Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Polysulfonylamines. CXX.[1] Bis(tetrahydrothiophene-*S*)gold(I) benzene-1,2-disulfonimidate

Steffi Friedrichs and Peter G. Jones*

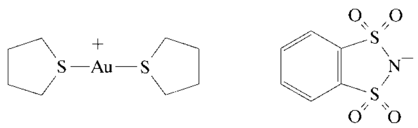
Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany
Correspondence e-mail: jones@xray36.anchem.nat.tu-bs.de

Received 14 June 1999
Accepted 14 October 1999

Both ions of the title compound, $[\mathrm{Au}(\mathrm{C_4H_8S})_2](\mathrm{C_6H_4NO_4S_2})$, display crystallographic twofold symmetry. The Au atom exhibits linear coordination, with Au—S = 2.2948 (14) Å and S—Au—S = 178.47 (9)°. The crystal packing consists of layers of anions connected by C—H···O hydrogen bonds; the cations occupy cavities in these layers and the ions are linked by Au···N contacts of 3.009 (7) Å. Further C—H···O interactions connect the layers.

Comment

We are interested in amine complexes of gold (Ahrens *et al.*, 1999). These are often obtained by displacement of tetrahydrothiophene (tht) from the complex (tht)AuCl. As an alternative tht–gold starting material, we wished to prepare the title compound, bis(tetrahydrothiophene-*S*)gold(I) benzene-1,2-disulfonimidate (see *Experimental*), (I), but initial attempts resulted in disappointing yields. The structure nevertheless presents some interesting features.



(I)

The title compound (Fig. 1) crystallizes with imposed twofold symmetry for the anion and cation (the N and Au atoms lie on special positions $0,y,\frac{1}{4}$ and $\frac{1}{2},y,\frac{1}{4}$, respectively). The gold centre displays the usual linear coordination, with Au—S = 2.2948 (14) Å and S—Au—S$^{\mathrm{i}}$ = 178.47 (9)° [symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$]. In $[(\mathrm{tht})_2\mathrm{Au}]^+[\mathrm{AuI}_2]^-$ (Ahrland *et al.*, 1984), the Au—S bonds were somewhat longer at 2.306 (7) and 2.335 (6) Å, and there was more deviation from linearity, with S—Au—S = 172.4 (2)°; this may be caused by association of the anion and cation *via* Au···Au interactions to form chains.

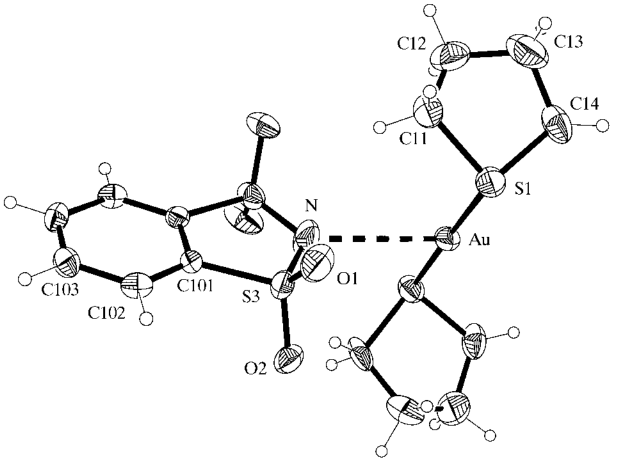


Figure 1
The structure of the title compound in the crystal. Ellipsoids represent 50% probability levels. H-atom radii are arbitrary. The Au···N contact is represented by a broken bond.

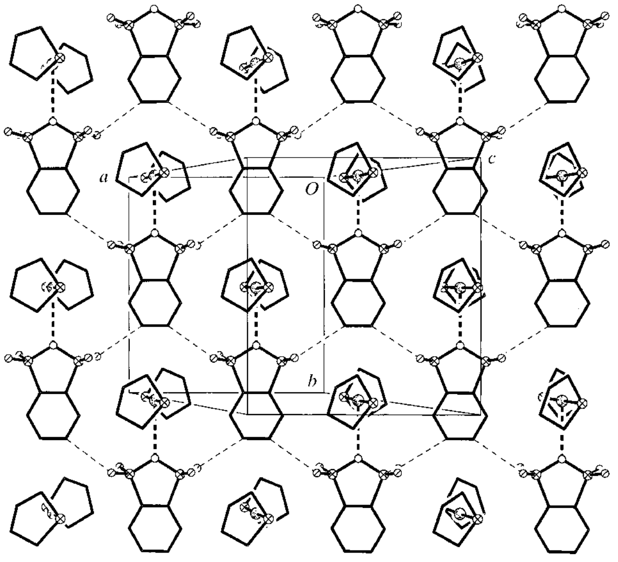


Figure 2
The layer structure of the title compound (H atoms have been omitted for clarity). C···O contacts (hydrogen bonds) are indicated by thin and Au···N contacts by thick dashed lines.

The tht ring adopts an envelope conformation, with C12 lying 0.58 (1) Å out of the plane of the other four atoms (r.m.s. deviation < 0.001 Å). The ring system of the anion is planar (r.m.s. deviation of nine atoms = 0.020 Å). Bond lengths and angles in both ring systems are as expected.

The crystal packing is determined not by Au···Au contacts [the shortest Au···Au distance is 7.9348 (9) Å], but rather by an Au···N contact of 3.009 (7) Å and by two short non-classical C—H···O hydrogen bonds (Table 1). A search for two-coordinate gold(I) complexes in the Cambridge Structural Database (Allen & Kennard, 1993) revealed only one shorter Au···N contact, namely 2.997 Å in adeninato(triethylphosphine)gold(I) (Tiekink *et al.*, 1989). The next shortest is 3.148 (19) Å in the related purine(dimethylphenylphos-

---

[1] Part CXIX: Wijaya *et al.* (1999).

phine)gold(I) compex (Pajunen *et al.*, 1995). There are no other Au$\cdots$N contacts less than 3.4 Å.

The H103$\cdots$O2 hydrogen bonds link the anions to form a layer structure (Fig. 2), the cavities of which are occupied by the cations. Perpendicular to the layers, H11*B*$\cdots$O2 hydrogen bonds link alternate anions and cations to form chains (not shown in Fig. 2). Neighbouring layers are therefore staggered.

Experimental

The title compound was obtained in low yield (14%) from the reaction between (tht)AuCl and the silver salt of the imide in acetonitrile. After filtering off insoluble material, the solution was allowed to stand in a refrigerator at 255 K. After several days, some small crystals had separated.

Crystal data

$[Au(C_4H_8S)_2](C_6H_4NO_4S_2)$
$M_r = 591.52$
Monoclinic, $C2/c$
$a = 10.6943\ (16)$ Å
$b = 11.7250\ (18)$ Å
$c = 16.374\ (3)$ Å
$\beta = 115.992\ (10)^\circ$
$V = 1845.5\ (5)$ Å^3
$Z = 4$

$D_x = 2.129$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 53 reflections
$\theta = 2.75$–11.35°
$\mu = 8.443$ mm^{-1}
$T = 173\ (2)$ K
Prism, colourless
$0.16 \times 0.12 \times 0.08$ mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scan (*XEMP*; Siemens, 1994*a*)
$T_{\min} = 0.382, T_{\max} = 0.509$
3205 measured reflections
1619 independent reflections
1307 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.039$
$\theta_{\max} = 25^\circ$
$h = -11 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -19 \rightarrow 18$
3 standard reflections every 247 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.044$
$S = 0.843$
1619 reflections
110 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0108P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.48$ e Å^{-3}

H atoms were placed at calculated positions and refined with a riding model. Light-atom U^{ij} values were restrained (command *DELU*). The N atom displays an appreciable component of the displacement parameter perpendicular to the anion plane, which may indicate slight disorder; removal of the N atom from the twofold axis, however, led to unstable refinement.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C103—H103$\cdots$O2$^{\rm i}$	0.95	2.40	3.165 (7)	138
C11—H11B$\cdots$O2$^{\rm ii}$	0.99	2.49	3.416 (8)	156

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

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994*b*); software used to prepare material for publication: *SHELXL*97.

We thank the Fonds der Chemischen Industrie for financial support, Mr A. Weinkauf for technical assistance and Professor A. Blaschette for advice on the use of disulfonylamine derivatives.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1354). Services for accessing these data are described at the back of the journal.

References

Ahrens, B., Jones, P. G. & Fischer, A. (1999). *Eur. J. Inorg. Chem.* pp. 1103–1110.
Ahrland, S., Norén, B. & Oskarsson, Å. (1985). *Inorg. Chem.* **24**, 1330–1333.
Allen, F. H. & Kennard. O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
Fait, J. (1991). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Pajunen, A., Kivekäs, R., Colacio, E. & Cuesta, R. (1995). *Acta Cryst.* C**51**, 2551–2554.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994*a*). *XEMP. An Empirical Absorption Correction Program.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994*b*). *XP. Molecular Graphics Program.* Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tiekink, E. R. T., Kurucsev, T. & Hoskins, B. F. (1989). *J. Crystallogr. Spectrosc. Res.* **19**, 823–839.
Wijaya, K., Moers, O., Blaschette, A. & Jones, P. G. (1999). *Z. Naturforsch. Teil B*. In the press.