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2,3-Bis(2-methylphenylimino)-1,4-dithiacyclohexane

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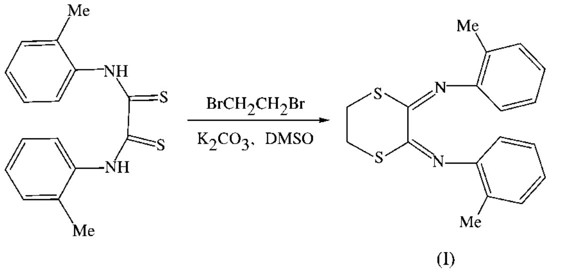
An *N*,*N*′-disubstituted dithiooxamide derivative reacts with 1,2-dibromoethane to produce the title compound, $C_{18}H_{18}N_2S_2$, a heterocycle with a double Schiff base. In the crystal structure, the molecule of the title compound lies on a twofold axis. Weak C—H···π interactions are the principal intermolecular forces, mediating the formation of layers parallel to the *ab* plane. Each molecule participates as donor and acceptor in two such contacts.

Comment

The chemistry of sulfur compounds has been the subject of intensive research in organic chemistry. As versatile intermediates and precursors, sulfur compounds have been employed to achieve the syntheses of many target compounds that are otherwise difficult to access, and thus sulfur has played an increasingly important role in organic synthesis. Organic sulfur compounds have also found important applications in organic conductors and superconductors, and in the life sciences field (Page, 1999).



(I)

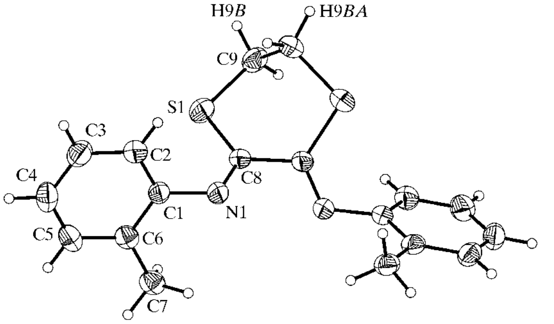


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

Thioamides are important organic sulfur compounds. The electronegativity of the S atom and the special electronic structure of these compounds make them more reactive than the corresponding amides, and thioamides take part in diverse chemical reactions. These compounds are of both academic and industrial interest because of their high stability and lack of odor, and because they can be synthesized and refined easily (Sosnicki *et al.*, 2001). More recently, some thioamides have shown a wide range of bioactivities and have been used as both anti-inflammatory and fungistatic reagents (Matysiak *et al.*, 2000) and also as antituberculins. Thioamides have also served as special reagents used both as chain terminators in Sanger-DNA sequencing (Schwarzer *et al.*, 2001) and as versatile intermediates in the syntheses of antitumor agents.

We have investigated the reaction of *N*,*N*′-bis(2-methylphenyl)dithiooxamide with 1,2-dibromoethane in the presence of potassium carbonate and have found that the product, (I), is a 1,4-dithiacyclohexane with a double Schiff base (see reaction scheme above). Such compounds are versatile intermediates in the synthesis of organic compounds because of their exceptional structures, and they have special utility in the synthesis of bioactive compounds (Jorgensen, 2000).

The crystal data show that the bond lengths and angles in (I) have unexceptional values and that there is a twofold axis in the molecule (Fig. 1 and Table 1). The S—Csp^3, S—Csp^2 and Csp^2=Nsp bond lengths [1.793 (3), 1.754 (2) and 1.258 (3) Å, respectively] are similar to those found for the compound

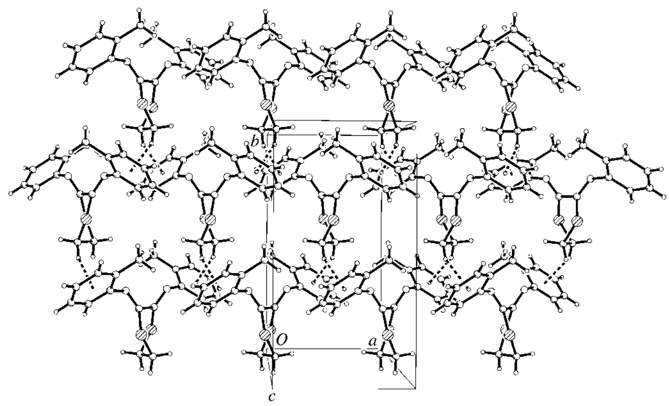


Figure 2
The crystal packing in (I), viewed along the *c* axis and showing the C—H···π short contacts (dashed lines).

reported by Ozarowski *et al.* (1988) [1.796 (3), 1.751 (2) and 1.263 (4) Å, respectively].

Recently, there has been increased interest in non-covalent hydrogen-bridged interactions involving π acceptors (Ni *et al.*, 2003). Jeffrey (1997) mentions this possibility and classifies these interactions as weak hydrogen bonds. In (I), weak C—H$\cdots\pi$ interactions play an important role; indeed, every molecule makes contacts of this type to two neighbors [C9—H9*B*$\cdots$*Cg*1($\frac{1}{2}-x$, $-\frac{1}{2}+y$, z); *Cg*1 is the center of gravity of the aromatic ring]. The molecule is packed into a layer structure, with aromatic rings acting as weak proton acceptors (see Fig. 2 and Table 2). The layers lie parallel to the *ab* plane and are stacked along the *c* axis, with molecules in adjacent layers having alternate orientations (adjacent layers are related by a *c*-glide perpendicular to the *b* axis).

Experimental

A solution of potassium carbonate (1.1 g, 8.0 mmol) and *N*,*N*′-bis(2-methylphenyl)dithiooxamide (1.2 g, 4.0 mmol) in anhydrous dimethyl sulfoxide (8 ml) was stirred at ~303–313 K for 1 h. 1,2-Dibromoethane (4.3 g, 22.9 mmol) was added slowly and the reaction mixture was kept at 333–338 K for 4 h, until all the dithiooxamide had disappeared (monitored by thin-layer chromatography). The mixture was cooled, ice water was added and the organic compound was extracted with petroleum ether. The solvent was removed *in vacuo* and the title compound was separated on a column of alumina with petroleum ether and ethyl acetate as eluants for stepwise elution. Yellow single crystals of (I) suitable for X-ray analysis were obtained by recrystallization from a mixture of petroleum ether and ethyl acetate.

Crystal data

$C_{18}H_{18}N_2S_2$
$M_r = 326.46$
Orthorhombic, *Pbcn*
$a = 6.7333$ (9) Å
$b = 12.6195$ (16) Å
$c = 19.827$ (3) Å
$V = 1684.7$ (4) Å^3
$Z = 4$
$D_x = 1.287$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2043 reflections
$\theta = 3.2$–24.4°
$\mu = 0.31$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.91$, $T_{\max} = 0.93$
9287 measured reflections
1941 independent reflections
1270 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 16$
$l = -22 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.147$
$S = 1.00$
1941 reflections
101 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.68$ e Å^{-3}
$\Delta\rho_{\min} = -0.81$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.754 (2)	N1—C1	1.417 (3)
S1—C9	1.793 (3)	C8—C8$^{\rm i}$	1.495 (5)
N1—C8	1.258 (3)	C9—C9$^{\rm i}$	1.489 (6)
C8—S1—C9	101.73 (12)	N1—C8—S1	123.79 (19)
C8—N1—C1	121.1 (2)	C8$^{\rm i}$—C8—S1	118.78 (15)
N1—C8—C8$^{\rm i}$	117.34 (18)		

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

Table 2
Hydrogen-bonding geometry (Å, °).

*Cg*1 is the center of gravity of the C1–C6 phenyl ring.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C9—H9*B*$\cdots$*Cg*1$^{\rm ii}$	0.97	2.81	3.683 (3)	150

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

All H atoms were positioned geometrically and refined as riding atoms, with isotropic displacement parameters set at $1.2U_{\rm eq}$ or $1.5U_{\rm eq}$ of the parent atoms. The distances to H atoms are in the range 0.93–0.97 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA1032). Services for accessing these data are described at the back of the journal.

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