Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-(*p*-Phenylenedimethylenedithio)bis(4,5-dihydro-1,3-thiazine)

De-Qian Shi,[a] Wei Wang,[a]* Jing Wang[a] and Hai-Jun Chi[b]

[a]School of Chemical Engineering, Anshan University of Science and Technology, Anshan 114002, People's Republic of China, and [b]School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: wjwyj82@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.126
Data-to-parameter ratio = 15.4

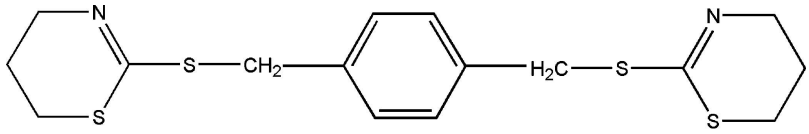
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 August 2006
Accepted 15 August 2006

The title compound, $C_{16}H_{20}N_2S_4$, was synthesized by the reaction of 1,4-dibromomethylbenzene and 1,3-thiazine-2-thione. The molecule is located on a crystallographic centre of inversion. The thiazine ring has an envelope conformation.

Comment

As a type of ditopic ligand, dithioethers can be used as bridging ligands in the construction of coordination polymers with soft metal ions. *N*-Heterocylic units have been synthesized and investigated (Sharma *et al.*, 1999; Constable *et al.*, 2002; Bu *et al.*, 2003; Hong *et al.*, 2000). Thiazine derivatives possess acaricide properties. In order to study the properties of these compounds, we have synthesized several new thiazine derivatives, and we present here the crystal structure of 2,2′-(*p*-phenylenedimethylenedithio)bis(4,5-dihydro-1,3-thiazine), (I).



(I)

Molecules of (I) have crystallographic inversion symmetry. The thiazine ring has an envelope conformation. Atom C2 deviates by 0.641 (3) Å from the plane of the remaining five ring atoms. Although the sum of the bond angles around C4 is 360°, two bond angles deviate significantly from the ideal value of 120° (Table 1).

Experimental

A solution of 1,4-dibromomethylbenzene (1.32 g, 5 mmol) in ethanol (5 ml) was added dropwise to a mixture of 1,3-thiazine-2-thione (1.46 g, 11 mmol), KOH (0.615 g, 11 mmol) and ethanol (5 ml). The

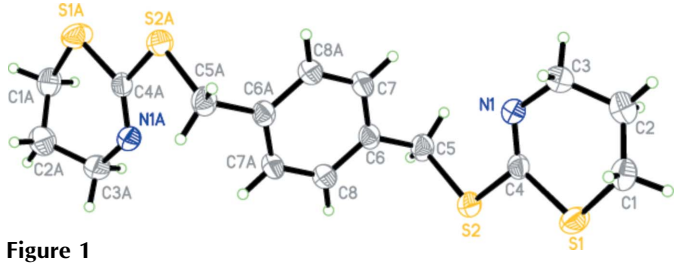


Figure 1
View of the molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Atoms with the suffix A are generated by the symmetry operation (−*x*, 1 − *y*, −*z*).

reaction mixture was then stirred for 24 h at room temperature. The mixture was added dropwise to water (30 ml) and a white precipitate appeared. This precipitate was filtered off, washed with water and recrystallized from ethanol and water (yield 65%, m.p. 350–351 K). Analysis calculated for $C_{16}H_{20}N_2S_4$: C 52.17, H 5.43, N 7.61%; found: C 52.09, H 5.41, N 7.65%. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a dichloromethane solution.

Crystal data

$C_{16}H_{20}N_2S_4$
M_r = 368.58
Monoclinic, $P2_1/c$
a = 14.280 (3) Å
b = 5.1581 (13) Å
c = 12.250 (3) Å
β = 103.782 (4)°
V = 876.3 (4) Å^3
Z = 2
D_x = 1.397 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.54 mm^{-1}
T = 293 (2) K
Block, colourless
0.20 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.837, T_{max} = 1.000
(expected range = 0.793–0.948)
4193 measured reflections
1540 independent reflections
1168 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.126
S = 1.06
1540 reflections
100 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 0.568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected bond angles (°).

N1—C4—S1	130.4 (2)	S1—C4—S2	106.73 (15)
N1—C4—S2	122.9 (2)		

All H atoms were positioned geometrically and refined as riding (C—H = 0.93 or 0.97 Å for aromatic and methylene H atoms, respectively). U_{iso}(H) values were set equal to 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge the 05 L003 project supported by the Education Department of Liao Ning Province in China.

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bu, X. H., Xie, Y. B., Li, J. R. & Zhang, R. H. (2003). *Inorg. Chem.* **42**, 7422–7430.
Constable, E. C., Housecroft, C. E., Kariuki, B. M., Kelly, N. & Smith, C. B. (2002). *Inorg. Chem. Commun.* **5**, 199–202.
Hong, M. C., Su, W. P., Cao, R., Fujita, M. & Lu, J. X. (2000). *Chem. Eur. J.* **6**, 427–431.
Sharma, C. V. K., Broker, G. A., Huddleston, J. G., Baldwin, J. W., Metzger, R. M. & Rogers, R. D. (1999). *J. Am. Chem. Soc.* **121**, 1137–1144.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.