

Bis(*N*-methyl-*N*-phenylthiocarbamoyl) disulfide

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Key indicators

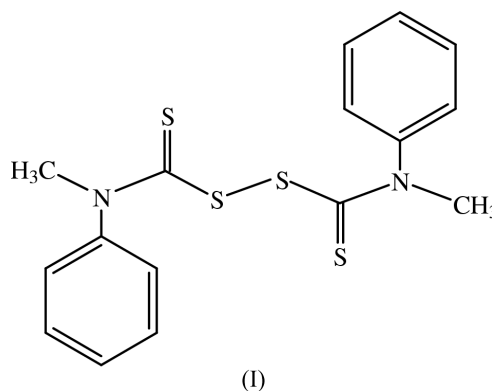
Single-crystal X-ray study
 $T = 293$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.071
 wR factor = 0.216
 Data-to-parameter ratio = 21.5

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

In the title molecule, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{S}_4$, the two dithiocarbamate groups, which are perpendicular to each other, are linked by an S—S bond. The dithiocarbamate groups adopt a *trans*-planar conformation and form a dihedral angle of $89.75(8)^\circ$ with each other. The phenyl rings make dihedral angles of $77.3(2)$ and $89.2(2)^\circ$ with the dithiocarbamate plane. The C—N bonds in the dithiocarbamate groups show partial double-bond character.

Comment

Bis(dialkylthiocarbamoyl) disulfide compounds are highly effective in breaking the dormancy of plant seeds, bulbs and tubers. These compounds can be used as herbicides or in the cultivation of crop plants during the off-season (Hideo *et al.*, 1974). Gasoline base-stocks are blended with 0.001–0.500% by weight of these compounds to obtain stable compounds suitable for long-term storage without sludge deposition (Kenichiro & Michiro, 1992). They are also used as additives to electrolytes for secondary lithium batteries. The use of these electrolytes prevents the growth of Li dendrites and results in a long life-cycle for the batteries (Masayuki, 1996).



The X-ray crystal structure of the title compound, (I) (Fig. 1), confirms that the molecule consists of two *N*-methyl-*N*-phenyldithiocarbamate units linked by an S—S bond. The two planar dithiocarbamate units are oriented perpendicular to each other, with a dihedral angle of $89.75(8)^\circ$ between them. The methyl C atoms are nearly coplanar with the NCSS units, with deviations of 0.010 (4) and 0.013 (5) Å. The bridging S2—S3 moiety is almost in the plane of each S₂CNMe subunit (Table 1). The two phenyl rings make angles of $77.3(2)$ and $89.2(2)^\circ$ with each S₂CNMe plane. The shorter N1—C8 and N2—C9 bond distances in the dithiocarbamate units are indicative of considerable double-bond character. The S—C, S=C and C—N bond distances are comparable

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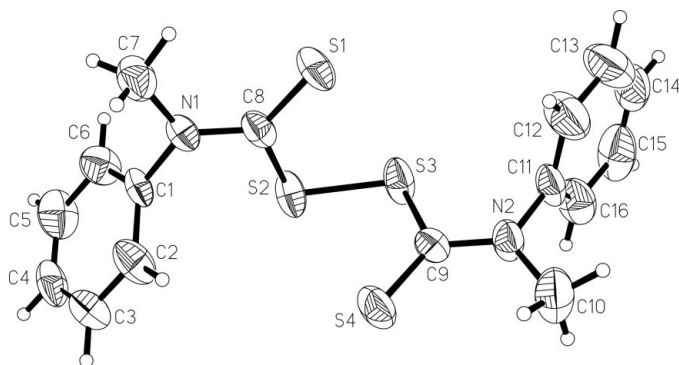


Figure 1
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

with those observed in related structures (Sharma *et al.*, 1991; Gimeno *et al.*, 1996; Jian *et al.*, 1999). Short $S2 \cdots C1$ [2.828 (3) Å], $S2 \cdots C9$ [2.980 (4) Å], $S3 \cdots C11$ [2.820 (4) Å], $S3 \cdots C8$ [2.985 (4) Å], $S1 \cdots C7$ [3.071 (4) Å] and $S4 \cdots C10$ [3.044 (5) Å] intramolecular contacts are observed in this structure.

Experimental

To a heated aqueous solution of sodium *N*-methyl-*N*-phenyldithiocarbamate was added, with stirring, a solution of lanthanum chloride. The white precipitate was collected by filtration. Colourless block crystals were obtained by recrystallizing the deposit from a solution of EtOH.

Crystal data

$C_{16}H_{16}N_2S_4$
 $M_r = 364.55$
Monoclinic, $P2_1/c$
 $a = 9.6233$ (2) Å
 $b = 10.7356$ (2) Å
 $c = 17.2999$ (3) Å
 $\beta = 91.624$ (1) $^\circ$
 $V = 1786.57$ (6) Å 3
 $Z = 4$

$D_x = 1.355$ Mg m $^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 5529 reflections
 $\theta = 2.1$ – 28.3 $^\circ$
 $\mu = 0.53$ mm $^{-1}$
 $T = 293$ (2) K
Block, colourless
 $0.26 \times 0.22 \times 0.08$ mm

Data collection

Siemens SMART CCD area-detector
 ω scans
Absorption correction: empirical (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.875$, $T_{\max} = 0.959$
12 183 measured reflections

4353 independent reflections
2395 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.083$
 $\theta_{\text{max}} = 28.3$ $^\circ$
 $h = -12 \rightarrow 11$
 $k = -14 \rightarrow 12$
 $l = -23 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.071$
 $wR(F^2) = 0.216$
 $S = 0.99$
4353 reflections
202 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1095P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.46$ e Å $^{-3}$
 $\Delta\rho_{\text{min}} = -0.78$ e Å $^{-3}$
Extinction correction: *SHELXTL*
Extinction coefficient: 0.012 (2)

Table 1

Selected geometric parameters (Å, $^\circ$).

S1–C8	1.647 (4)	N1–C1	1.447 (4)
S2–C8	1.810 (4)	N1–C7	1.477 (5)
S2–S3	2.0128 (14)	N2–C9	1.339 (5)
S3–C9	1.805 (4)	N2–C11	1.444 (5)
S4–C9	1.652 (4)	N2–C10	1.469 (5)
N1–C8	1.339 (5)		
S3–S2–C8–S1	3.7 (3)	S2–S3–C9–S4	5.2 (3)

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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