Table 3. *Selected Au···Au intermolecular distances (Å) for unsupported two-coordinate S—Au—S complexes*

Complex	Au···Au
$[Au(thiourea)_2]Br$[a]	3.40
$[Au(thiourea)_2]ClO_4$[b]	3.58
$[Au(ethylthiourea)_2]Cl.H_2O$[c]	none
$[Au\{SC(NHCH_3)OCH_2CH_3\}_2]Cl$[d]	none
$[Au(SC_5N)_2]ClO_4$[e]	3.285
$[Au\{SC(NHCH_3)_2\}_2]ClO_4$[f]	3.620 (4)
$[Au\{SC(NEt)_2\}_2]ClO_4$[f]	4.294 (2)

References: (*a*) Porter, Fackler, Costamagna & Schmidt (1992); (*b*) Chi-Chang, Shi-Xiong & Rong-Sheng (1990); (*c*) Jones, Guy & Sheldrick (1976); (*d*) Castellato, Fracasso, Grayiani, Sindellari, Gonyáles & Nicolini (1990); (*e*) Uson *et al.* (1990); (*f*) present study.

Data were collected using a Siemens SMART CCD-based (charge-coupled device) diffractometer equipped with an LT-2 low-temperature apparatus operating at 213 K. A suitable crystal was chosen and mounted on a glass fiber using grease. Data were measured using ω scans of 0.3° per frame for 30 s, such that a hemisphere was collected. A total of 1271 frames was collected with a final resolution of 0.85 Å. The first 50 frames were recollected at the end of data collection to monitor for decay. Cell parameters were retrieved using *SMART* software and refined using *SAINT* (Siemens, 1995) on all observed reflections. Data reduction was performed using the *SAINT* software which corrects for Lp and decay. Absorption corrections were applied using *SADABS* supplied by George Sheldrick based on the method of Blessing (1995). The structures were solved by direct methods using the *SHELXS*86 (Sheldrick, 1985) program and refined by the least-squares method on F^2 using *SHELXL*93 (Sheldrick, 1993), incorporated in *SHELXTL/PC* (Sheldrick, 1990). Both compounds did not diffract very well beyond 45° and these data were not used for refinement. C-bound H atoms were placed in idealized positions [C—H = 0.96 Å and $U(H)$ = 0.08 Å^3 (fixed)]. N-bound and O-bound H atoms were located in a difference Fourier map. The H-atom parameters were not refined. Compound (2) was refined as a racemic twin using *SHELXTL/PC* software. Attempts to place the complex in a higher space group failed to give satisfactory results [Flack parameter = 0.14 (4)].

For both compounds, data collection: *ASTRO* (Siemens, 1995); cell refinement: *SMART*; molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *CIFTAB* in *SHELXL*93.

Support from the National Science Foundation CHE 8708625 and the Robert A. Welch Foundation is gratefully acknowledged. We would like to thank the Single-Crystal X-ray Diffraction Laboratory at the University of Idaho for data collection on the Siemens SMART CCD diffractometer system. Funding for the laboratory was provided by the NSF-Idaho EPSCoR program under NSF OSR-9350539 and the M. J. Murdock Charitable Trust, Vancouver, Washington.

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Acta Cryst. (1997). C**53**, 1558–1561

Poly{guanidinium bis[dichloromercury(II)]-μ_4-chloro}

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(*Received 5 February 1997; accepted 27 May 1997*)

Abstract

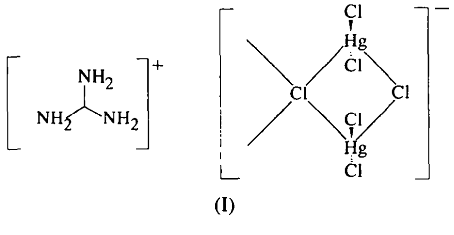
Both the $[C(NH_2)_3]^+$ cations and the $[Hg_2Cl_5]^-$ anions of the title compound occupy special positions on two different twofold axes, one passing through the central Cl atom of the anion (along [100]), the other along a C—N bond of the cation (along [010]). The coordination polyhedron around the Hg atom can be described as

Acta Crystallographica Section C
ISSN 0108-2701 © 1997

a distorted octahedron of six Cl atoms, with two short [2.306 (5) and 2.322 (4) Å], two long [2.905 (7) and 2.906 (7) Å] and two very long [3.271 (6) and 3.344 (6) Å] Hg—Cl distances. In the crystal structure, there are alternate layers of cations and polyanions. Taking the very long Hg—Cl contacts into account, the polyanions are built of $Hg_2Cl_5^-$ units (which can be described as two $HgCl_2$ fragments connected *via* a chlorine anion), interconnected through the common square-planar coordinated Cl atom. Two factors act to stabilize the structure of the crystal, *i.e.* the electrostatic interaction between the adjacent layers and the network of N—H···Cl hydrogen bonds.

Comment

It is known that guanidinium cations, $C(NH_2)_3^+$, are capable of forming stable ionic crystals with metal–halide complex anions (Mellor, 1936). However, studies concerning these compounds are still fragmentary. Recently one can observe growing interest in this group (Grottel, Kozak, Małuszyńska & Pająk, 1992; Waśkowska, Janczak & Czapla, 1993; Zaleski & Pietraszko, 1994*a*,*b*), stimulated by the interesting properties of the guanidinium cation. This simple chemical unit is planar, or nearly planar in the solid state, and exhibits high symmetry, often close to D_{3h}. Furthermore, it is a potential donor of six H atoms. Under certain conditions, these properties can be used for controlling crystal structure on the molecular level (Russell, Etter & Ward, 1994). Crystals built of two-dimensional motifs seem to be the most attractive; the role of hydrogen bonding and other electrostatic interactions in such structures is especially noteworthy (Katrusiak & Szafrański, 1996). Numerous investigations indicate that crystals based on molecular cations and metal–halide anions show a strong tendency towards formation of two-dimensional anionic sublattices. The type of sublattice depends, among other things, on the stoichiometry of the crystal, which can often be modified in a simple way by changing the molar ratio of ingredients in solution or the thermodynamic conditions of crystal growth. In this work, we present the crystal structure of $[C(NH_2)_3]^+[Hg_2Cl_5]^-$, (I), in which the molar ratio of $C(NH_2)_3Cl$ and $HgCl_2$ is 1:2.



(I)

The coordination numbers of the Hg^{2+} ion range from two to six, a consequence of its large volume and spherical charge distribution, as it has complete 4*f* and 5*d* electron shells. The most frequently occurring coordination is a linear one, with a coordination number (CN) of 2. The octahedral coordination (CN = 6) is often encountered in halides and it is typically formed by four weak interactions with the central Hg atom of the almost linear HgX_2 unit. For chlorides, it has been pointed out (Ben Salah, Bats, Fuess & Daoud, 1983) that the distortion of the $HgCl_2$ pseudo-molecule compared with the geometry of the uncomplexed $HgCl_2$ molecule, depends on the number of weak Hg—Cl interactions. The Hg—Cl distances are in the range 2.25–2.40 Å for short covalent bonds and 2.7–3.5 Å for weak interactions. For the 'free' uncomplexed $HgCl_2$ molecule, the Hg—Cl bond distances of 2.28–2.31 Å [for example, crystalline $HgCl_2$ (Subramanian & Seff, 1980), and the 3:2 complex of $HgCl_2$ and thiourea (Brotherton & White, 1973)] are only slightly longer than 2.252 (5) Å for gaseous $HgCl_2$ (Kashiwabara, Konaka & Kimura, 1973), and the Cl—Hg—Cl angle is equal or very close to the ideal value of 180°.

The coordination polyhedron of mercury in the title compound can be described as a distorted octahedron, with two short [2.306 (5) and 2.322 (4) Å], two long [2.905 (7) and 2.906 (7) Å] and two very long [3.271 (6) and 3.344 (6) Å] Hg—Cl bonds. As expected, the $HgCl_2$ unit is distorted by the presence of the longer Hg—Cl contact; the short covalent Hg—Cl bonds are significantly longer than for gaseous or uncomplexed $HgCl_2$, and the angle between the short bonds is much smaller than 180° [163.6 (2)°], *i.e.* the $HgCl_2$ pseudo-molecule is bent. The $Hg_2Cl_5^-$ anion could therefore be considered as formed by two $HgCl_2$ molecules bridged by a Cl^- anion ($Cl1^-$), which is located almost equidistant from both Hg atoms. A similar arrangement has been observed in a number of previously described complexes, for example, in $[(CH_3)_2NH_2]^+Hg_2Cl_5^-$ (Ben Salah, Bats, Fuess & Daoud, 1982) and in chloro-*N*,*N*,*N*′,*N*′-tetramethylformamidinium bis[dichloromercury(II)] chloride (Aharoni, Kapon & Reisner, 1989). However, in the present case, the Cl1 atom lies at almost equal distances from four Hg atoms (also as a consequence of the relatively high internal symmetry of the complex), with a nearly ideal square-planar disposition of the Hg atoms. Therefore, it should probably be more appropriate to describe the anion as either $2n(HgCl_2).nCl^-$ or $(Hg_2Cl_5)_n^{n-}$. These polyanions expand along the [100] direction and the neighboring polyanions, related by a $[\frac{1}{2},\frac{1}{2},0]$ translation vector, are connected by very long Hg—Cl contacts. So, in the crystal structure, there are layers of polyanions parallel to the (001) plane (Fig. 1), around $z = 0$ and $z = \frac{1}{2}$, and between these negatively charged layers there are positively charged layers of guanidinium cations, at $z = \frac{1}{4}$ and $z = \frac{3}{4}$. Such an arrangement of the ionic species results in electrostatic attraction between adjacent layers, which strongly stabilizes the structure of the crystal. The three-dimensional network of N—H···Cl hydrogen bonds is the other factor stabilizing the crystal. This network is highly symmetric due to the internal symmetry of both cations and anions. Every H atom takes part in one strong and one weak hydrogen bond. H atoms from the N1 amino group are hydrogen bonded to successive Cl atoms from one polyanion, while those from N2 connect two neighboring polyanions in the same layer (Fig. 2).

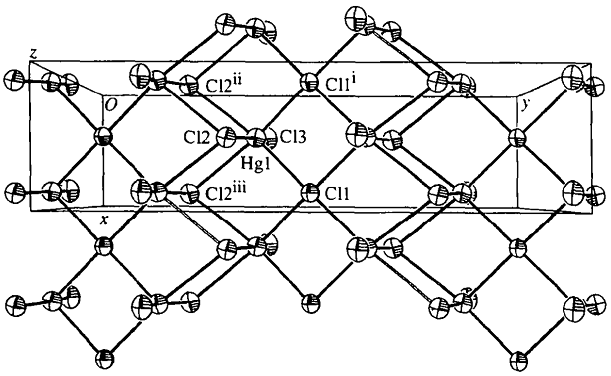


Fig. 1. The displacement ellipsoid view, perpendicular to the [001] direction, of a fragment of the polyanionic layer. The ellipsoids are drawn at the 50% probability level. Very long Hg—Cl contacts are drawn as open bonds. [Symmetry codes: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, -z$; (iii) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.]

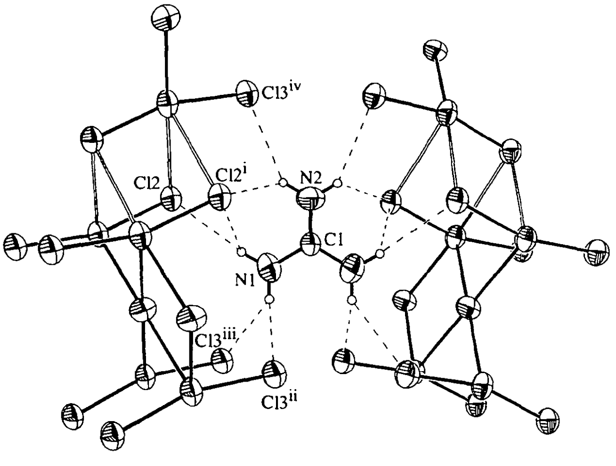


Fig. 2. The scheme of the hydrogen bonds between the cation and anions, as seen approximately perpendicular to the (100) plane. Displacement ellipsoids are drawn at the 50% probability level, with H atoms as spheres of arbitrary radii. Hydrogen bonds are depicted as dashed lines and very long Hg—Cl contacts as open bonds.

Differential thermal analysis performed in the temperature range between 100 and 400 K did not reveal any transformation of the structure of the title compound. This result is in agreement with the structural properties of the crystal.

Experimental

Crystals suitable for X-ray analysis were grown from an aqueous solution containing $C(NH_2)_3$ and $HgCl_2$ in the molar ratio 2:1. The solution was acidified by the addition of HCl and left to evaporate slowly at room temperature. The stoichiometry of the crystal was found to be the reverse of that of the solution.

Crystal data

$(CH_6N_3)[Hg_2Cl_5]$
$M_r = 638.52$
Orthorhombic
$C222_1$
$a = 4.0367(10)$ Å
$b = 14.956(3)$ Å
$c = 18.404(3)$ Å
$V = 1111.1(4)$ Å^3
$Z = 4$
$D_x = 3.817$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 44 reflections
$\theta = 4.30$–$20.28°$
$\mu = 28.751$ mm^{-1}
$T = 293(2)$ K
Block
$0.40 \times 0.08 \times 0.08$ mm
Colorless

Data collection

Kuma KM-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.041$, $T_{max} = 0.100$
938 measured reflections
938 independent reflections

752 reflections with $I > 2\sigma(I)$
$\theta_{max} = 30.06°$
$h = 0 \rightarrow 5$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 25$
3 standard reflections every 100 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.188$
$S = 1.087$
938 reflections
53 parameters
H-atom parameters constrained to parent site
$w = 1/[\sigma^2(F_o^2) + (0.1404P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 4.46$ e Å^{-3}
$\Delta\rho_{min} = -3.28$ e Å^{-3}
Extinction correction: *SHELXL93*
Extinction coefficient: 0.0023 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.18 (6)

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}
Hg1	0.3705 (3)	0.36785 (5)	−0.03689 (4)	0.0572 (4)
Cl1	0.871 (2)	1/2	0	0.0576 (13)
Cl2	0.385 (2)	0.3003 (3)	0.0767 (2)	0.0582 (10)
Cl3	0.364 (2)	0.3931 (4)	−0.1605 (3)	0.0664 (12)
C1	1	0.3847 (17)	1/4	0.050 (5)
N1	0.883 (8)	0.4270 (13)	0.1942 (10)	0.071 (5)
N2	1	0.298 (2)	1/4	0.086 (12)

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

Hg1—Cl3	2.306 (5)	Hg1—Cl1	2.906 (7)
Hg1—Cl2	2.322 (4)	Hg1—Cl2^{ii}	3.271 (6)
Hg1—Cl1^{i}	2.905 (7)	Hg1—Cl2^{iii}	3.344 (6)

Cl3—Hg1—Cl2	163.6 (2)	Cl1$^{\rm i}$—Hg1—Cl2$^{\rm ii}$	99.2 (2)
Cl3—Hg1—Cl1$^{\rm i}$	96.4 (2)	Cl1—Hg1—Cl2$^{\rm iii}$	97.5 (2)
Cl2—Hg1—Cl1$^{\rm i}$	95.9 (2)	Cl2—Hg1—Cl2$^{\rm iii}$	81.6 (2)
Cl3—Hg1—Cl1	97.3 (2)	Cl3—Hg1—Cl2$^{\rm iii}$	85.1 (2)
Cl2—Hg1—Cl1	93.9 (2)	Cl1$^{\rm i}$—Hg1—Cl2$^{\rm iii}$	174.1 (2)
Cl1—Hg1—Cl1$^{\rm i}$	88.0 (2)	Cl2$^{\rm ii}$—Hg1—Cl2$^{\rm iii}$	75.3 (2)
Cl1—Hg1—Cl2$^{\rm ii}$	172.5 (2)	Hg1$^{\rm iv}$—Cl1—Hg1	180.0 (3)
Cl2—Hg1—Cl2$^{\rm ii}$	83.3 (2)	Hg1$^{\rm v}$—Cl1—Hg1	88.0 (2)
Cl3—Hg1—Cl2$^{\rm ii}$	84.2 (2)	Hg1—Cl1—Hg1$^{\rm vi}$	92.0 (3)

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

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

D—H···A	H···A	D···A	D—H···A
N1—H1A···Cl2	2.69	3.508 (3)	159
N1—H1A···Cl2$^{\rm i}$	3.13	3.517 (3)	111
N1—H1B···Cl3$^{\rm ii}$	2.74	3.374 (3)	132
N1—H1B···Cl3$^{\rm iii}$	2.85	3.466 (3)	131
N2—H2A···Cl3$^{\rm iv}$	2.63	3.346 (3)	142
N2—H2A···Cl2$^{\rm i}$	3.15	3.548 (5)	111

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

The largest peaks and holes in the ΔF Fourier map are within 1.0 Å of the Hg1 atom site.

Data collection: *Kuma KM*-4 *Users Guide* (Kuma Diffraction, 1991). Cell refinement: *Kuma KM*-4 *Users Guide*. Data reduction: *Kuma KM*-4 *Users Guide*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *Stereochemical Workstation* (Siemens, 1989). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1294). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1561–1564

Bis(*N*,*N*-dimethyl-1,3-propanediamine)bis(thiocyanato-*N*)nickel(II), NiL_2(NCS)$_2$, and its Solid-State Isomer [L = NH$_2$(CH$_2$)$_3$N(CH$_3$)$_2$]

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(*Received 6 December 1996; accepted 17 April 1997*)

Abstract

The coordination polyhedron about the Ni$^{\rm II}$ atom in NiL_2(NCS)$_2$, (1), where L = NH$_2$(CH$_2$)$_3$N(CH$_3$)$_2$, is a distorted octahedron with the two thiocyanate N and the two unsubstituted N atoms of the ligand defining the equatorial plane; the two dimethylated N atoms occupy *trans*-axial positions. The six-membered chelate rings are in stable chair conformations. Complex (1), [Ni(C$_5$H$_{14}$N$_2$)$_2$(NCS)$_2$], undergoes a thermochromic solid-state phase transition producing an isomer, (2). With the cell volume of (2) approximately double that of (1) and the crystal system remaining triclinic, the space group of the isomer (2) is $P1$ or $P\bar{1}$.

Comment

Thermally induced solid-state phase transitions in nickel(II)–diamine complexes are documented in the literature (Mukherjee, Mukherjee, Ray, Ghosh & Ray Chaudhuri, 1990; Ihara, Satake, Suzuki & Uehara, 1991; Koner *et al.*, 1995; Pariya *et al.*, 1996). The probable mechanism of the phase transition in these complexes is mostly associated with the changes in the geometry of the metal coordination and/or the conformation of the diamine chelate rings (Ihara, Fukuda & Sone, 1987; Roy, Ghosh & Ray Chaudhuri, 1989; Pariya *et al.*, 1995). The present work was undertaken as part of our ongoing program studying the effect of diamine substituents on phase transition and to construct a hierarchy. A