Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

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

	x	y	z	U_{eq}
Cu1	0.02025 (5)	0.13736 (8)	0.27088 (4)	0.0348 (3)
Cl1	−0.06963 (12)	−0.0144 (2)	0.13690 (9)	0.0525 (4)
Cl2	−0.02378 (13)	0.4119 (2)	0.21378 (9)	0.0506 (4)
N1	0.1573 (3)	−0.0303 (6)	0.3314 (3)	0.0350 (10)
N2	0.0250 (3)	0.1991 (5)	0.4009 (3)	0.0332 (10)
C1	0.1621 (5)	−0.1945 (7)	0.2977 (4)	0.0412 (13)
C2	0.2583 (5)	−0.3027 (8)	0.3413 (4)	0.051 (2)
C3	0.3487 (5)	−0.2486 (9)	0.4220 (4)	0.056 (2)
C4	0.3433 (5)	−0.0819 (9)	0.4591 (4)	0.050 (2)
C5	0.2471 (4)	0.0254 (7)	0.4113 (3)	0.0376 (13)
C6	0.2378 (5)	0.2084 (7)	0.4463 (4)	0.0445 (13)
C7	0.1316 (4)	0.2298 (7)	0.4722 (3)	0.0351 (12)
C8	0.1399 (5)	0.2790 (7)	0.5618 (4)	0.0448 (14)
C9	0.0397 (6)	0.2996 (8)	0.5802 (4)	0.0513 (15)
C10	−0.0690 (5)	0.2687 (7)	0.5076 (4)	0.0465 (14)
C11	−0.0751 (4)	0.2173 (7)	0.4181 (3)	0.0378 (12)
C12	−0.1897 (5)	0.1826 (9)	0.3371 (4)	0.053 (2)

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

Cu1—N1	2.010 (4)	C3—C4	1.397 (9)
Cu1—N2	2.013 (4)	C4—C5	1.376 (7)
Cu1—Cl1	2.224 (2)	C5—C6	1.508 (8)
Cu1—Cl2	2.242 (2)	C6—C7	1.496 (7)
N1—C5	1.346 (6)	C7—C8	1.379 (7)
N1—C1	1.359 (7)	C8—C9	1.358 (8)
N2—C11	1.345 (6)	C9—C10	1.371 (8)
N2—C7	1.346 (6)	C10—C11	1.391 (7)
C1—C2	1.370 (8)	C11—C12	1.479 (7)
C2—C3	1.353 (8)		
N1—Cu1—N2	89.7 (2)	C5—C4—C3	119.0 (5)
N1—Cu1—Cl1	96.64 (12)	N1—C5—C4	120.9 (5)
N2—Cu1—Cl1	146.99 (13)	N1—C5—C6	117.6 (4)
N1—Cu1—Cl2	142.80 (12)	C4—C5—C6	121.5 (5)
N2—Cu1—Cl2	94.36 (12)	C7—C6—C5	112.5 (4)
Cl1—Cu1—Cl2	99.70 (6)	N2—C7—C8	121.5 (5)
C5—N1—C1	119.4 (4)	N2—C7—C6	115.1 (4)
C5—N1—Cu1	116.7 (3)	C8—C7—C6	123.4 (4)
C1—N1—Cu1	123.9 (3)	C9—C8—C7	120.5 (5)
C11—N2—C7	118.8 (4)	C8—C9—C10	118.1 (5)
C11—N2—Cu1	122.2 (3)	C9—C10—C11	120.4 (5)
C7—N2—Cu1	119.0 (3)	N2—C11—C10	120.7 (5)
N1—C1—C2	121.4 (5)	N2—C11—C12	116.5 (4)
C3—C2—C1	119.6 (6)	C10—C11—C12	122.8 (5)
C2—C3—C4	119.6 (5)		

Data collection and cell refinement: *P3/P4-PC Diffractometer Program* (Siemens, 1991). Data reduction: *SHELXTL/PC XDISK* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL/PC XS*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC XP*. Software used to prepare material for publication: *SHELXL93 CIFTAB*.

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Acta Cryst. (1994). C**50**, 1884–1886

Diamminesilver(I) Bis(biuretato-N^1,N^5)-nickelate(II) Hexahydrate

A. Pajunen and S. Pajunen

Division of Inorganic Chemistry, Department of Chemistry, University of Helsinki, PO Box 6, FIN 00014 Helsinki, Finland

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Abstract

The title compound, $[Ag(NH_3)_2]_2[Ni(C_2H_3N_3O_2)_2].6H_2O$, consists of planar $[Ni(biu)_2]^{2-}$ anions (biu is the biuret ligand), with square-planar coordination of the Ni atom, and almost linear diamminesilver(I) cations. The cations and anions are stacked in a columnar fashion along the *a* axis. The packing is determined by intermolecular hydrogen bonds involving the cations, anions and water molecules.

Comment

Biuret easily forms complexes with metal ions and the structures of the copper(II) and cobalt(III) complexes (Birker & Beurskens, 1974; Freeman, Smith & Taylor, 1961; Pajunen & Pajunen, 1982) have been determined. However, only a few nickel(II)–biuretato complexes have been discussed (Traube, 1922) and no crystal

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: BK1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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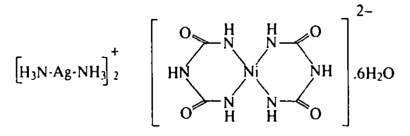
structure determination has been carried out. The present structure determination was undertaken in order to determine the coordination geometry around the Ni atom, the anion being located at a crystallographic centre of symmetry. The title structure (I) consists of diamminesilver(I) cations, bis(biuretato)nickelate(II) anions and water molecules. The diamminesilver(I) cation has an almost linear structure with Ag—N bond lengths of 2.119 (5) and 2.125 (5) Å and an N—Ag—N angle of 170.0 (2)°. In the complex anion the coordination about the Ni atom is square planar with Ni—N distances of 1.868 (4) and 1.869 (4) Å. The anion is almost planar with a mean deviation of the atoms from the least-squares plane of 0.016 (6) Å. The bond lengths and angles within the biuret ligand are essentially the same as those found in the analogous copper and cobalt complexes.



(I)

The diamminesilver(I) cations are located above and below the $[Ni(biu)_2]^{2-}$ planes and the cations and anions are stacked in a columnar fashion parallel to the *a* axis. Along a stack, the cations and anions are nearly equally spaced with the shortest intermolecular Ag···N(1)($x-1$, y, z), Ag···N(3) and Ag···Ni distances being 3.177 (4), 3.293 (4) and 3.478 (1) Å, respectively. All water H atoms form hydrogen bonds with O···O separations in the range 2.701 (7)–2.940 (7) Å.

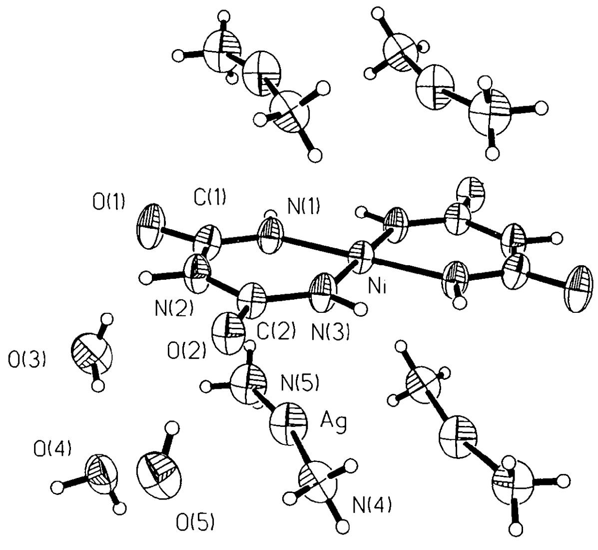


Fig. 1. A drawing of the title structure showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

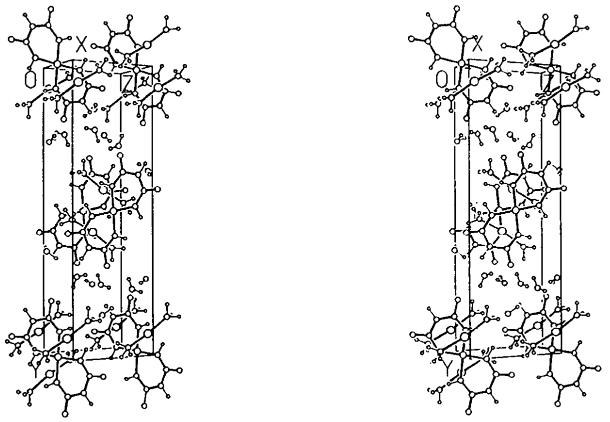


Fig. 2. Packing diagram of the unit cell.

Experimental

The compound was prepared according to the procedure described by Traube & Wolff (1927).

Crystal data

$[Ag(NH_3)_2]_2[Ni(C_2H_3N_3O_2)_2].6H_2O$
$M_r = 652.83$
Monoclinic
$P2_1/n$
$a = 6.647$ (3) Å
$b = 21.162$ (3) Å
$c = 7.737$ (3) Å
$\beta = 112.02$ (3)°
$V = 1008.9$ (6) Å^3
$Z = 2$
$D_x = 2.149$ Mg m^{-3}
$D_m = 2.12$ (3) Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–13°
$\mu = 2.909$ mm^{-1}
$T = 293$ (2) K
Prism
0.4 × 0.2 × 0.1 mm
Yellow

Data collection

Nicolet *P*3 diffractometer
ω–2θ scans
Absorption correction: empirical
$T_{\min} = 0.812$, $T_{\max} = 0.945$
2442 measured reflections
2258 independent reflections
1892 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0430$
$\theta_{\max} = 27.50°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 27$
$l = -10 \rightarrow 9$
3 standard reflections monitored every 60 reflections
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.0663$
$wR(F^2) = 0.1361$
$S = 1.089$
2256 reflections
164 parameters
H atoms riding with C—H, N—H = 0.96 and O—H = 0.85 Å

$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2 + 2.6307P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.058$
$\Delta\rho_{\max} = 1.410$ e Å^{-3}
$\Delta\rho_{\min} = -0.911$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

JUSSI KIVIKOSKI

Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, England

VICENTE FERNÁNDEZ

Departamento de Quimica (Inorgánica), Universidad Autóma de Madrid, Cantoblanco, 28049 Madrid, Spain

JUDITH A. K. HOWARD

Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, England

JÜRGEN HARTUNG

Departamento de Quimica (Inorgánica), Universidad Autóma de Madrid, Cantoblanco, 28049 Madrid, Spain

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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