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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
R factor = 0.068
wR factor = 0.214
Data-to-parameter ratio = 17.3

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

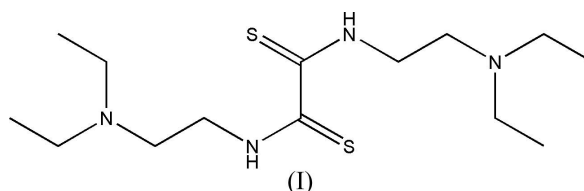
N,N'-Bis[2-(diethylamino)ethyl]dithiooxalamide

In the title compound, $\text{C}_{14}\text{H}_{30}\text{N}_4\text{S}_2$, the molecules lie about crystallographic inversion centres. A weak $\text{N}-\text{H}\cdots\text{S}$ hydrogen-bonding interaction is observed in the crystal structure.

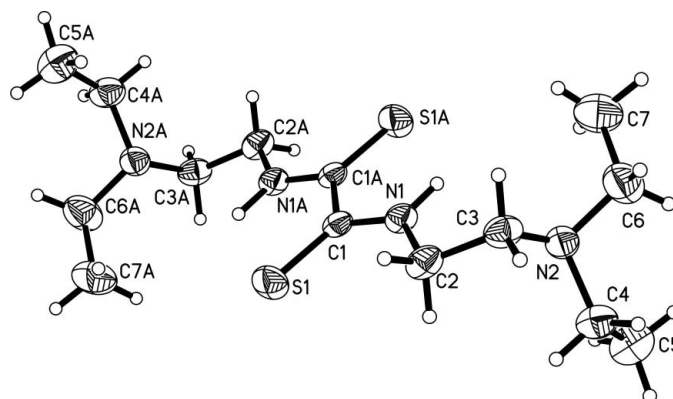
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Comment

The title compound, (I), is a representative of the relatively scarcely studied *N*-substituted thiooxamides, which may be used as versatile nitrogen- and sulfur-containing ligands for multinuclear metal complexes with special magnetic properties (Hurd *et al.*, 1960). Only a few such molecules have been reported (Cui *et al.*, 2004), because of the difficulties in their preparation and purification.



The molecular structure of the title compound, (I), is illustrated in Fig. 1. The asymmetric unit contains one half-molecule, with the other half generated by an inversion centre, which lies at the mid-point of the $\text{C1}-\text{C1A}$ bond [symmetry code: (A) $-x, 1-y, 2-z$]. The $\text{C2}-\text{N1}-\text{C1}-\text{S1}$ and $\text{C2}-\text{N1}-\text{C1}-\text{C1}$ torsion angles (Table 1) indicate that these atoms are almost coplanar. A view of the molecular packing down the *b* axis is shown in Fig. 2. The crystal packing is stabilized by a weak $\text{N}-\text{H}\cdots\text{S}$ hydrogen-bonding interaction (Table 2).



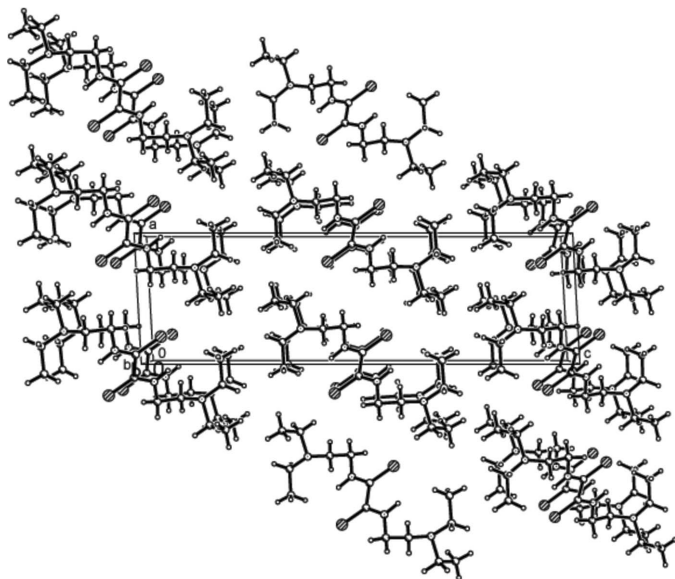


Figure 2
The crystal packing of (I), viewed along the *b* axis.

Experimental

The title compound was prepared according to the procedure reported by Hurd *et al.* (1961). The reaction was initiated by the addition of dithioamide (1 mmol, 1.2 g) and *N,N*-diethylethylenediamine (2.32 g, 2 mmol) to ethanol in a flask. The solution was stirred at room temperature for 24 h to give an orange precipitate (yield 66%; m.p. 322 K). Single crystals suitable for crystallographic analysis were obtained by slow evaporation of an *n*-hexane solution. IR (KBr, ν cm^{-1}): 3200, 2969, 2821, 1524, 1386, 1245, 1067, 946, 833, 743. MS ($M+1$): 319.

Crystal data

$\text{C}_{14}\text{H}_{30}\text{N}_4\text{S}_2$	$D_x = 1.132 \text{ Mg m}^{-3}$
$M_r = 318.54$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 697 reflections
$a = 7.264$ (4) Å	$\theta = 2.8\text{--}20.4^\circ$
$b = 5.317$ (3) Å	$\mu = 0.28 \text{ mm}^{-1}$
$c = 24.239$ (15) Å	$T = 294$ (2) K
$\beta = 93.158$ (10)°	Prism, orange
$V = 934.9$ (9) Å ³	$0.20 \times 0.16 \times 0.10 \text{ mm}$
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	1639 independent reflections
φ and ω scans	737 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	$R_{\text{int}} = 0.095$
$T_{\text{min}} = 0.936$, $T_{\text{max}} = 0.972$	$\theta_{\text{max}} = 25.0^\circ$
4382 measured reflections	$h = -8 \rightarrow 8$
	$k = -6 \rightarrow 3$
	$l = -26 \rightarrow 28$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.214$
 $S = 1.01$
 1639 reflections
 95 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0908P)^2 + 0.1316P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$

Table 1

Selected torsion angles (°).

C2–N1–C1–C1 ⁱ	–179.9 (5)	C2–N1–C1–S1	–1.3 (7)
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Symmetry code: (i) $-x, -y + 1, -z + 2$.

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N1–H1A \cdots S1 ⁱ	0.96 (5)	2.39 (5)	2.931 (4)	115 (4)

Symmetry code: (i) $-x, -y + 1, -z + 2$.

H atoms on C atoms were positioned geometrically and refined as riding (C–H = 0.96 or 0.97 Å). The H atom bonded to N1 was refined freely [N–H = 0.96 (5) Å]. For NH and CH₂ groups, $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{carrier atom})$ and for the methyl groups they were set equal to $1.5U_{\text{eq}}(\text{carrier atom})$. The crystal showed a weak diffracting ability, which accounts for the rather low ratio of observed reflections to parameters (45%).

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.

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