

## Secondary interactions in gold(I) complexes with thione ligands.

### 5. Two ionic compounds of the form bis(thione)gold(I) bis(4-halobenzene-sulfonyl)amide<sup>1</sup>

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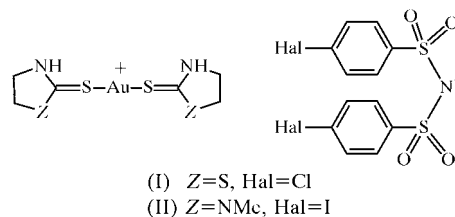
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Bis(1,3-thiazolidine-2-thione- $\kappa S^2$ )gold(I) bis(4-chlorobenzene-sulfonyl)amide,  $[\text{Au}(\text{C}_3\text{H}_5\text{NS}_2)_2](\text{C}_{12}\text{H}_8\text{Cl}_2\text{NS}_2\text{O}_4)$ , has no imposed symmetry. Classical  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds link the residues to form chains parallel to the  $b$  axis. Weaker interactions involve  $\text{C}-\text{H}\cdots\text{O}$ ,  $\text{C}-\text{H}\cdots\text{Au}$  and a number of  $X\cdots\text{Cl}$  contacts ( $X = \text{Cl}$ ,  $\text{S}$  or  $\text{Au}$ ) clustered in the region  $y \simeq \frac{1}{4}$ . In bis(1-methylimidazolidine-2-thione- $\kappa S^2$ )-gold(I) bis(4-iodobenzenesulfonyl)amide,  $[\text{Au}(\text{C}_4\text{H}_8\text{N}_2\text{S})_2](\text{C}_{12}\text{H}_8\text{I}_2\text{NS}_2\text{O}_4)$ , the Au atom of the cation and the N atom of the anion lie on the twofold axis ( $0, y, \frac{1}{4}$ ) in the space group  $C2/c$ . The formula unit forms a self-contained ring with two symmetry-equivalent  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, and weak  $\text{C}-\text{H}\cdots\text{X}$  ( $X = \text{O}$ ,  $\text{I}$  or  $\text{S}$ ),  $\text{Au}\cdots\text{I}$  and  $\text{I}\cdots\text{I}$  contacts are observed. In both compounds, the anions display extended conformations.

### Comment

We are interested in the secondary interactions involved in the packing of bis(thione)gold(I) complexes and have published a short series of papers reporting 12 structures so far (Friedrichs & Jones, 2004*a,b,c*, 2006). We report here the final two structures in the series, namely bis(thiazolidine-2-thione)-gold(I) bis(4-chlorobenzenesulfonyl)amide, (I), and bis(1-methylimidazolidine-2-thione)gold(I) bis(4-iodobenzenesulfonyl)amide, (II). The thione ligand names in (I) and (II) are hereinafter abbreviated to tzt and Me-etu, respectively (imidazolidine-2-thione is also known by the trivial name ethylenethiourea = etu). The structures of  $[\text{Au}(\text{tzt})_2]^+$  with the anions  $\text{Cl}^-$  (as the monohydrate; Akrivos *et al.*, 1994), camphorsulfonate (Friedrichs & Jones, 2004*b*) and dimesylamide [ $^-\text{DMS}$ ,  $\text{N}(\text{SO}_2\text{CH}_3)_2^-$ ; Friedrichs & Jones, 2004*c*], and

of  $[\text{Au}(\text{Me-etu})_2]^+$  with the anions  $\text{Cl}^-$  (Friedrichs & Jones, 2004*a*), camphorsulfonate (Friedrichs & Jones, 2004*b*) and DMS (Friedrichs & Jones, 2004*c*) will be referred to for comparison.



Compound (I) crystallizes with one formula unit in the asymmetric unit (Fig. 1). In compound (II), both the anion and the cation display crystallographic twofold symmetry, whereby atoms Au1 and N1 lie on the twofold axis ( $0, y, \frac{1}{4}$ ) (Fig. 2). The  $\text{Au1}\cdots\text{N1}$  separation is only 3.257 (4) Å.

Important structural parameters are summarized in Tables 1 and 3. Bond lengths and angles are as expected. The stereochemical disposition of the rings is governed by the torsion angles  $\text{Au}-\text{S}=\text{C}-\text{NH}$  and  $\text{C}-\text{S}\cdots\text{S}-\text{C}$ . The former, which determine which of the ring heteroatoms is *endo* or *exo* with respect to the Au centre, are, as invariably observed for these cations, antiperiplanar (ring S *endo*) for tzt and synperiplanar (ring NH *endo*) for Me-etu. However, the  $\text{C}-\text{S}\cdots\text{S}-\text{C}$  torsion angles of the cations across the linear gold(I) centres are much more variable, with absolute values for the three above-mentioned tzt compounds of 40, 9 and 10 (two independent cations), and 90°, respectively, and for the three above-mentioned Me-etu compounds of 180, 72 and 70, and 168°, respectively. Here, the values are 154° for (I) and 74° for (II). The torsion angles given here are absolute values rounded to the nearest degree; for exact values, see Tables 1 and 3 or the original publications.

The ligand heterocycles are approximately planar, but the r.m.s. deviations are appreciably non-zero [0.11 and 0.12 Å for (I), and 0.08 Å for (II)], with some correspondingly non-zero

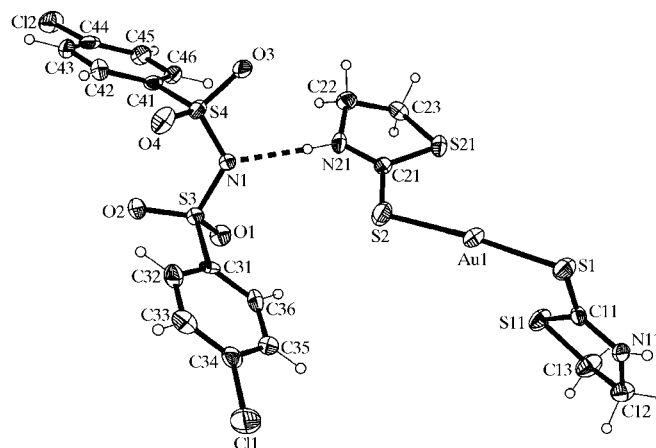


Figure 1

The asymmetric unit of compound (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The dashed line represents a hydrogen bond.

<sup>1</sup> This publication also forms part CLXXVII of the series Polysulfonylamines. Part 4/Part CLXXVI: Friedrichs & Jones (2006).

torsion angles [maxima 25 and 26° for (I) and 18° for (II) about the C—C bonds].

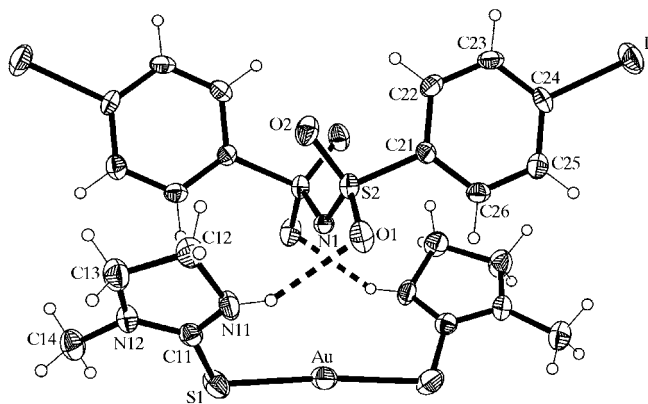
The bis(benzenesulfonyl)amide anions can display either a folded conformation with approximate local mirror symmetry ('hairpin' form) or an extended conformation with local twofold symmetry. In either case, two N—S—N—O torsion angles are approximately 180°, leading to a W-shaped O—S—N—S—O sequence; our convention is to assign these anti-periplanar O atoms the odd numbers, whereas the even-numbered O atoms are involved in synclinal S=O bonds. Each Cn1—Cn2 ring bond is generally synperiplanar to the adjacent S=O(sc) bond. For a more complete description of the standard geometry, see Lozano *et al.* (2004) and Henschel *et al.* (2005). In compounds (I) and (II), both anions display the extended conformation, with ring inter-centroid distances of 7.31 and 6.82 Å, interplanar angles of 25.3 (2) and 5.0 (3)°, and C—S···S—C torsion angles (across the central N atom) of 154 and 134°, respectively. This contrasts with the folded conformations observed for the [Au(etu)<sub>2</sub>]<sup>+</sup> salts of the same

anions (Friedrichs & Jones, 2006), with inter-centroid distances of *ca* 3.6–3.7 Å and torsion angles close to zero. The validity of the local symmetry can be expressed as  $\Delta\tau_{\text{SN}}$ , the average difference between related pairs of absolute torsion angles about the S—N bonds. This is exactly zero for (II) by symmetry, but is unusually large for (I) at 35°. The irregular geometry of (I) is also seen in the high synperiplanar O2—S3—C31—C32 torsion angle of 28°.

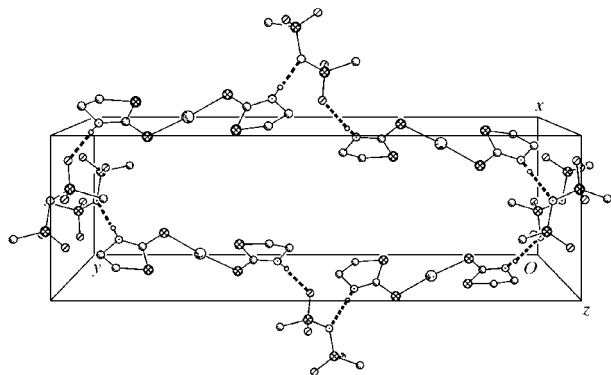
The classical hydrogen-bonding patterns for (I) and (II) would be expected to be relatively simple, because the cations each have two NH donor functions rather than the four of the etu derivatives. Previous derivatives with tzt have formed simple chain or ring motifs. In (I), the N21—H···N1 hydrogen bond within the asymmetric unit is augmented by N11—H···O3 *via* the 2<sub>1</sub> operator to form chains of molecules with overall direction parallel to the *b* axis (Fig. 3 and Table 3). The graph set (Bernstein *et al.*, 1995) is C<sub>2</sub><sup>2</sup>(12).

Previous Me-etu derivatives (and an isopropyl etu derivative; Hussain & Isab, 1985) have displayed either chain structures or self-contained rings within the asymmetric unit. Compound (II) forms its classical hydrogen bonds within the formula unit (Fig. 2). The graph set is R<sub>2</sub><sup>2</sup>(14). The N11—H···O1 interaction is significantly non-linear, and this may be caused by a long H11···N1 contact as a weak branch of an asymmetric three-centre system (Table 4).

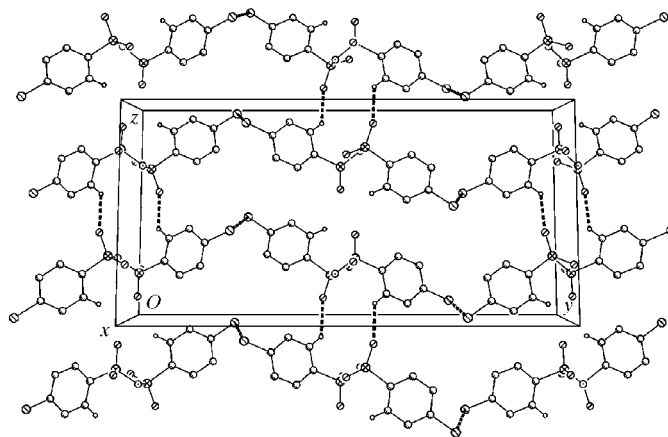
Non-classical ('weak') hydrogen bonding (Desiraju & Steiner, 1999) may be expected in systems where the number of acceptors exceeds the number of classical donors. In our previous publications, we have drawn attention to C—H···X interactions (X = O, but also Cl, S or Au), to short Au···S and S···S contacts, and, in one case, to the otherwise well known Au···Au (aurophilic) interactions (Schmidbaur, 1990), which are, however, not common in the thione complexes. For compound (I), two C—H···O interactions are observed between anions. There is also a short Cl1···Cl2 contact of 3.549 (2) Å [symmetry code: 1 - *x*, - $\frac{1}{2}$  + *y*,  $\frac{1}{2}$  - *z*; C—Cl···Cl angles = 78.0 (2) and 90.9 (2)°], so that it is sensible to speak of an anion substructure (Fig. 4). The other C—H···O inter-



**Figure 2**  
The formula unit of compound (II). Only the asymmetric unit is numbered. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines represent hydrogen bonds.



**Figure 3**  
Classical hydrogen bonding in compound (I), viewed perpendicular to the *ab* plane. Dashed lines indicate hydrogen bonds. For clarity, the aromatic rings are represented by the *ipso*-C atoms only.

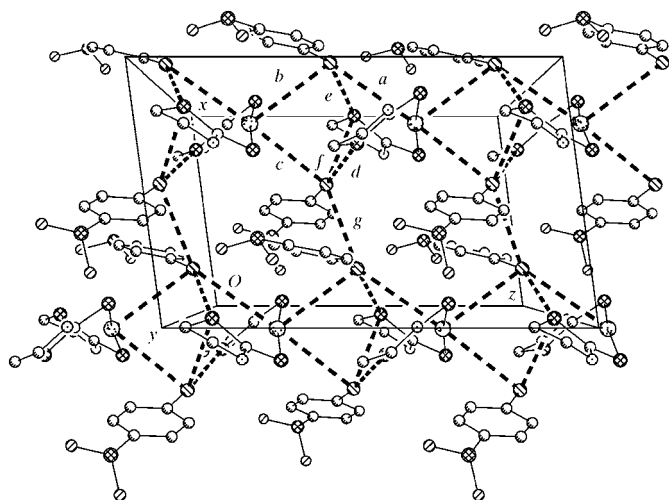


**Figure 4**  
The anion substructure of (I), viewed parallel to the *a* axis, showing the C—H···O and Cl···Cl interactions (dashed lines).

actions in Table 3 connect *a*-translated chains of Fig. 3. There are no C—H...Cl or C—H...S interactions.

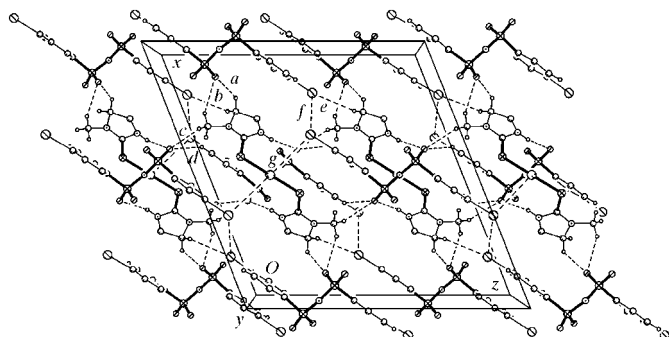
Compound (I) also shows a large number of short contacts to the exposed Cl atoms of the anions. These are concentrated around the region  $y \simeq \frac{1}{4}$  (Fig. 5). The C—H...Au contact (Table 3) also belongs to this part of the structure, but for clarity it is not shown in Fig. 5.

The extended packing of compound (II) is shown in Fig. 6. The dimers line up approximately parallel to the *a* axis and form an extended 'tube' parallel to the *c* axis in the region  $x \simeq \frac{1}{2}$ .



**Figure 5**

Short contacts (dashed lines) in (I) to Cl atoms in the region  $y \simeq \frac{1}{4}$ , viewed parallel to the *b* axis. Italic labels correspond to the following contacts: (a) Au...Cl1( $x+1, y, z$ ) 3.862 (2) Å; (b) Au...Cl1( $x+1, -y+\frac{1}{2}, z-\frac{1}{2}$ ) 4.005 (2) Å; (c) Au...Cl2( $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$ ) 3.768 (2) Å; (d) S11...Cl2( $-x+1, -y+1, -z$ ) 3.334 (2) Å; (e) S21...Cl1( $x+1, -y+\frac{1}{2}, z-\frac{1}{2}$ ) 3.539 (2) Å; (f) S21...Cl2( $-x+1, -y+1, -z$ ) 3.649 (2) Å; (g) Cl1...Cl2( $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$ ) 3.549 (2) Å. For clarity, anions are represented as SO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>Cl fragments.



**Figure 6**

A packing diagram for (II), viewed parallel to the *b* axis at  $y \simeq 0$ . A horizontal row of four dimers (arranged like backslashes) can be recognized across the centre of the diagram, with neighbouring anions top and bottom. Italic labels correspond to the following contacts (see Table 4 for details of hydrogen bonds; details of other interactions are given here): (a) H13A...O2; (b) H14C...O2; (c) H14B...O1; (d) C25...O1; (e) H13B...I; (f) I...I( $-x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$ ) 4.1116 (7) Å; (g) Au...I( $x, -y+1, z-\frac{1}{2}$ ) 3.9467 (6) Å. Classical hydrogen bonds (cf. Fig. 2) are shown as dashed lines but are not labelled.

They are connected amongst themselves and to neighbouring tubes *via* C—H...O and C—H...I interactions and Au...I and I...I contacts (for details, see Table 4 and the caption to Fig. 6). These tubes are connected in the *b* direction by the H23...S1 hydrogen bond.

Since these are provisionally the last structures in the series of 14, some concluding remarks are in order. The hope was to measure enough structures to permit generalized conclusions as to hydrogen-bonding patterns, and perhaps even to make cautious predictions of those patterns in as yet undetermined structures. With the benefit of hindsight, we can see that the systems have too many degrees of structural freedom. These may be classified into five categories, as follows:

(i) Cation flexibility: the C—S...S—C torsion angles may exhibit any values, and this is certainly the greatest barrier to 'crystal engineering'. The planarity of the heterocyclic rings and the Au—S—C—N torsion angles can also vary, but generally to a very limited extent.

(ii) Anion flexibility: the bis(benzenesulfonyl)amides can adopt the folded or extended conformation. Other anions used [DMS, benzene-1,2-di(sulfonylamide) and camphor-sulfonate] are less flexible.

(iii) Use of potential hydrogen-bond donor and acceptor functions: in general, all NH donors of the cations are used, although sometimes with unexpected acceptors (e.g. I of *p*-I-C<sub>6</sub>H<sub>4</sub> groups; Friedrichs & Jones, 2006). However, whereas the disulfonylamides often use just the O-atom acceptors, they sometimes utilize the central N atom as well as or instead of the O atoms.

(iv) Multi-centre hydrogen bonding: although most classical hydrogen bonds have been of the normal two-centre type, some systems {in particular [Au(etu)<sub>2</sub>]<sup>+</sup>[(*p*-Cl-C<sub>6</sub>H<sub>4</sub>)-SO<sub>2</sub>]<sub>2</sub>N]<sup>-</sup>; Friedrichs & Jones, 2006} exhibit three- or even four-centre hydrogen bonds.

(v) Non-classical hydrogen bonding and other 'weak' contacts: in general, the classical hydrogen bonds may be seen as structure-determining. However, C—H...*X* (*X* = O, halogen, S or Au) and contacts such as Au...*X* (*X* = O, N, halogen, S or Au) or S...*X* (*X* = S or halogen) are very difficult to predict but may have an important effect on the packing in some cases (e.g. when the classical hydrogen bonding leads to small closed units such as rings, the packing of the rings is generally determined by the weaker contacts).

## Experimental

Compounds (I) and (II) were prepared from the corresponding chlorides. The chloride (1 mmol) was suspended in methanol (30 ml) and treated with a solution of the silver disulfonylamide (1 mmol) (kindly provided by Professor A. Blaschette) in acetonitrile (5 ml). The cloudy reaction mixture was stirred for 1.5 h at room temperature in the dark. After filtering off the precipitated AgCl, the colourless filtrate was stored at 255 K for 12 h to yield crystals of (I) and (II).

For compound (I), yield: 0.258 g (65%); m.p. 426 K; <sup>1</sup>H NMR (*d*<sub>6</sub>-DMSO): δ 3.69 [4H, *t*, <sup>3</sup>*J*(H—H) = 8.3 Hz, CH<sub>2</sub>], 4.24 (4H, *t*, CH<sub>2</sub>), 7.44 (4H, *m*, H<sub>m</sub>), 7.45 (4H, *m*, H<sub>o</sub>), 11.80 (2H, *bs*, NH); MS (NBA; *A* = anion, *K* = cation): FAB (negative) *m/z* = 364 (100%, [*A*]<sup>-</sup>), 366

(75%,  $[A + 2H]^+$ ), 753 (8%,  $[K + H]^+$ ); FAB (positive)  $m/z = 316$  (21%,  $[K - (C_3H_6NS_2)]^+$ ), 434 (5%,  $[K - H]^+$ ), 435 (100%,  $[K]^+$ ), 437 (18%,  $[K + 2H]^+$ ), 750 (12%,  $[2K - (C_3H_6NS_2) - H]^+$ ), 752 (4%,  $[2K - (C_3H_6NS_2) + H]^+$ ); elemental analysis calculated: C 27.00, H 2.27, N 5.25, S 24.03, Cl 8.86%; found: C 26.54, H 2.20, N 5.18, S 23.80, Cl 8.61%.

For compound (II), yield: 0.302 g (62%); m.p. 441 K;  $^1H$  NMR ( $d_6$ -DMSO):  $\delta$  3.06 (6H, s,  $NCH_3$ ), 3.63 (4H,  $m$ , second-order spectrum of ABNM spin system,  $CH_2$ ), 3.85 (4H,  $m$ , second-order spectrum of ABNM spin system,  $CH_2$ ), 7.37 [4H,  $dt$ ,  $^3J(H_o-H_m) = 8.5$  Hz,  $H_o$ ], 7.73 (4H,  $dt$ ,  $H_m$ ), 9.11 (4H,  $bs$ , NH); MS (NBA): FAB (negative)  $m/z = 548$  (100%,  $[A]^+$ ), 421 (13%,  $[A - I]^+$ ); FAB (positive)  $m/z = 313$  (16%,  $[K - (CH_3-C_3H_6N_2S)]^+$ ), 399 (4%,  $[K - 2CH_3]^+$ ), 427 (13%,  $[K - 2H]^+$ ), 429 (100%,  $[K]^+$ ), 430 (12%,  $[K + H]^+$ ), 741 (2%,  $[2K - (CH_3-C_3H_6N_2S)]^+$ ); elemental analysis calculated: C 24.57, H 2.48, N 7.17, S 13.12%; found: C 24.61, H 2.48, N 7.06, S 13.01%.

## Compound (I)

### Crystal data

$[Au(C_3H_5NS_2)_2](C_{12}H_8Cl_2NO_4S_2)$   
 $M_r = 800.58$   
 Monoclinic,  $P2_1/c$   
 $a = 7.9342$  (10) Å  
 $b = 25.491$  (3) Å  
 $c = 12.6874$  (8) Å  
 $\beta = 97.639$  (8)°  
 $V = 2543.3$  (5) Å<sup>3</sup>

$Z = 4$   
 $D_x = 2.091$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 6.52$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Prism, colourless  
 $0.32 \times 0.28 \times 0.16$  mm

### Data collection

Siemens P4 diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scan  
 (XEMP; Siemens, 1994)  
 $T_{min} = 0.202$ ,  $T_{max} = 0.352$   
 5152 measured reflections  
 4483 independent reflections

2947 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.026$   
 $\theta_{max} = 25.0^\circ$   
 3 standard reflections  
 every 247 reflections  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.053$   
 $S = 0.83$   
 4483 reflections  
 316 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.64$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.49$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 (Sheldrick, 1997)  
 Extinction coefficient: 0.00069 (5)

**Table 1**

Selected geometric parameters (Å, °) for (I).

Au—S1	2.2873 (14)	S1—C11	1.696 (6)
Au—S2	2.2782 (15)	S2—C21	1.711 (5)
S2—Au—S1	171.23 (5)	C21—S2—Au	107.64 (19)
C11—S1—Au	103.45 (19)	S4—N1—S3	122.4 (3)
C11—S1—S2—C21	−82.4 (3)	S4—N1—S3—O1	145.7 (3)
Au—S1—C11—N11	−166.0 (5)	S4—N1—S3—O2	15.4 (4)
Au—S1—C11—S11	13.7 (4)	S4—N1—S3—C31	−102.3 (3)
N11—C12—C13—S11	−25.3 (6)	S3—N1—S4—O4	52.1 (4)
Au—S2—C21—N21	−174.7 (4)	S3—N1—S4—O3	180.0 (3)
Au—S2—C21—S21	6.7 (4)	S3—N1—S4—C41	−67.0 (4)
N21—C22—C23—S21	−26.3 (5)	O2—S3—C31—C32	−27.8 (5)
C31—S3—S4—C41	−153.9 (3)	O4—S4—C41—C42	7.6 (5)

**Table 2**

Hydrogen-bond geometry (Å, °) for (I).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N21—H21 $\cdots$ N1	0.86 (5)	2.01 (5)	2.864 (6)	174 (6)
N11—H11 $\cdots$ O3 <sup>i</sup>	0.84 (4)	2.03 (3)	2.838 (6)	161 (5)
C32—H32 $\cdots$ O4 <sup>ii</sup>	0.95	2.65	3.362 (6)	132
C46—H46 $\cdots$ O1 <sup>iii</sup>	0.95	2.43	3.182 (6)	136
C22—H22B $\cdots$ O1 <sup>iii</sup>	0.99	2.48	3.403 (7)	154
C12—H12A $\cdots$ O2 <sup>iv</sup>	0.99	2.66	3.269 (7)	120
C12—H12A $\cdots$ O4 <sup>iv</sup>	0.99	2.68	3.649 (8)	167
C43—H43 $\cdots$ Au <sup>v</sup>	0.95	2.95	3.690 (5)	136

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

## Compound (II)

### Crystal data

$[Au(C_4H_8N_2S_2)_2](C_{12}H_8I_2NO_4S_2)$   
 $M_r = 977.45$   
 Monoclinic,  $C2/c$   
 $a = 17.4002$  (14) Å  
 $b = 10.1254$  (10) Å  
 $c = 17.1493$  (18) Å  
 $\beta = 111.596$  (8)°  
 $V = 2809.3$  (5) Å<sup>3</sup>

$Z = 4$   
 $D_x = 2.311$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 7.77$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Prism, pale yellow  
 $0.28 \times 0.16 \times 0.16$  mm

### Data collection

Siemens P4 diffractometer  
 $\omega$  scans  
 Absorption correction:  $\psi$  scan  
 (XEMP; Siemens, 1994)  
 $T_{min} = 0.249$ ,  $T_{max} = 0.289$   
 4651 measured reflections  
 3218 independent reflections

2494 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.020$   
 $\theta_{max} = 27.5^\circ$   
 3 standard reflections  
 every 247 reflections  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.047$   
 $S = 0.89$   
 3218 reflections  
 170 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0199P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.52$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.61$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97  
 (Sheldrick, 1997)  
 Extinction coefficient: 0.00162 (4)

**Table 3**

Selected geometric parameters (Å, °) for (II).

Au—S1	2.2806 (11)	S1—C11	1.713 (4)
S1 <sup>i</sup> —Au—S1	174.84 (6)	S2 <sup>i</sup> —N1—S2	120.1 (3)
C11—S1—Au	107.28 (13)	S1—C11—C11 <sup>i</sup> —S1 <sup>i</sup>	−74.0 (3)
S1—C11—C11 <sup>i</sup> —S1 <sup>i</sup>	169.3 (3)	S2 <sup>i</sup> —N1—S2—O1	173.29 (12)
Au—S1—C11—N11	−11.0 (4)	S2 <sup>i</sup> —N1—S2—O2	44.83 (13)
C21—S2—S2 <sup>i</sup> —C21 <sup>i</sup>	−133.6 (3)	S2 <sup>i</sup> —N1—S2—C21	−73.42 (14)
		O2—S2—C21—C22	−10.2 (3)

Symmetry code: (i)  $-x, y, -z + \frac{1}{2}$ .

N-bound H atoms were refined freely, but with DFIX restraints ( $N-H = 0.85$  Å, with a notional s.u. of 0.02 Å). Methyl H atoms were identified in difference syntheses, idealized and refined as rigid groups allowed to rotate but not tip ( $C-H = 0.98$  Å and  $H-C-H =$

**Table 4**

Hydrogen-bond geometry (Å, °) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N11—H11 $\cdots$ O1	0.84 (4)	2.21 (3)	2.956 (4)	149 (4)
N11—H11 $\cdots$ N1	0.84 (4)	2.81 (4)	3.329 (4)	122 (3)
C13—H13A $\cdots$ O2 <sup>ii</sup>	0.99	2.52	3.328 (5)	139
C14—H14C $\cdots$ O2 <sup>ii</sup>	0.98	2.50	3.356 (5)	145
C14—H14B $\cdots$ O1 <sup>iii</sup>	0.98	2.63	3.501 (5)	148
C25—H25 $\cdots$ O1 <sup>iv</sup>	0.95	2.53	3.378 (5)	149
C13—H13B $\cdots$ I <sup>v</sup>	0.99	3.12	3.924 (4)	139
C23—H23 $\cdots$ S1 <sup>vi</sup>	0.95	2.83	3.645 (4)	144

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

109.5°). Other H atoms were treated using a riding model starting from calculated positions (aromatic C—H = 0.95 Å and methylene C—H = 0.99 Å), with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

For both compounds, data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3058). Services for accessing these data are described at the back of the journal.

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