamellar crystal 0.04 × 0.24 × 0.52 mm, STADI-2 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation, cell parameters from 11 reflections with  $5.0 < \theta < 19.0^\circ$ . For data collection,  $\omega$  scans with scan width  $1.0 + 0.5(\sin\mu/\tan\theta)^\circ$ ,  $\theta_{\text{max}} = 25^\circ$ ,  $h -34 \rightarrow 33$ ,  $k 0 \rightarrow 11$ ,  $l 0 \rightarrow 28$ , no significant crystal movement or decay, initial absorption correction using  $\psi$  scans, 6166 reflections, 5916 unique ( $R_{\text{int}} = 0.016$ ), giving 2161 with  $F > 6\sigma(F)$  for structure

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