

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U <sub>eq</sub>
I1	0.3005 (1)	0.7313 (1)	0.0456 (2)	0.050 (1)
I2	0.5668 (1)	0.7759 (1)	0.3699 (2)	0.054 (1)
I3	0.4424 (1)	0.6581 (1)	0.4338 (2)	0.062 (1)
I4	0.1389 (1)	0.4202 (1)	0.2899 (2)	0.080 (1)
I5	0.0553 (1)	0.5482 (1)	0.3552 (2)	0.084 (1)
Cu1	0.4753 (2)	0.7733 (1)	0.0948 (4)	0.095 (1)
Cu2	0.3996 (2)	0.7311 (1)	0.3167 (3)	0.081 (1)
Cu3	0.0496 (2)	0.4728 (1)	0.4241 (4)	0.078 (1)
Na1	0.0670 (5)	0.3534 (2)	0.5288 (10)	0.062 (2)
O11	0.1873 (9)	0.3045 (5)	0.4837 (17)	0.068 (4)
O12	0.1988 (9)	0.3497 (4)	0.7444 (17)	0.062 (4)
O13	0.0158 (9)	0.3326 (5)	0.7858 (17)	0.066 (4)
O14	-0.1054 (8)	0.3446 (4)	0.5125 (18)	0.061 (4)
O15	0.0088 (10)	0.3080 (5)	0.3055 (17)	0.075 (4)
C10	0.2735 (13)	0.3228 (7)	0.5289 (28)	0.072 (7)
C11	0.2785 (15)	0.3286 (7)	0.6991 (24)	0.074 (7)
C12	0.1773 (15)	0.3420 (8)	0.8952 (27)	0.077 (7)
C13	0.0733 (14)	0.3553 (7)	0.9056 (25)	0.070 (7)
C14	-0.0844 (12)	0.3385 (7)	0.7877 (27)	0.069 (6)
C15	-0.1350 (14)	0.3210 (7)	0.6404 (28)	0.069 (7)
C16	-0.1411 (14)	0.3313 (8)	0.3600 (30)	0.082 (8)
C17	-0.0817 (13)	0.2947 (7)	0.3001 (26)	0.069 (7)
C18	0.0794 (14)	0.2783 (7)	0.2681 (30)	0.075 (7)
C19	0.1758 (14)	0.2942 (8)	0.3092 (25)	0.072 (7)
Na2	0.3481 (6)	0.5424 (3)	0.7462 (10)	0.072 (2)
O21	0.2072 (15)	0.5076 (7)	0.8119 (24)	0.108 (6)
O22	0.2550 (22)	0.5826 (8)	0.9020 (32)	0.155 (10)
O23	0.4495 (12)	0.5851 (7)	0.9163 (23)	0.108 (6)
O24	0.5151 (15)	0.5293 (8)	0.7248 (25)	0.121 (8)
O25	0.3762 (15)	0.4707 (6)	0.7778 (26)	0.112 (6)
O31	0.2999 (11)	0.5693 (7)	0.5004 (21)	0.115 (7)
C20	0.1500 (31)	0.5250 (18)	0.9003 (69)	0.223 (27)
C21	0.1693 (26)	0.5643 (17)	0.9442 (62)	0.222 (30)
C22	0.3013 (26)	0.6043 (12)	1.0127 (40)	0.128 (13)
C23	0.4034 (29)	0.6189 (8)	0.9566 (34)	0.135 (15)
C24	0.5337 (21)	0.5931 (11)	0.8510 (42)	0.126 (13)
C25	0.5802 (24)	0.5514 (17)	0.8106 (48)	0.190 (24)
C26	0.5373 (25)	0.4878 (11)	0.7388 (43)	0.131 (12)
C27	0.4634 (28)	0.4641 (10)	0.6853 (45)	0.140 (14)
C28	0.2962 (32)	0.4507 (12)	0.7405 (55)	0.206 (24)
C29	0.2165 (22)	0.4656 (12)	0.8151 (55)	0.165 (18)

Table 2. Selected geometric parameters (Å)

I1—Cu2	2.545 (3)	I4—Cu3	2.484 (3)
I1—Cu2 <sup>i</sup>	2.787 (3)	I4...Na1	3.220 (8)
I1—Cu1	2.811 (3)	I5—Cu3	2.557 (4)
I2—Cu1	2.530 (3)	I5—Cu3 <sup>iii</sup>	2.587 (3)
I2—Cu2	2.771 (3)	Cu1...Cu2 <sup>i</sup>	2.472 (4)
I2—Cu1 <sup>ii</sup>	2.897 (4)	Cu1...Cu2	2.643 (4)
I3—Cu2	2.649 (4)	Cu3...Cu3 <sup>iii</sup>	2.678 (6)
I3—Cu1 <sup>ii</sup>	2.655 (4)		

Symmetry codes: (i)  $x, \frac{3}{2}-y, z-\frac{1}{2}$ ; (ii)  $x, \frac{3}{2}-y, \frac{1}{2}+z$ ; (iii)  $-x, 1-y, 1-z$ .

Data collection, cell refinement and data reduction: Siemens XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXS86. Software used to prepare material for publication: SHELXL93. Extinction effects were observed to be negligible. Absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Table 4.2.4.2).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Andersson, S. & Jagner, S. (1985a). *Acta Chem. Scand. Ser. A*, **39**, 181–186.
- Andersson, S. & Jagner, S. (1985b). *Acta Chem. Scand. Ser. A*, **39**, 423–428.
- Andersson, S. & Jagner, S. (1987a). *Acta Chem. Scand. Ser. A*, **41**, 230–236.
- Andersson, S. & Jagner, S. (1987b). *Acta Chem. Scand. Ser. C*, **43**, 1089–1091.
- Asplund, M. & Jagner, S. (1984a). *Acta Chem. Scand. Ser. A*, **38**, 135–139.
- Asplund, M. & Jagner, S. (1984b). *Acta Chem. Scand. Ser. A*, **38**, 297–301.
- Asplund, M., Jagner, S. & Nilsson, M. (1982). *Acta Chem. Scand.* **A36**, 751–755.
- Banci, L., Bencini, A., Dei, A. & Gatteschi, D. (1984). *Inorg. Chim. Acta*, L11–L12.
- Batsanov, A. S., Struchkov, Yu. T., Ukhin, L. Yu. & Dolgoplova, N. A. (1982). *Inorg. Chim. Acta*, **63**, 17–22.
- Hartl, H., Brudgam, I. & Mahjour-Hassan-Abadi, F. (1985). *Z. Naturforsch. Teil B*, **40**, 1032–1039.
- Hartl, H. & Mahjour-Hassan-Abadi, F. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 772–773.
- Jagner, S. & Helgesson, G. (1991). *Adv. Inorg. Chem.* **37**, 1–41.
- Rath, N. P. & Holt, E. M. (1985). *J. Chem. Soc. Chem. Commun.* pp. 665–667.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. Univ. of Göttingen, Germany.
- Shibaeva, R. P. & Kaminskii, V. F. (1981). *Sov. Phys. Crystallogr.* **26**, 188–190.

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## 3,5-Diphenyl-1,2-dithiolium Diiodoauride(I)

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## Abstract

The title complex, (C<sub>15</sub>H<sub>11</sub>S<sub>2</sub>)[AuI<sub>2</sub>], consists of a 3,5-diphenyldithiolium cation and a diiodoauride anion. The Au<sup>I</sup> center is linearly coordinated by two I atoms.

**Comment**

A view of the cation and the anion is shown in Fig. 1. The structure of the anion  $\text{AuI}_2^-$  is similar to those of the previously reported compounds  $[\text{Au}(\text{py})_2][\text{AuI}_2]$  (Adams, Hiller & Strahle, 1982) and  $[\text{N}(\text{n-C}_4\text{H}_9)_4][\text{AuI}_2]$  (Braunstein, Muller & Bogge, 1986).

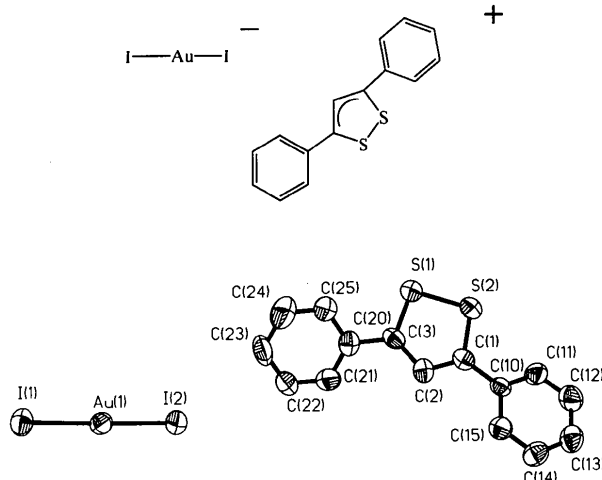


Fig. 1. A view of the molecule of  $(\text{C}_{15}\text{H}_{11}\text{S}_2)[\text{AuI}_2]$ . Displacement ellipsoids have been drawn at the 50% probability level. H atoms have been omitted for clarity.

**Experimental**

The title compound was obtained by the reaction of  $(\text{C}_{15}\text{H}_{11}\text{S}_2)\text{I}$  (Hendrickson & Martin, 1973) with  $[\text{Au}(\text{tht})\text{Cl}]$  (tht = tetrahydrothiophene) (Uson, Laguna & Laguna, 1989) in  $\text{CH}_2\text{Cl}_2$  solution. Crystals suitable for X-ray analysis were obtained by recrystallization from a dichloromethane–diethyl ether solution.

**Crystal data**

$(\text{C}_{15}\text{H}_{11}\text{S}_2)[\text{AuI}_2]$   
 $M_r = 706.06$   
 Monoclinic  
 $P2_1/c$   
 $a = 7.5238 (8) \text{ \AA}$   
 $b = 22.525 (4) \text{ \AA}$   
 $c = 10.628 (1) \text{ \AA}$   
 $\beta = 95.594 (9)^\circ$   
 $V = 1792.6 (4) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 2.62 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 12.25\text{--}16^\circ$   
 $\mu = 11.82 \text{ mm}^{-1}$   
 $T = 298 \text{ K}$   
 Rectangular  
 $0.40 \times 0.30 \times 0.20 \text{ mm}$   
 Red

**Data collection**

R3m/E diffractometer  
 $\omega$  scans  
 Absorption correction: empirical  
 $T_{\min} = 0.620$ ,  $T_{\max} = 0.978$   
 2672 measured reflections  
 2638 independent reflections  
 1666 observed reflections  
 $[F_o^2 > 3\sigma(F_o^2)]$

$R_{\text{int}} = 0.0266$   
 $\theta_{\text{max}} = 22.7^\circ$   
 $h = 0 \rightarrow 9$   
 $k = 0 \rightarrow 25$   
 $l = -12 \rightarrow 12$   
 3 standard reflections monitored every 97 reflections  
 intensity variation: none

**Refinement**

Refinement on  $F$   
 $R = 0.0307$   
 $wR = 0.0336$   
 $S = 1.010$   
 1666 reflections  
 181 parameters  
 H-atom parameters not refined  
 $w = 1/[\sigma^2(F_o) + 0.00100F_o^2]$

$(\Delta/\sigma)_{\text{max}} = 0.003$   
 $\Delta\rho_{\text{max}} = 0.61 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.86 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$U_{\text{eq}}$
Au(1)	0.2494 (1)	-0.0497 (1)	0.1794 (1)	0.057 (1)
I(1)	0.2897 (1)	0.0576 (1)	0.2486 (1)	0.066 (1)
I(2)	0.2097 (1)	-0.1563 (1)	0.1047 (1)	0.070 (1)
S(1)	0.7160 (4)	-0.4135 (1)	-0.3881 (3)	0.059 (1)
S(2)	0.7723 (4)	-0.5013 (1)	-0.3781 (3)	0.057 (1)
C(1)	0.7672 (13)	-0.5066 (4)	-0.2173 (9)	0.048 (4)
C(2)	0.7316 (14)	-0.4552 (4)	-0.1561 (10)	0.049 (4)
C(3)	0.7111 (14)	-0.4038 (4)	-0.2277 (10)	0.049 (4)
C(10)	0.8043 (15)	-0.5654 (4)	-0.1617 (9)	0.048 (4)
C(11)	0.8042 (16)	-0.6156 (5)	-0.2340 (10)	0.060 (4)
C(12)	0.8473 (17)	-0.6697 (5)	-0.1828 (11)	0.068 (5)
C(13)	0.8892 (16)	-0.6746 (5)	-0.0520 (12)	0.067 (5)
C(14)	0.8841 (15)	-0.6250 (5)	0.0235 (11)	0.062 (4)
C(15)	0.8407 (15)	-0.5717 (5)	-0.0303 (11)	0.057 (4)
C(20)	0.6752 (14)	-0.3440 (4)	-0.1804 (11)	0.051 (4)
C(21)	0.5788 (16)	-0.3354 (5)	-0.0803 (11)	0.062 (5)
C(22)	0.5462 (18)	-0.2796 (5)	-0.0391 (12)	0.071 (5)
C(23)	0.6013 (18)	-0.2302 (5)	-0.0993 (12)	0.078 (5)
C(24)	0.6974 (18)	-0.2391 (6)	-0.2023 (14)	0.091 (6)
C(25)	0.7314 (16)	-0.2939 (5)	-0.2438 (12)	0.063 (5)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Au(1)—I(1)	2.536 (1)	Au(1)—I(2)	2.538 (1)
S(1)—S(2)	2.024 (4)	S(1)—C(3)	1.723 (11)
S(2)—C(1)	1.717 (10)	C(1)—C(2)	1.368 (14)
C(1)—C(10)	1.467 (14)	C(2)—C(3)	1.385 (14)
C(3)—C(20)	1.472 (14)	C(10)—C(11)	1.367 (15)
C(10)—C(15)	1.404 (15)	C(11)—C(12)	1.360 (15)
C(12)—C(13)	1.400 (17)	C(13)—C(14)	1.378 (16)
C(14)—C(15)	1.354 (15)	C(20)—C(21)	1.359 (17)
C(20)—C(25)	1.400 (15)	C(21)—C(22)	1.361 (17)
C(22)—C(23)	1.369 (18)	C(23)—C(24)	1.385 (20)
C(24)—C(25)	1.344 (17)		
I(1)—Au(1)—I(2)	178.6	S(2)—S(1)—C(3)	95.5 (4)
S(1)—S(2)—C(1)	95.4 (4)	S(2)—C(1)—C(2)	116.1 (8)
S(2)—C(1)—C(10)	116.2 (7)	C(2)—C(1)—C(10)	127.7 (9)
C(1)—C(2)—C(3)	117.6 (10)	S(1)—C(3)—C(2)	115.2 (8)
S(1)—C(3)—C(20)	118.4 (8)	C(2)—C(3)—C(20)	126.3 (10)
C(1)—C(10)—C(11)	122.1 (9)	C(1)—C(10)—C(15)	120.2 (9)
C(11)—C(10)—C(15)	117.7 (9)	C(10)—C(11)—C(12)	121.9 (10)
C(11)—C(12)—C(13)	119.3 (10)	C(12)—C(13)—C(14)	119.9 (10)
C(13)—C(14)—C(15)	119.5 (11)	C(10)—C(15)—C(14)	121.6 (10)
C(3)—C(20)—C(21)	121.8 (10)	C(3)—C(20)—C(25)	119.9 (10)
C(21)—C(20)—C(25)	118.2 (10)	C(20)—C(21)—C(22)	120.7 (11)
C(21)—C(22)—C(23)	121.9 (12)	C(22)—C(23)—C(24)	117.2 (12)
C(23)—C(24)—C(25)	121.6 (12)	C(20)—C(25)—C(24)	120.3 (12)

The crystal was mounted in a random orientation on a glass fiber. Axial dimensions and triclinic symmetry were verified by axial rotation photographs and Delaunay reduction. A variable scan rate was used,  $2.80\text{--}29.0^\circ \text{ min}^{-1}$ , in a scan range of  $-0.8^\circ$  in  $x$  from  $K\alpha_1$  to  $0.8^\circ$  from  $K\alpha_2$ . Background intensities were estimated from a 96-step peak profile. Lorentz and polarization corrections were applied. Structure determination

was carried out using *SHELXTL* (Sheldrick, 1985). The Au-atom position was determined by direct methods and the remaining non-H atoms were located using difference Fourier techniques. All non-H atoms were refined anisotropically and H atoms were placed in idealized positions with fixed isotropic displacement parameters [ $U(H) = 0.08 \text{ \AA}^2$ ].

These studies were supported by the Welch Foundation and the National Science Foundation (grant CHE-8708625).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: ST1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Adams, H.-N., Hiller, W. & Strahle, J. (1982). *Z. Anorg. Allg. Chem.* **485**, 81–91.  
 Braunstein, P., Muller, A. & Bogge, H. (1986). *Inorg. Chem.* **25**, 2104–2106.  
 Hendrickson, A. R. & Martin, R. L. (1973). *J. Org. Chem.* **38**, 2548–2549.  
 Sheldrick, G. M. (1985). *SHELXTL User's Manual*. Revision 5.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.  
 Uson, R., Laguna, A. & Laguna, M. (1989). *Inorg. Synth.* **26**, 85–91.

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## Bis(tetraethylammonium) Tetrabromocadmate

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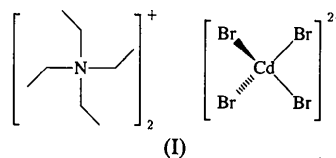
### Abstract

The crystal structure of the title compound,  $[(C_2H_5)_4N]_2[CdBr_4]$ , has been determined by single-crystal X-ray diffraction methods. The crystal structure consists of isolated  $CdBr_4$  tetrahedra, two cations in a swastika conformation (one ordered, one disordered) and one cation in a *trans* conformation. All cations show strong thermal motion. Automatic structure solution packages failed to yield a chemically relevant result. The structure solution by manual interpretation of the Patterson map is reported here.

### Comment

Bis(tetraethylammonium) tetrahalometallates(II),  $[(C_2H_5)_4N]_2[MX_4]$ , with  $M = Fe, Co, Ni, Cu, Zn, Cd$ , and  $X = Cl, Br, I$ , have been the subject of numerous investigations. So far only the crystal structures of the room-temperature phase of  $[(C_2H_5)_4N]_2[NiCl_4]$  and the isotopic Co compound have been reported (Stucky, Folkers & Kistenmacher, 1967), in which the metal was found to be tetrahedrally coordinated by Cl atoms and the cation to have a disordered swastika conformation.

Ross, Siddiqi & Tyrrell (1972) confirmed the tetrahedral coordination of Cd in the corresponding tetrabromo- and tetraiodocadmates. Investigations of the optical birefringence showed that the title compound, (I), belongs to the tetragonal crystal system and undergoes two phase transitions at 39 and 311 K (Vlokh, Polovinko, Mokryi & Sveleba, 1991; Sveleba *et al.*, 1993). In this work we present the crystal structure of the compound in its room-temperature phase.



Buerger-precession and de Jong Bouman photographs show a  $\sqrt{2}/2a \times \sqrt{2}/2b \times c$  substructure. This substructure may be the reason why neither automatic heavy-atom nor direct methods using *Xtal3.2* (Hall, Flack & Stewart, 1992) and *SHELXS86* (Sheldrick, 1985) yielded anything chemically discernible. Therefore, the structure solution by manual interpretation of the Patterson map was attempted.

Inspection of the  $(uv0)$ -Harker plane confirmed  $P\bar{4}2_1m$  as the correct space group. Furthermore, this Harker plane exhibits three prominent vectors (marked with 'X' in Fig. 1) which delimit an equilateral triangle, the sides of which correspond to interatomic distances of about 3.5 Å identifying it as a face of a  $CdBr_4$  tetrahedron.

Thus, the basic feature of this structure is  $CdBr_4$  tetrahedra each with their threefold axis parallel to the crystallographic  $c$  axis. This is confirmed by a strong vector at  $(00w)$  corresponding to an interatomic distance of 2.6 Å, which in turn is consistent with a Cd—Br bond distance.

In projection along the crystallographic  $c$  axis the anions should appear as equilateral triangles with a Cd and a Br atom at the centre of mass. Assuming an ordered arrangement of the tetrahedra and avoiding close contacts (*i.e.* less than 3.5 Å) between Br atoms, it is possible to confine the triangles to positions where one of their bisectors lies on the mirror line of the plane group  $p4gm$ . Thus two Br atoms and the Cd atom should lie on the positions  $2b$  or  $4m$  of  $p4gm$ . The substructure mentioned above leads to a very strong vector in  $(\frac{1}{2} \frac{1}{2} 0)$ .