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Bis(tetra-*n*-propylammonium) bis(2-thioxo-1,3-dithiole-4,5-dithiolato)mercurate(II)

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In the title compound, $[(C_3H_7)_4N]_2[Hg(C_3S_5)_2]$, each Hg atom is tetracoordinated by four S atoms in a distorted tetrahedral environment with Hg—S distances of 2.5217 (6)–2.5411 (8) Å. The crystal structure is mainly stabilized by van der Waals forces.

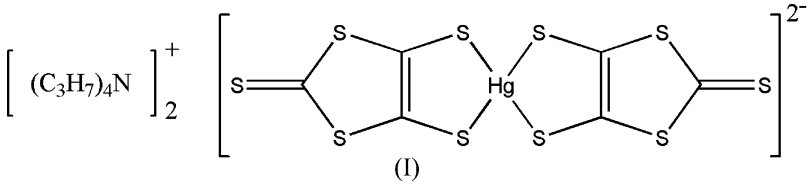
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.070
Data-to-parameter ratio = 35.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Transition metal complexes of 4,5-dimercapto-1,3-dithiole-2-thione (H_2L) are often used as electrical conductors and superconductors (Svenstrup & Becher, 1995; Cassoux, 1999; Pullen & Olk, 1999; Robertson & Cronin, 2002), and non-linear optical materials (Winter *et al.*, 1992; Zuo *et al.*, 1996; Wang *et al.*, 1999; Bai *et al.*, 1999; Dai *et al.*, 2000; Liu *et al.*, 2002). The title compound, (I), belongs to this family of complexes.



(I)

The asymmetric unit of (I) contains one $[HgL_2]^{2-}$ anion and two $[(C_3H_7)_4N]^+$ cations (Fig. 1). The ligand L shows its typical behaviour as a bidentate ligand and the Hg^{2+} ion is coordinated by four S atoms from the two ligands. The Hg—S bond lengths [2.5217 (7)–2.5411 (8) Å] (Table 1) and S—Hg—S bond angles [88.65 (2)–124.08 (2)°] (Table 1) show that Hg has a distorted tetrahedral coordination environment. The crystal structure (Fig. 2) exhibits no classical hydrogen bonds and is mainly stabilized by van der Waals forces.

Experimental

The synthesis of (I) involved a modification of literature methods (Steimeck & Kirmse, 1979; Wang *et al.*, 1998). To degassed dimethylformamide (DMF, 40 ml), CS_2 (20 ml) was added and the mixture was cooled to 273 K. Sodium (1.21 g) was added to the solution and the mixture was stirred vigorously with cooling until the reaction was complete. Several ml of MeOH were added slowly. Solutions of first $HgCl_2$ (4.24 g) dissolved in 25–28% NH_3 (40 ml), and then Pr_4NBr (6.97 g) in water (30 ml) were added consecutively with stirring at room temperature. The mixture was stirred overnight; the product was isolated by filtration and washed with water and MeOH, affording black crystals of (I). The high optical-quality brown single crystals used for X-ray structure analysis were obtained by slow evaporation of an acetone solution.

Crystal data

$(C_{12}H_{28}N)_2[Hg(C_3S_5)_2]$
$M_r = 965.96$
Triclinic, $P\overline{1}$
$a = 8.2566$ (3) Å
$b = 12.5783$ (4) Å
$c = 21.3073$ (6) Å
$\alpha = 83.0340$ (10)°
$\beta = 82.9180$ (10)°
$\gamma = 84.6280$ (10)°
$V = 2172.79$ (12) Å^3
$Z = 2$
$D_x = 1.476$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 4.04$ mm^{-1}
$T = 296$ (2) K
Prism, brown
0.22 × 0.15 × 0.14 mm

Data collection

Bruker APEX2 diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.493$, $T_{max} = 0.601$
(expected range = 0.466–0.568)
23094 measured reflections
13700 independent reflections
8094 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 32.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.070$
$S = 0.84$
13700 reflections
389 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.67$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00472 (15)

Table 1
Selected geometric parameters (Å, °).

Hg1—S7	2.5217 (7)	Hg1—S5	2.5305 (8)
Hg1—S4	2.5220 (6)	Hg1—S6	2.5411 (8)
S7—Hg1—S4	124.08 (2)	S7—Hg1—S6	88.65 (2)
S7—Hg1—S5	119.69 (2)	S4—Hg1—S6	122.69 (3)
S4—Hg1—S5	88.83 (2)	S5—Hg1—S6	116.09 (3)

All H atoms were positioned geometrically, with C—H = 0.96 Å (CH_2 groups) or 0.97 Å (CH_3 groups), and refined using a riding model, with $U_{iso}(H) = 1.2$ (1.5 times for CH_3 groups) times $U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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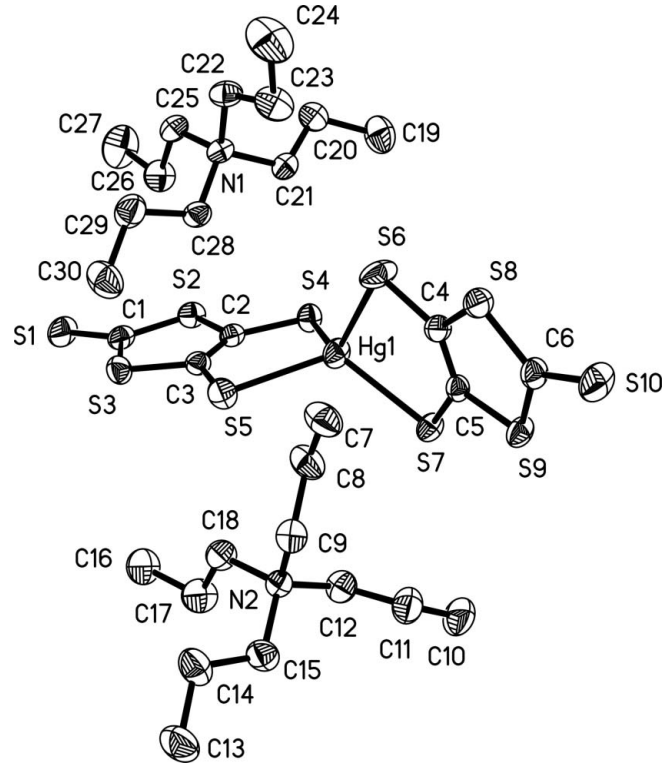

Figure 1
The molecular structure of (I), showing the atomic labelling scheme and 50% probability displacement ellipsoids. H atoms are omitted for clarity.

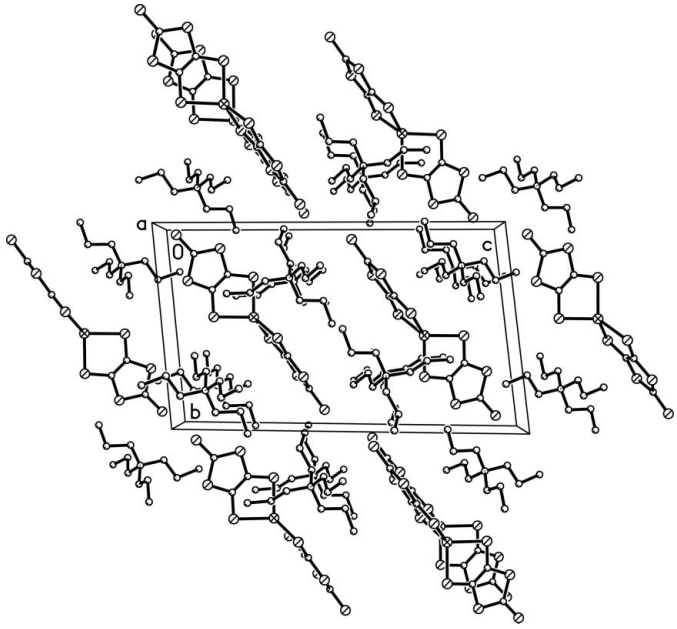

Figure 2
The packing of (I), viewed down the *a* axis. H atoms are omitted for clarity.

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