

Bis(ethylenethiourea)gold(I) Chloride Hydrate

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Abstract. $C_6H_{14}ON_4ClS_2Au$, triclinic, $P\bar{1}$, $a=8.828$ (6), $b=9.017$ (4), $c=10.414$ (6) Å, $\alpha=114.63$ (4), $\beta=92.14$ (6), $\gamma=116.05$ (4)°, $U=651.9$ Å³, $Z=2$, $D_x=2.317$ g cm⁻³, $\mu=113.3$ cm⁻¹ (Mo $K\alpha$). The structure consists of etu_2Au^+ cations (etu =ethylenethiourea) somewhat distorted from the expected linear coordination, with uncoordinated Cl^- anions; the water molecule forms a hydrogen-bonding bridge between the two *cis* etu rings. The structure was refined to an R of 0.0844 for 2388 independent observed reflexions.

Introduction. The structure of $etu_2AuCl \cdot H_2O$ has been determined as part of a study of coordination number in Au^I compounds.

Colourless crystals were obtained as described by Morgan & Burstall (1928) and were stable to X-rays. Intensities were determined on a Syntex $P2_1$ four-circle diffractometer with Mo $K\alpha$ radiation, a graphite monochromator, and a crystal $0.1 \times 0.15 \times 0.18$ mm. 3012 reflexions were measured in the range $2^\circ < 2\theta < 55^\circ$; of these, 2388 unique reflexions with $F > 4\sigma(F)$ were used in the structure determination. L_p and numerical absorption corrections were made. The cell used was both the Dirichlet and Delaunay cell. No cell of higher symmetry could be found with the lattice geometry program *XCELL*.

The non-hydrogen atoms were located from Patterson and difference syntheses. C–H distances were fixed at 1.08 Å, N–H distances at 0.98 Å, H–C–H angles at 109.5° and H-atom temperature factors at 0.1 Å². The H atoms of water were not located.

The final $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o|$ was 0.0854, with a corresponding R of 0.0844; the weighting scheme was $w = 1/[\sigma^2(F) + 0.01F^2]$ which gave a mean $w\Delta^2$ varying only slightly with $\sin \theta$ or $|F_o|$. Complex neutral atom scattering factors were used. An empirical isotropic extinction parameter x was included in the refinement; it refined to 433 (8) $\times 10^{-9}$ (where F_c is multiplied by $[1 - x|F_c|^2/\sin \theta]$). Final atomic coordinates and temperature factors are given in Tables 1–3, with derived bond lengths, bond angles and non-bonded distances in Tables 4–6.*

Table 1. Atom coordinates ($\times 10^4$) and isotropic temperature factors (Å² $\times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
C(11)	3976 (19)	2204 (20)	−684 (15)	40 (3)
C(12)	4358 (24)	3075 (26)	−2471 (19)	54 (4)
C(13)	6095 (24)	3483 (26)	−1715 (19)	55 (4)
C(21)	8239 (21)	2247 (22)	4097 (17)	45 (3)
C(22)	10941 (26)	2650 (29)	4807 (20)	61 (4)
C(23)	10713 (26)	2788 (30)	3375 (21)	63 (4)
N(11)	3152 (20)	2211 (20)	−1742 (15)	53 (3)
N(12)	5624 (17)	2888 (18)	−608 (14)	45 (3)
N(21)	9186 (21)	2088 (23)	4965 (17)	59 (4)
N(22)	9127 (19)	2796 (20)	3226 (15)	51 (3)
S(1)	2892 (5)	1265 (7)	367 (5)	
S(2)	6178 (7)	1872 (9)	4193 (5)	
O(1)	8829 (15)	3775 (16)	1057 (12)	49 (2)
Cl(1)	9361 (5)	1859 (6)	7927 (4)	
Au(1)	4809 (1)	1705 (1)	2198 (1)	

Table 2. Anisotropic temperature factors (Å² $\times 10^3$)

The temperature factor exponent takes the form:
 $-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^*)$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S(1)	39 (2)	64 (3)	58 (2)	35 (2)	18 (2)	18 (2)
S(2)	64 (3)	86 (3)	54 (2)	43 (2)	26 (2)	35 (3)
Cl(1)	47 (2)	57 (2)	54 (2)	32 (2)	20 (2)	31 (2)
Au(1)	46 (1)	39 (1)	45 (1)	20 (1)	14 (1)	18 (1)

Table 3. Hydrogen atom coordinates ($\times 10^4$)

	<i>x</i>	<i>y</i>	<i>z</i>
H(11)	3988	2115	−3631
H(12)	4401	4358	−2311
H(13)	7057	4964	−1214
H(14)	6586	2670	−2469
H(21)	11896	3989	5725
H(22)	11299	1601	4669
H(23)	10631	1588	2445
H(24)	11786	4068	3483
H(15)	1926	1788	−2139
H(16)	6624	3095	29
H(25)	8967	1689	5715
H(26)	8900	3145	2497

Table 4. Bond lengths (Å)

C(11)–N(11)	1.302 (28)	C(11)–N(12)	1.291 (22)
C(11)–S(1)	1.723 (20)	C(12)–C(13)	1.510 (34)
C(12)–N(11)	1.463 (28)	C(13)–N(12)	1.465 (30)
C(21)–N(21)	1.301 (33)	C(21)–N(22)	1.330 (26)
C(21)–S(2)	1.713 (22)	C(22)–C(23)	1.560 (38)
C(22)–N(21)	1.444 (29)	C(23)–N(22)	1.407 (33)
N(11)–C(12)	1.463 (28)	S(1)–Au(1)	2.279 (8)
S(2)–Au(1)	2.278 (9)		

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31928 (16 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

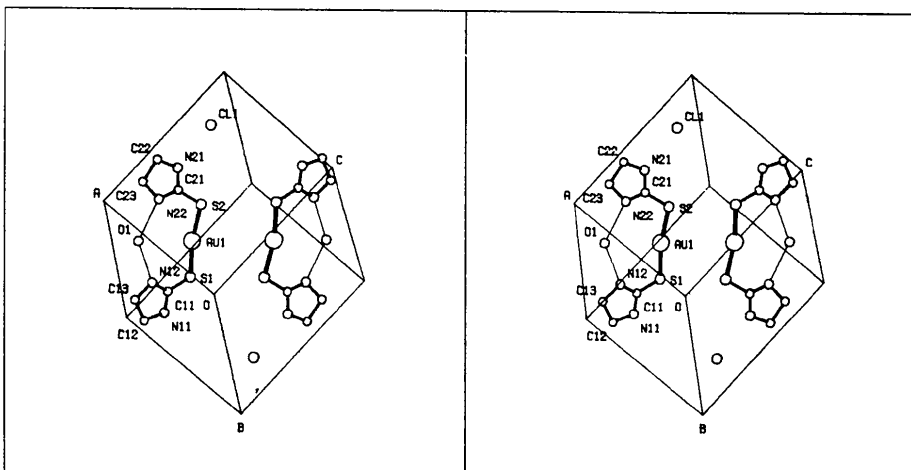


Fig. 1. Stereo pair of the unit-cell contents, showing H-bonding contacts between O and N atoms.

Table 5. Bond angles ($^{\circ}$)

N(11)—C(11)—N(12)	111.1 (18)	N(11)—C(11)—S(1)	121.9 (14)
N(12)—C(11)—S(1)	126.9 (17)	C(13)—C(12)—N(11)	103.0 (20)
C(12)—C(13)—N(12)	101.7 (17)	N(21)—C(21)—N(22)	110.5 (19)
N(21)—C(21)—S(2)	122.0 (16)	N(22)—C(21)—S(2)	127.4 (19)
C(23)—C(22)—N(21)	99.3 (20)	C(22)—C(23)—N(22)	103.8 (19)
C(11)—N(11)—C(12)	111.4 (17)	C(11)—N(12)—C(13)	112.6 (18)
C(21)—N(21)—C(22)	113.2 (21)	C(21)—N(22)—C(23)	110.7 (21)
C(11)—S(1)—Au(1)	111.2 (7)	C(21)—S(2)—Au(1)	109.8 (8)
S(1)—Au(1)—S(2)	167.1 (2)		

Table 6. Selected non-bonded distances (\AA)

O(1)—N(22)	2.79	O(1)—N(12)	2.85
Cl(1)—N(21)	3.18	Cl(1)—N(11a)	3.21
Au(1)—Au(1b)	4.45	Au(1)—Cl(1c)	3.62

Key to symmetry operations relating designated atoms to reference atoms at x, y, z : (a) $1.0 + x, y, 1.0 + z$; (b) $1.0 - x, -y, -z$; (c) $1.0 - x, -y, 1.0 - z$.

Discussion. Au^{I} forms several complexes of the form L_2AuCl which may be either three-coordinate or ionic. $(\text{Ph}_3\text{P})_2\text{AuCl}$ has been shown to be three-coordinate (Baenziger, Dittmore & Doyle, 1974). $\text{Et}_2\text{AuCl} \cdot \text{H}_2\text{O}$ was originally formulated as ionic since it is water soluble, and this investigation has confirmed its ionic nature.

The etu_2Au^+ cation would be expected to show linear coordination as does $(\text{Ph}_2\text{MeP})_2\text{Au}^+$ (Guy, Jones & Sheldrick, 1976) but is significantly distorted, with a S—Au—S angle of 167.1° . Au^{I} compounds can show distorted two-coordination; $\text{Ph}_3\text{PAu}(\text{S}_2\text{CNET}_2)$ is basically linear with monodentate thiocarbamate (Wijnhoven, Bosman & Beurskens, 1972), its small distortion from linearity (the P—Au—S angle is 175.7°) presumably caused by a weak interaction between the Au atom and the second S atom of the dithiocarbamate (Au—S = 3.02 \AA). This is a limiting case of three-coordination, as discussed by Clegg (1976). In etu_2AuCl , the Au—Cl distance is 3.62 \AA and any interaction must be far too weak to cause the significant distortion. We attribute the distortion, and the unexpected *cis* configuration of

the ligands, to the effect of H-bonding between the O atom of water and the N—H groups on the rings (Fig. 1); the distances O(1)—N(12) and O(1)—N(22) are typical H-bonded N—O distances. The Cl^- ions may also be involved in H-bonding with N—H groups since the distances Cl(1)—N(21) and Cl(1)—N(11a) are quite short (for non-bonded distances see Table 6).

The Au—S distances of $2.278 (9)$ and $2.279 (8) \text{ \AA}$ are very similar to Au—S distances in $(\text{C}_3\text{H}_7)_2\text{NCS}_2\text{Au}$ dimer [$2.28 (2) \text{ \AA}$; Hesse & Jennische, 1972], $(\text{C}_4\text{H}_9)_2\text{NCS}_2\text{Au}$ dimer [$2.28 (1) \text{ \AA}$; Jennische, 1976] and $\text{Ph}_3\text{PAu}(\text{S}_2\text{CNET}_2)$ [$2.338 (3) \text{ \AA}$; Wijnhoven *et al.*, 1972]. There are no short Au—Au contacts typical of many Au^{I} compounds, e.g. 3.30 \AA in (piperidine) AuCl (Guy, Jones, Mays & Sheldrick, 1976).

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