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N,*N*′-Bis[2-(dimethylamino)ethyl]dithiooxamide

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In the title compound, $C_{10}H_{22}N_4S_2$, molecules lie across crystallographic inversion centres. Weak N—H···N and N—H···S hydrogen-bonding interactions are observed in the molecular structure.

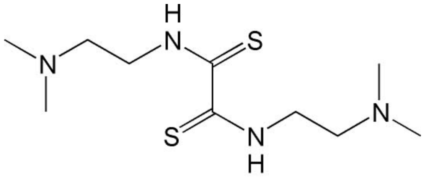
Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.116
Data-to-parameter ratio = 14.9

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

Comment

N-coordinating-group substituted dithiooxamides can be used to synthesize multinuclear metal complexes with S and N as donor atoms (Hurd *et al.*, 1960). A few studies on dinuclear or trinuclear metal complexes coordinated by dithiooxamide with substituents 2-aminomethylpyridine (Cui *et al.*, 1999), 2-aminoethylpyridine (Cui *et al.*, 2004), amino acids (Veit *et al.*, 1984; Castineiras *et al.*, 2001) and 2-hydroxyethyl (Girerd *et al.*, 1978) have been reported. We report here the preparation and X-ray crystal structure determination of the title compound, (I).



(I)

The asymmetric unit of (I) contains one half-molecule, with the other half generated by a centre of inversion, which lies at the midpoint of the C1—C1^i bond [symmetry code: (i) $1 - x$, $-y$, $1 - z$; Fig. 1]. The geometry of atom N2 is observed to be pyramidal in character, with the sum of the bond angles (Table 1) around atom N2 equal to about 332°. Weak intramolecular N—H···N and N—H···S hydrogen-bonding interactions are observed in the molecular structure. A view of the molecular packing down the *b* axis is shown in Fig. 2.

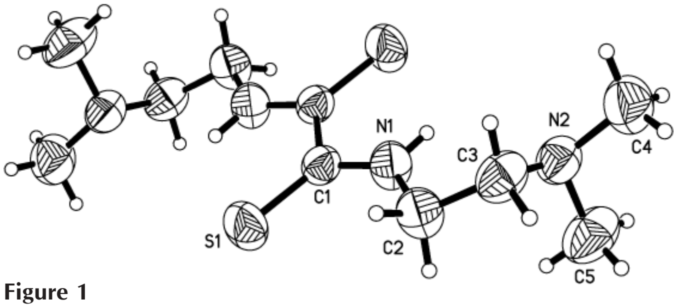


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. Only the major component of the disordered dimethylaminoethyl group is shown. Unlabelled atoms are related to labelled atoms by ($1 - x$, $-y$, $1 - z$).

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Experimental

Compound (I) was prepared according to the literature procedure of Hurd *et al.* (1961). One molar equivalent of dithiooxamide and two molar equivalents of *N*,*N*-dimethylethylenediamine were added to ethanol and stirred at room temperature for 24 h, to obtain (I) as an orange powder (yield 74%; m.p. 365–366 K). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a petroleum ether solution. IR (KBr, cm^{-1}): ν 3164, 1520, 1462, 1309, 1043, 962, 914, 835, 781.

Crystal data

$C_{10}H_{22}N_4S_2$
$M_r = 262.44$
Monoclinic, $P2_1/c$
$a = 9.964$ (2) Å
$b = 7.2840$ (16) Å
$c = 11.462$ (3) Å
$\beta = 110.917$ (3)°
$V = 777.1$ (3) Å^3
$Z = 2$
$D_x = 1.122$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1018 reflections
$\theta = 3.4$–20.3°
$\mu = 0.33$ mm^{-1}
$T = 293$ (2) K
Block, orange
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.902$, $T_{\max} = 0.943$
4992 measured reflections
1828 independent reflections
979 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.023$
$\theta_{\max} = 27.9°$
$h = -13 \rightarrow 12$
$k = -9 \rightarrow 6$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.116$
$S = 0.99$
1828 reflections
123 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.0516P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.11$ e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6496 (17)	N2—C5	1.447 (6)
N1—C1	1.2993 (19)	N2—C3	1.453 (6)
N1—C2	1.469 (4)	N2—C4	1.456 (6)
N1—C2′	1.492 (7)	C2—C3	1.491 (6)
C1—C1$^{\text{i}}$	1.524 (3)		
C1—N1—C2	128.6 (2)	C5—N2—C4	110.2 (5)
C1—N1—C2′	117.5 (5)	C3—N2—C4	110.7 (5)
N1—C1—S1	125.09 (13)	C5′—N2′—C3′	110.2 (8)
C1$^{\text{i}}$—C1—S1	121.65 (17)	C5′—N2′—C4′	110.6 (8)
C5—N2—C3	111.9 (6)	C3′—N2′—C4′	110.9 (9)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S1$^{\text{i}}$	0.90	2.37	2.910 (2)	119
N1—H1*B*···N2	0.90	2.35	2.782 (7)	110

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

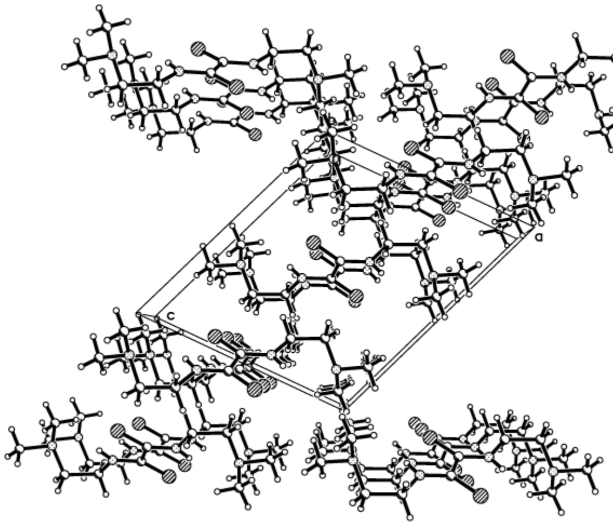

Figure 2
The crystal packing of (I), viewed along the *b* axis. Only the major component of the disordered dimethylaminoethyl group is shown.

The dimethylaminoethyl group is found to be disordered over two orientations, with occupancies of 0.666 (4) and 0.334 (4). All H atoms were positioned geometrically and refined as riding (C—H = 0.96 or 0.97 Å and N—H = 0.90 Å). For NH and CH$_2$ groups, U_{iso}(H) values were set equal to $1.2U_{\text{eq}}$(carrier atom), and for the methyl groups, they were set equal to $1.5U_{\text{eq}}$(carrier atom).

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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