same idealized C_2 symmetry as the $\mathrm{PPh_3}$ analogue (2) (Farrugia *et al.*, 1981). Apart from the hydride-bridged metal–metal vectors the Os atoms have approximate octahedral coordination, and the Pt atoms square-planar geometry (Table 2). Though the differences between the dihedral butterfly angle Pt(1)—Os(1)—Os(2)—Pt(2) = 84·2 (1)° and the non-bonding Pt(1)···Pt(2) separation = 3·230 (1) Å compared with corresponding parameters in (2) [82·9° and 3·206 (1) Å] are statistically significant, they are only marginal. The $\mathrm{Os_2Pt_2}$ framework is evidently rather insensitive to the nature of the phosphine ligand. The slightly greater butterfly angle in (1) compared with (2) may be a result of steric repulsion between the larger cyclohexylphosphine ligands [Tolman cone angles 170 *vs* 145° for $\mathrm{PCy_3}$ and $\mathrm{PPh_3}$ respectively (Tolman, 1977)], or a consequence of the greater electron-releasing ability of $\mathrm{PCy_3}$ [electronic parameters 2056·4 and 2068·9 $\mathrm{cm^{-1}}$ for $\mathrm{PCy_3}$ *vs* $\mathrm{PPh_3}$ (Tolman, 1977)]. Formal electron density at the cluster arising from ligand donation has been suggested as important in determining the butterfly angle in cluster complexes (Carty *et al.*, 1982).

References

BOGDAN, P. L., HORWITZ, C. P. & SHRIVER, D. F. (1986). *J. Chem. Soc. Chem. Commun.* pp. 553–555.

BRUCE, M. I. & NICHOLSON, B. K. (1983). *J. Organomet. Chem.* **250**, 627–638.

CARTY, A. J., MACLAUGHLIN, S. A., WAGNER, J. V. & TAYLOR, N. J. (1982). *Organometallics*, **1**, 1013–1015.

COPPENS, P. (1970). In *Crystallographic Computing*, edited by F. R. AHMED, p. 255. Copenhagen: Munksgaard.

FARRUGIA, L. J., HOWARD, J. A. K., MITRPRACHACHON, P., STONE, F. G. A. & WOODWARD, P. (1981). *J. Chem. Soc. Dalton Trans.* pp. 1274–1277.

GILMORE, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

International Tables for X-ray Crystallography (1974). Vol. IV. pp. 71–151. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MALLINSON, P. R. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

ORPEN, A. G. (1980). *J. Chem. Soc. Dalton Trans.* pp. 2509–2516.

TOLMAN, C. A. (1977). *Chem. Rev.* **77**, 313–348.

Acta Cryst. (1988). C**44**, 820–822

Structure of Dichlorodiquinolinecobalt(II): Isomorphism with the Analogous Co, Ni and Zn Halogenides

BY L. GOLIČ

Department of Chemistry and Chemical Technology, University E. Kardelj, 61000 Ljubljana, Yugoslavia

AND A. MIRČEVA

Institute of Chemistry, Cyril and Methodius University, 91000 Skopje, Yugoslavia

(*Received* 26 *November* 1987; *accepted* 19 *January* 1988)

Abstract. $[\mathrm{CoCl_2(C_9H_7N)_2}]$, $M_r = 388\cdot16$, triclinic, $P\bar{1}$, $a = 8\cdot789\,(1)$, $b = 9\cdot554\,(1)$, $c = 11\cdot026\,(1)$ Å, $\alpha = 80\cdot26\,(1)$, $\beta = 72\cdot19\,(1)$, $\gamma = 71\cdot83\,(1)^\circ$, $V = 835\,(1)$ Å^3, $Z = 2$, $F(000) = 394$, $D_m = 1\cdot53\,(2)$ Mg m^{-3} (by flotation), $D_x = 1\cdot544$ Mg m^{-3}, $\mu(\mathrm{Mo}\,K\alpha) = 1\cdot347$ mm^{-1}, $\lambda(\mathrm{Mo}\,K\alpha) = 0\cdot71069$ Å, $T = 293\,(1)$ K. The structure was solved by Patterson and Fourier methods and refined to $R = 0\cdot059$ for 4302 contributing reflections. The Co atom is tetrahedrally surrounded by two halogen and two N atoms [the mean Co—Cl and Co—N distances are 2·244 (4) and 2·05 (2) Å], the coordination tetrahedron being slightly distorted.

Introduction. The structure determination of the title compound was undertaken as part of studies of MQ_2X_2 complexes (M denotes a divalent metal of the transition series, Q = quinoline, X = halogen element Cl, Br, I or CNS group).

Experimental. Prepared by refluxing cobaltous chloride and quinoline in a 1:2 mole ratio (Brown, Nuttal & Sharp, 1964); dark blue prismatic crystals obtained from ethanol. Cell dimensions determined by least-squares refinement of θ angles of 60 strong reflections in the range $10 < \theta < 12^\circ$, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scan mode, 2θ scan width $(0\cdot7 + 0\cdot3\tan\theta)^\circ$, aperture $(2\cdot4 + 0\cdot9\tan\theta)$ mm, maximum scan time 40 s, $2\theta_{\max}$ 60°, the hemisphere with index range $0 \le h \le 12$, $-13 \le k \le 13$ and $-15 \le l \le 15$ measured, crystal size $0\cdot2 \times 0\cdot2 \times 0\cdot4$ mm, three standard reflections ($25\bar{1}$, 440, 435) checked every 140 reflections measured; no significant decay; orientation

0108-2701/88/050820-03$03.00

controlled every 240 reflections using three reflections, 4880 independent reflections, 3323 observed reflections [$I > 2{\cdot}5\sigma(I)$], $\sigma(I)$ based on counting statistics. Position of the Co atom determined from Patterson map, other non-hydrogen atoms found from a Fourier synthesis. H atoms located at calculated positions, not refined; anisotropic full-matrix least squares for all non-hydrogen atoms, H atoms included in the last cycle of the refinement with isotropic thermal parameter $U = 0{\cdot}06$ Å^2, the empirical weighting function $w = W_fW_s$ where $W_f(|F_o| < 5{\cdot}0) = |F_o|/5{\cdot}0$, $W_f(|F_o| > 8{\cdot}0) = 8{\cdot}0/|F_o|$, $W_f(5{\cdot}0 \le |F_o| \le 8{\cdot}0) = 1{\cdot}0$, $W_s(\sin\theta < 0{\cdot}39) = [(\sin\theta)/0{\cdot}39]^{1{\cdot}5}$, $W_s(\sin\theta > 0{\cdot}46) = [0{\cdot}46/(\sin\theta)]^{1{\cdot}5}$, $W_s(0{\cdot}39 \le \sin\theta \le 0{\cdot}46) = 1{\cdot}0$ was applied to keep $\sum w(\Delta F)^2$ uniform over the ranges of $|F_o|$ and $(\sin\theta)/\lambda$; in the final refinement cycle there were 4302 contributing reflections (m), 208 variables (n), $m/n = 20{\cdot}7$, $S = [\sum w(\Delta F)^2/(m-n)]^{1/2} = 0{\cdot}66$, average Δ/σ 0·01, maximum $\Delta/\sigma = 0{\cdot}05$, maximum and minimum peak heights in final difference Fourier map were 0·60 and −0·62 e Å^{-3}; $R = 0{\cdot}059$, $wR = 0{\cdot}093$; atomic scattering and dispersion factors for the neutral atoms Co, Cl, N and C from Cromer & Mann (1968) and Cromer & Liberman (1970), and for H atoms from Stewart, Davidson & Simpson (1965); DEC-10 computer at RCU Ljubljana, *XRAY*76 system of crystallographic programs (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).

Discussion. The final fractional coordinates with U_{eq} (Hamilton, 1959) for non-H atoms are listed in Table 1.* Fig. 1 illustrates the molecular structure of the compound together with the atom-labelling scheme used. A view of the unit cell is shown in Fig. 2. Interatomic distances and angles are given in Table 2.

The crystal structure consists of CoQ_2Cl$_2$ molecules. The Co atom is four-coordinated by two halogen and two N atoms from the quinoline ligands. The Co—Cl distances, 2·246 (2) and 2·241 (1) Å, are in agreement with those found in the structure of the analogous γ-picoline compound [average Co—Cl = 2·23 Å: Laing & Carr (1975)], and are slightly shorter than those in

Table 1. *Final fractional coordinates* (× 10^4) *and equivalent isotropic temperature factors* U_{eq} (Å^2 × 10^3)

	x	y	z	U_{eq}*
Co	4042 (1)	3021 (1)	2056 (1)	39·5 (3)
Cl(1)	2106 (2)	4330 (1)	1051 (1)	64 (1)
Cl(2)	3117 (2)	1596 (1)	3787 (1)	56 (1)
N(1)	5873 (5)	1674 (4)	744 (3)	41 (2)
C(1)	5672 (6)	1797 (5)	−412 (4)	48 (2)
C(2)	6607 (7)	762 (6)	−1328 (4)	54 (2)
C(3)	7802 (6)	−439 (5)	−1013 (5)	50 (2)
C(4)	8085 (5)	−599 (4)	200 (4)	40 (2)
C(5)	9315 (6)	−1802 (5)	587 (5)	49 (2)
C(6)	9589 (6)	−1890 (5)	1754 (5)	53 (2)
C(7)	8635 (6)	−797 (5)	2605 (5)	52 (2)
C(8)	7402 (6)	361 (5)	2274 (4)	45 (2)
C(9)	7107 (5)	495 (4)	1073 (4)	37 (2)
N(2)	4968 (5)	4460 (4)	2583 (4)	44 (2)
C(11)	6579 (6)	4372 (6)	2112 (6)	57 (3)
C(12)	7335 (8)	5301 (7)	2475 (7)	74 (3)
C(13)	6393 (9)	6320 (7)	3306 (7)	81 (3)
C(14)	4673 (7)	6472 (5)	3805 (4)	58 (2)
C(15)	3606 (11)	7549 (6)	4646 (6)	80 (4)
C(16)	1953 (11)	7665 (7)	5088 (6)	76 (4)
C(17)	1276 (8)	6708 (7)	4725 (6)	63 (3)
C(18)	2272 (6)	5638 (5)	3909 (5)	51 (2)
C(19)	3990 (6)	5507 (4)	3452 (4)	45 (2)

* U_{eq} is defined as $\frac{1}{3}$ of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Molecular dimensions*

Interatomic distances (Å) and angles (°) about Co atom

Co—Cl(1)	2·246 (2)	Co—N(1)	2·061 (3)
Co—Cl(2)	2·241 (1)	Co—N(2)	2·037 (5)
Cl(1)—Co—Cl(2)	114·5 (1)	Cl(2)—Co—N(1)	108·4 (1)
Cl(1)—Co—N(1)	106·2 (1)	Cl(2)—Co—N(2)	108·9 (1)
Cl(1)—Co—N(2)	108·3 (1)	N(1)—Co—N(2)	110·5 (2)

Interatomic distances (Å) and angles (°) in the quinoline ligands 1 and 2

Quinoline ligand 1		Quinoline ligand 2	
N(1)—C(1)	1·321 (7)	N(2)—C(11)	1·331 (6)
C(1)—C(2)	1·414 (7)	C(11)—C(12)	1·429 (10)
C(2)—C(3)	1·371 (7)	C(12)—C(13)	1·345 (9)
C(3)—C(4)	1·408 (7)	C(13)—C(14)	1·410 (9)
C(4)—C(5)	1·419 (6)	C(14)—C(15)	1·410 (7)
C(4)—C(9)	1·425 (5)	C(14)—C(19)	1·420 (9)
C(5)—C(6)	1·365 (9)	C(15)—C(16)	1·358 (12)
C(6)—C(7)	1·407 (7)	C(16)—C(17)	1·399 (12)
C(7)—C(8)	1·375 (6)	C(17)—C(18)	1·376 (7)
C(8)—C(9)	1·402 (7)	C(18)—C(19)	1·411 (7)
C(9)—N(1)	1·384 (5)	C(19)—N(2)	1·375 (5)
C(1)—N(1)—C(9)	118·7 (3)	C(11)—N(2)—C(19)	118·8 (5)
N(1)—C(1)—C(2)	123·8 (4)	N(2)—C(11)—C(12)	122·3 (5)
C(1)—C(2)—C(3)	118·7 (5)	C(11)—C(12)—C(13)	119·3 (6)
C(2)—C(3)—C(4)	119·5 (4)	C(12)—C(13)—C(14)	120·1 (8)
C(3)—C(4)—C(9)	118·9 (3)	C(13)—C(14)—C(19)	118·2 (5)
C(5)—C(4)—C(9)	118·5 (4)	C(15)—C(14)—C(19)	118·8 (6)
C(4)—C(5)—C(6)	120·8 (4)	C(14)—C(15)—C(16)	120·8 (8)
C(5)—C(6)—C(7)	120·4 (4)	C(15)—C(16)—C(17)	120·6 (6)
C(6)—C(7)—C(8)	120·2 (5)	C(16)—C(17)—C(18)	120·6 (6)
C(7)—C(8)—C(9)	120·8 (4)	C(17)—C(18)—C(19)	119·8 (6)
N(1)—C(9)—C(4)	120·4 (4)	N(2)—C(19)—C(14)	121·2 (4)
C(4)—C(9)—C(8)	119·3 (3)	C(14)—C(19)—C(18)	119·3 (4)

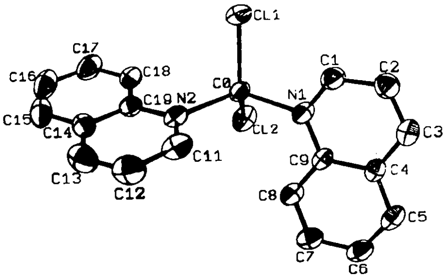

Fig. 1. *ORTEP* (Johnson, 1965) view of the molecule. The atoms are represented by thermal ellipsoids at the 50% probability level.

---

* Lists of structure factors, anisotropic thermal parameters, calculated H coordinates and least-squares-plane equations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44698 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

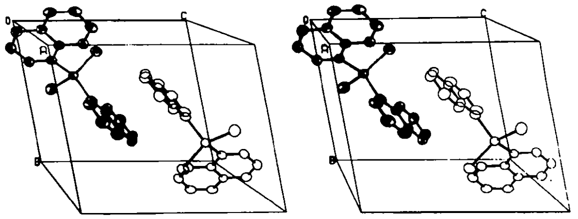

Fig. 2. Stereoview of the molecular packing.

Table 3. *The unit-cell parameters of the isomorphous compounds of the type MQ_2X_2*

	a(Å)	b(Å)	c(Å)	α(°)	β(°)	γ(°)
CoQ_2Cl_2	8·789(2)	9·554(2)	11·026(2)	80·26(2)	72·19(2)	71·83(2)
CoQ_2Br_2	8·689(3)	9·652(2)	11·238(3)	80·74(2)	74·83(3)	73·26(2)
ZnQ_2Cl_2	8·822(2)	9·552(2)	11·028(2)	80·33(2)	72·36(2)	71·69(2)
ZnQ_2Br_2	8·677(1)	9·646(3)	11·276(3)	80·85(2)	74·51(4)	73·27(3)
NiQ_2Cl_2	8·703(1)	9·511(1)	11·075(1)	81·36(1)	73·12(1)	72·01(1)

The unit-cell parameters have been determined by least-squares methods as described in the text for all five compounds.

the octahedrally coordinated pyridine analogue [average Co—Cl = 2·50 Å: Dunitz (1957)]. The Co—N distances, 2·061 (3) and 2·037 (5) Å, are nearly the same as the corresponding distances in the analogous tetrahedral compound of γ-picoline (Co—N = 2·06 and 2·03 Å), and slightly shorter than those in the pyridine complex cited above, where the average Co—N distance is 2·14 Å. The angles range from 106·2 (1) to 114·5 (1)° within the coordination tetrahedron.

The bond-length and angle ranges in the quinoline ligands are in agreement with the corresponding ranges observed in other related structures, *e.g.* tetra-*n*-butylammonium tribromo(quinoline)nickelate(II) (Horrocks, Templeton & Zalkin, 1968) and bis(acetylacetonato)-bis(6-methylquinoline)cobalt(II) (Hursthouse & Malik, 1978). The quinoline ligands are planar. The maximum deviations of the atoms from the corresponding mean planes of the ligands are 0·029 and 0·026 Å, and the average deviations are 0·019 and 0·016 Å. The interplanar angle between the two independent ligands is 66·2 (5)°.

The title compound is isomorphous with the analogous complexes CoQ_2Br_2, ZnQ_2Cl_2, ZnQ_2Br_2 and NiQ_2Cl_2 (blue isomer), as shown by the comparison of their unit-cell parameters (Table 3).

The financial support of the Research Community of Slovenia is gratefully acknowledged.

References

Brown, D. H., Nuttal, R. H. & Sharp, D. W. A. (1964). *J. Inorg. Nucl. Chem.* **26**, 1151–1156.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Dunitz, J. D. (1957). *Acta Cryst.* **10**, 307–313.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Horrocks, W. D., Templeton, D. H. & Zalkin, A. (1968). *Inorg. Chem.* **7**, 2303–2311.

Hursthouse, M. B. & Malik, K. M. A. (1978). *Acta Cryst.* B**34**, 1355–1357.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Laing, M. & Carr, G. (1975). *Acta Cryst.* B**31**, 2683–2684.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1988). C**44**, 822–825

The Structure of μ-(1,1-Dicyanoethylene-2,2-dithiolato-*S*,*S*′)-bis(triphenylphosphine)digold(I) Diethyl Ether Solvate

By Md. Nazrul I. Khan, Suning Wang, Dwight D. Heinrich and John P. Fackler Jr*

Laboratory for Molecular Structure and Bonding, Department of Chemistry, Texas A&M University, College Station, Texas 77843, *USA*

(*Received* 21 *July* 1987; *accepted* 5 *January* 1988)

Abstract. $[Au_2(C_4N_2S_2)(C_{18}H_{15}P)_2].C_4H_{10}O$, M_r = 1132·82, monoclinic, $C2/c$, a = 23·556 (3), b = 14·655 (4), c = 16·138 (3) Å, β = 128·95 (1)°, V = 4332·3 (14) Å^3, D_x = 1·73 g cm^{-3}, Z = 4, μ = 71·4 cm^{-1}, λ(Mo $K\alpha$) = 0·71073 Å, F(000) = 2184, T = 298 K. Convergence to final conventional R values of R = 0·0344 and wR = 0·0367 was obtained using 195 variable parameters and 2260 reflections with $|F_o|^2 > 3\sigma(|F_o|^2)$. The structure consists of two units of $AuPPh_3$ bridged by a 1,1-dicyanoethylene-2,2-dithio-

---

* To whom correspondence should be addressed.

0108-2701/88/050822-04$03.00