Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ag	0.05637 (8)	0.07364 (2)	0.04848 (6)	0.0528 (2)
Ni	1/2	0	0	0.0283 (2)
O1	0.6311 (7)	0.1889 (2)	0.0317 (5)	0.0459 (9)
O2	0.6125 (6)	0.0759 (2)	0.5136 (5)	0.0377 (8)
O3	0.2378 (10)	0.2611 (2)	−0.0100 (7)	0.0630 (13)
O4	0.0557 (7)	0.2291 (2)	0.2461 (6)	0.0489 (9)
O5	0.2623 (9)	0.1412 (2)	0.5424 (7)	0.0581 (11)
N1	0.5514 (7)	0.0847 (2)	−0.0372 (6)	0.0340 (9)
N2	0.6221 (8)	0.1250 (2)	0.2601 (6)	0.0368 (9)
N3	0.5512 (7)	0.0169 (2)	0.2501 (5)	0.0340 (9)
N4	0.0666 (9)	0.0298 (3)	0.2987 (7)	0.0454 (11)
N5	0.0214 (10)	0.1308 (3)	−0.1858 (7)	0.0545 (13)
C1	0.6006 (8)	0.1331 (2)	0.0765 (6)	0.0336 (10)
C2	0.5930 (8)	0.0706 (2)	0.3453 (7)	0.0314 (9)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Ag—N5	2.119 (5)	O2—C2	1.264 (6)
Ag—N4	2.125 (5)	N1—C1	1.309 (6)
Ni—N1	1.868 (4)	N2—C2	1.377 (6)
Ni—N3	1.869 (4)	N2—C1	1.384 (6)
O1—C1	1.268 (6)	N3—C2	1.325 (6)
N5—Ag—N4	170.0 (2)	O1—C1—N1	124.7 (4)
N1—Ni—N3^i	89.8 (2)	O1—C1—N2	115.5 (4)
N1—Ni—N3	90.2 (2)	N1—C1—N2	119.8 (4)
C1—N1—Ni	131.4 (3)	O2—C2—N3	124.7 (4)
C2—N2—C1	128.0 (4)	O2—C2—N2	116.1 (4)
C2—N3—Ni	131.2 (3)	N3—C2—N2	119.3 (4)

Symmetry code: (i) $1 - x, -y, -z$.

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	H···A	D···A	D—H···A
O3—H301···O1	2.10 (2)	2.940 (7)	171 (7)
O3—H302···O4	1.93 (2)	2.765 (7)	167 (7)
O4—H401···O1^i	1.99 (2)	2.816 (7)	164 (7)
O4—H402···O1^{ii}	1.98 (6)	2.701 (7)	142 (8)
O5—H501···O4	2.03 (2)	2.871 (7)	173 (8)
O5—H502···O2	1.97 (3)	2.787 (7)	160 (7)

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

Data collection: *Nicolet P*3 *Software* (Nicolet XRD Corporation, 1980). Cell refinement: *Nicolet P*3 *Software*. Data reduction: *Nicolet P*3 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(2,4-dimorpholino-6-phenyl-1,3,5-oxadiazinium) Hexachlorodicuprate

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Abstract

The title compound, $2C_{17}H_{21}N_4O_3^+.Cu_2Cl_6^{2-}$, has thermochromic properties. The oxadiazinium and phenyl rings are planar whereas the morpholine rings have chair conformations.

Comment

The title compound (1) was synthesized by the reaction of bis[(*N*-morpholinothiocarbonyl)benzamidato]-copper(II) with $SOCl_2$ in a similar manner to that described by Hartung, Beyer, Fernández, Tudela & Gutiérrez-Puebla (1991) and Hartung, Beyer, Gutiérrez-Puebla, Fernández & Olk (1992) for related compounds. Reversible dependence of colour on temperature is known as thermochromism (Day, 1963). Such change in colour is often associated with temperature-dependent changes in the coordination geometry of the chromophore (Grenthe, Paoletti, Sandström & Glikberg, 1979). (1) is a thermochromic coordination compound

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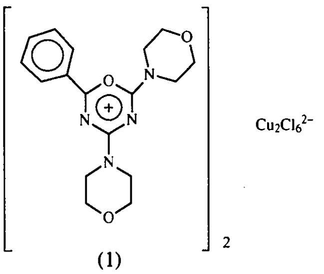
and the crystal structure gives information needed to explain the thermochromic transitions. In this case the thermochromism is expected to be caused by deformation of the $Cu_2Cl_6^{2-}$ group as a function of temperature. The crystal is brown at room temperature. At 140 K the crystals are greenish yellow, but as yet the low-temperature crystal structure has not been solved.



(1)

In (1) bond distances in the cationic 1,3,5-oxadiazinium ring agree well with those previously reported (Rushton, Schwalbe & Stevens, 1983; Al-Talib, Jochims, Zsolnai & Huttner, 1985; Hartung, Beyer, Gutiérrez-Puebla, Fernández & Olk, 1992). The oxadiazinium ring is planar [maximum deviation −0.045 Å for C(4)], as is the phenyl ring, and the angle between these rings is 2.6°. The morpholine rings adopt chair conformations and their mean planes form angles of 22.9 and 15.3° with the oxadiazinium ring. The structure of the centrosymmetric $Cu_2Cl_6^{2-}$ anion can be described as two flattened tetrahedra sharing an edge. The shortest distance calculated between the anion and the cation is 3.244 (6) Å [Cl(1)···O(1)].

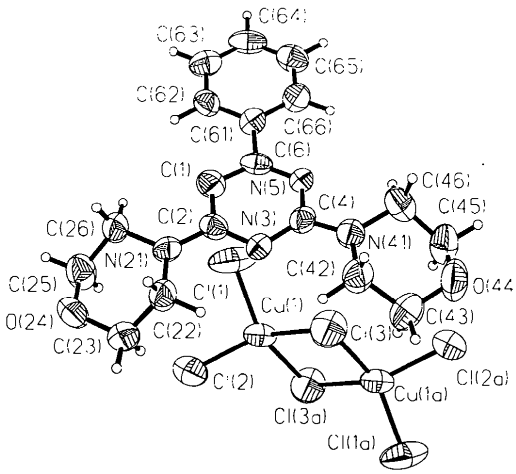


Fig. 1. View of (1) with non-H atoms labelled. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

Crystal data

$2(C_{17}H_{21}N_4O_3)[Cu_2Cl_6]$
$M_r = 998.6$
Monoclinic
$P2_1/n$
$a = 8.384$ (4) Å
$b = 17.147$ (4) Å
$c = 14.889$ (4) Å
$\beta = 99.26$ (3)°
$V = 2112$ (1) Å^3
$Z = 2$
$D_x = 1.57$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 4$–7°
$\mu = 1.44$ mm^{-1}
$T = 296$ (2) K
Square rod
0.30 × 0.20 × 0.20 mm
Brown

Data collection

Rigaku AFC-6*S* four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3851 measured reflections
3708 independent reflections
1774 observed reflections [$I > 1.5\sigma(I)$]

$\theta_{max} = 25.00°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 20$
$l = -17 \rightarrow 17$
3 standard reflections monitored every 150 reflections intensity variation: none

Refinement

Refinement on *F*
$R = 0.056$
$wR = 0.056$
$S = 0.66$
1774 reflections
253 parameters
H-atom parameters not refined
Unit weights applied

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Cu(1)	0.4898 (1)	0.92164 (8)	1.42961 (7)	0.0626
Cl(1)	0.4216 (4)	0.9142 (2)	1.2820 (2)	0.0980
Cl(2)	0.5388 (5)	0.7974 (2)	1.4572 (2)	0.1072
Cl(3)	0.3303 (4)	1.0285 (2)	1.4500 (2)	0.0865
O(1)	0.0773 (7)	0.9213 (4)	1.1469 (4)	0.0573
O(24)	−0.051 (1)	0.6814 (4)	1.2937 (5)	0.0724
O(44)	−0.0660 (9)	1.2168 (4)	1.4214 (5)	0.0805
N(3)	−0.0281 (8)	0.9647 (4)	1.2766 (4)	0.0474
N(5)	0.0965 (8)	1.0522 (4)	1.1858 (4)	0.0416
N(21)	−0.0335 (9)	0.8358 (4)	1.2362 (4)	0.0530
N(41)	−0.0329 (9)	1.0967 (4)	1.2991 (5)	0.0543
C(2)	0.005 (1)	0.9090 (5)	1.2214 (5)	0.0467
C(4)	0.012 (1)	1.0366 (5)	1.2536 (5)	0.0473
C(6)	0.122 (1)	0.9948 (6)	1.1340 (5)	0.0473
C(22)	−0.126 (1)	0.8159 (5)	1.3086 (6)	0.0605
C(23)	−0.057 (1)	0.7431 (6)	1.3558 (6)	0.0730
C(25)	0.052 (1)	0.7014 (6)	1.2303 (7)	0.0715
C(26)	−0.010 (1)	0.7711 (5)	1.1753 (6)	0.0596
C(42)	−0.136 (1)	1.0870 (6)	1.3686 (6)	0.0675
C(43)	−0.078 (1)	1.1389 (7)	1.4480 (7)	0.0802
C(45)	0.048 (1)	1.2233 (6)	1.3608 (8)	0.0796
C(46)	−0.004 (1)	1.1777 (6)	1.2754 (7)	0.0695
C(61)	0.2026 (9)	1.0072 (5)	1.0554 (5)	0.0470
C(62)	0.231 (1)	0.9469 (5)	0.9977 (6)	0.0544
C(63)	0.301 (1)	0.9613 (7)	0.9220 (6)	0.0654
C(64)	0.346 (1)	1.0356 (7)	0.9030 (6)	0.0583
C(65)	0.319 (1)	1.0957 (6)	0.9592 (7)	0.0643
C(66)	0.248 (1)	1.0827 (6)	1.0350 (6)	0.0595

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

Cu(1)—Cu(1^{i})	3.395 (3)	N(5)—C(4)	1.350 (10)
Cu(1)—Cl(1)	2.183 (3)	N(5)—C(6)	1.290 (10)
Cu(1)—Cl(2)	2.197 (3)	N(21)—C(2)	1.324 (10)
Cu(1)—Cl(3)	2.317 (3)	N(21)—C(22)	1.463 (10)
Cu(1)—Cl(3^{i})	2.313 (3)	N(21)—C(26)	1.466 (10)
O(1)—C(2)	1.361 (9)	N(41)—C(4)	1.321 (10)
O(1)—C(6)	1.339 (10)	N(41)—C(42)	1.460 (10)
O(24)—C(23)	1.411 (10)	N(41)—C(46)	1.464 (11)
O(24)—C(25)	1.415 (10)	C(6)—C(61)	1.456 (11)
O(44)—C(43)	1.401 (12)	C(22)—C(23)	1.502 (12)
O(44)—C(45)	1.420 (12)	C(25)—C(26)	1.493 (12)
N(3)—C(2)	1.321 (10)	C(42)—C(43)	1.497 (13)
N(3)—C(4)	1.336 (10)	C(45)—C(46)	1.495 (13)
Cl(1)—Cu(1)—Cl(2)	98.2 (1)	O(1)—C(2)—N(3)	124.1 (8)
Cl(1)—Cu(1)—Cl(3)	96.6 (1)	O(1)—C(2)—N(21)	115.9 (8)
Cl(2)—Cu(1)—Cl(3)	146.8 (1)	N(3)—C(2)—N(21)	120.0 (8)
Cl(1)—Cu(1)—Cl(3^{i})	146.5 (1)	N(3)—C(4)—N(5)	123.9 (8)
Cl(2)—Cu(1)—Cl(3^{i})	97.7 (1)	N(3)—C(4)—N(41)	118.9 (8)
Cl(3)—Cu(1)—Cl(3^{i})	85.7 (1)	N(5)—C(4)—N(41)	117.2 (8)
Cu(1)—Cl(3)—Cu(1^{i})	94.3 (1)	O(1)—C(6)—N(5)	123.5 (7)
C(2)—O(1)—C(6)	115.9 (7)	O(1)—C(6)—C(61)	115.5 (8)
C(23)—O(24)—C(25)	110.3 (8)	N(5)—C(6)—C(61)	121.0 (8)
C(43)—O(44)—C(45)	110.0 (8)	N(21)—C(22)—C(23)	109.3 (8)
C(2)—N(3)—C(4)	114.8 (7)	O(24)—C(23)—C(22)	111.6 (8)
C(4)—N(5)—C(6)	117.1 (7)	O(24)—C(25)—C(26)	111.5 (8)
C(2)—N(21)—C(22)	121.1 (7)	N(21)—C(26)—C(25)	109.6 (7)
C(2)—N(21)—C(26)	123.6 (7)	N(41)—C(42)—C(43)	109.8 (8)
C(22)—N(21)—C(26)	114.7 (7)	O(44)—C(43)—C(42)	111.8 (9)
C(4)—N(41)—C(42)	121.7 (8)	O(44)—C(45)—C(46)	111.5 (9)
C(4)—N(41)—C(46)	122.9 (8)	N(41)—C(46)—C(45)	109.2 (8)
C(42)—N(41)—C(46)	114.8 (7)		

Symmetry code: (i) $1-x, 2-y, 3-z$.

ψ scans indicated that absorption was negligible in this case (minimum transmission factor 0.96, average 0.98). The H atoms were placed geometrically (C—H distance 1.00 Å) and treated as riding atoms with a fixed $U_{\rm iso} = 0.08$ Å^2. MSC/AFC software was used for data collection and cell determination, and *TEXSAN* software (Molecular Structure Corporation, 1989) for data reduction. The structure was solved by direct methods using *SHELXS*86 programs (Sheldrick, 1990), refined by full-matrix least squares and prepared for publication using the *CRYSTALS* program (Watkin, Carruthers & Betteridge, 1990). Molecular graphics were prepared using *SHELXTL-Plus* (Sheldrick, 1989).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dichloro(ethylenediamine)platinum(II)

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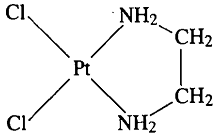
Abstract

The crystal structure of a new polymorph of dichloro(1,2-ethanediamine)platinum(II), $[PtCl_2(C_2H_8N_2)]$, has been determined and is described. Average Pt—N and Pt—Cl bond lengths are 2.032 and 2.318 Å, respectively.

Comment

Two crystal products were obtained from the reaction of $[Pt(en)Cl_2(OH)_2]$ with acetic anhydride in dichloromethane (en = ethylenediamine). One was the expected compound, $[Pt(en)Cl_2(OCOCH_3)_2]$ (Ellis, Er & Hambley, unpublished work), and the other was found to be a new polymorph of $[Pt(en)Cl_2]$ and is described herein.



(I)

$[Pt(en)Cl_2]$ is a close analogue of the widely used anticancer drug cisplatin {*cis*-$[Pt(NH_3)_2Cl_2]$} and has similar anticancer activity (Cleare & Hoeschele, 1973). In the structure of the previously reported polymorph, the complex was found to lie on a twofold axis in the orthorhombic space group $C222_1$

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