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Bis(dipropylthiocarbamoyl) disulfide†

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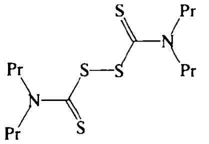
Abstract

In the title compound, $C_{14}H_{28}N_2S_4$, the two dithiocarbamate groups, which are perpendicular to each other, are bridged by an S—S bond. The C—N bonds in the dithiocarbamate groups show partial double-bond character.

Comment

The 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 basestocks are blended with 0.001–0.5wt% of these compounds to obtain a stable compound suitable for long-term storage without sludge deposition (Kenichiro & Michiro, 1992). They are also used as an additive for electrolytes of secondary lithium batteries. The use of these electrolytes prevents growth of Li dendrites and renders the batteries a longer life (Masayuki, 1996).



(I)

The title molecule, (I), consists of two dipropyldithiocarbamate units bridged by an S—S bond. The two planar dithiocarbamate units, containing a C—S and a C=S bond, are oriented perpendicular to each other with a dihedral angle of 89.98 (4)°. The shorter N1—C1 and N2—C8 bond distances [1.326 (3) and 1.337 (3) Å] in the dithiocarbamate units are indicative of considerable double-bond character. The S—C, S=C and C—N bond distances are comparable with those observed in the related structures (Sharma *et al.*, 1991; Gimeno *et al.*, 1996; Jian *et al.*, 1999). Short C2···S1 [2.893 (3) Å], C5···S2 [3.039 (3) Å], C9···S3 [2.910 (3) Å] and C12···S4 [3.061 (3) Å] intramolecular contacts are observed in this structure. The crystal structure is stabilized by van der Waals interactions.

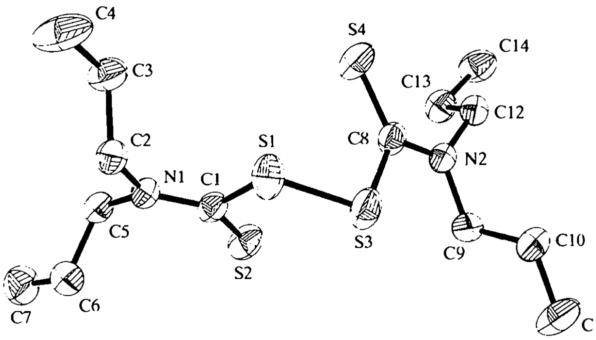


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

To a heated aqueous solution of *N*,*N*′-dipropyldithiocarbamate sodium salt was added a solution of lanthanum chloride with stirring. The white precipitate which formed was collected by filtration. The deposit was recrystallized from an acetonitrile solution.

Crystal data

$C_{14}H_{28}N_2S_4$
$M_r = 352.62$
Orthorhombic
Pbca
$a = 11.0232\,(2)$ Å
$b = 15.1645\,(2)$ Å
$c = 24.2826\,(1)$ Å
$V = 4059.11\,(9)$ Å^3
$Z = 8$
$D_x = 1.154$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8192 reflections
$\theta = 2.69$–$33.18°$
$\mu = 0.462$ mm^{-1}
$T = 293\,(2)$ K
Block
0.52 × 0.40 × 0.34 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.79$, $T_{max} = 0.86$
24592 measured reflections

4662 independent reflections
3205 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.50°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.143$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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† Alternative name: dithiobis(*N*,*N*-dipropylthioformamide).

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$S = 1.06$
4662 reflections
185 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 1.6893P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—C1	1.814 (3)	N1—C2	1.475 (3)
S1—S3	1.997 (1)	N1—C5	1.478 (3)
S2—C1	1.653 (2)	N2—C8	1.337 (3)
S3—C8	1.820 (3)	N2—C9	1.472 (3)
S4—C8	1.646 (3)	N2—C12	1.472 (3)
N1—C1	1.326 (3)		
C1—N1—C2—C3	92.0 (3)	C8—N2—C9—C10	−96.9 (3)
C1—N1—C5—C6	91.4 (3)	C8—N2—C12—C13	−90.0 (3)

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the intensity of duplicate reflections, and was found to be negligible.

All H atoms were geometrically fixed and allowed to ride on the attached atoms; rotating group refinement was used for the methyl groups. Atoms C4 and C11 of the propyl groups show high thermal vibrations but no indications of disorder were observed.

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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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1256). Services for accessing these data are described at the back of the journal.

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p-Hydroxybenzaldehyde nicotinoylhydrazone monohydrate

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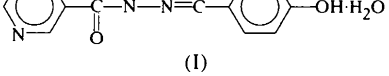
Abstract

The title compound, $C_{13}H_{11}N_3O_2{\cdot}H_2O$, is in the hydrazide tautomeric form and the configuration of the azomethine C=N double bond [1.275 (3) Å] is *E*. In the solid state, the molecules are stacked parallel to (110) planes and form a hydrogen-bonded column-like supramolecular structure involving two independent water molecules on twofold axes.

Comment

Arylhydrazones have been investigated extensively in recent years because of their biological activity, especially as potent inhibitors for many enzymes (Ma *et al.*, 1994; Lu *et al.*, 1996; Fun, Lu, Duan, Tian, You, Gong & Guo, 1997; Fun, Lu, Duan, Tian, You, Guo & Gong, 1997). As part of our work on the synthesis and characterization of arylhydrazone compounds, we report here the structure of *p*-hydroxybenzaldehyde nicotinoylhydrazone monohydrate, (I).



(I)

The geometry of the molecule agrees well with those found in related structures (Fun, Lu, Duan, Tian, You,

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