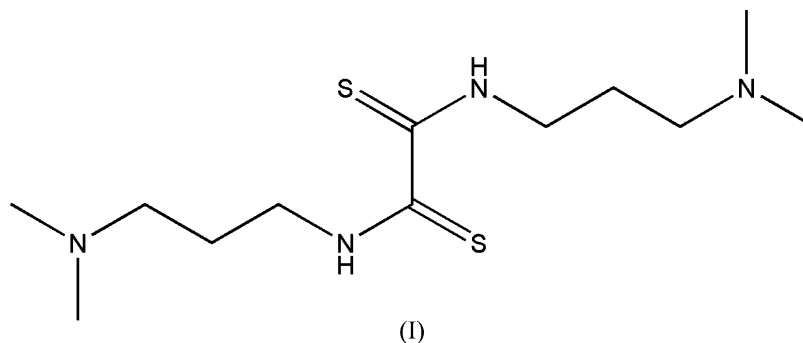


N,N'-Bis(3-dimethylaminopropyl)dithiooxamideJian-Zhong Cui,* Dan Zhang,
Fan-Li Lu, Hong-Ling Gao and
Hai-Tao WangDepartment of Chemistry, Tianjin University,
Tianjin 300072, People's Republic of ChinaCorrespondence e-mail:
cuijianzhong@tju.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.045
 wR factor = 0.151
Data-to-parameter ratio = 16.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Molecules of the title compound, $\text{C}_{12}\text{H}_{26}\text{N}_4\text{S}_2$, have $2/m$ symmetry. Intramolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds are observed.Received 27 October 2005
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Comment

Substituted dithiooxamides with N-coordinating groups can be used to synthesize multinuclear metal complexes with S- and N-donor atoms. A few studies on such ligands (Hurd *et al.*, 1960; Bermejo *et al.*, 1998; Cui, Zhang, Pan *et al.*, 2005; Cui, Zhang, Song *et al.*, 2005; Cui, Zhang, Zhang *et al.*, 2005) and their dinuclear or trinuclear metal complexes coordinated by substituted dithiooxamides with 2-(aminomethyl)pyridine (Cui *et al.*, 1999), 2-(aminoethyl)pyridine (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 X-ray crystal structure determination of the title compound, (I).The asymmetric unit of (I) contains one quarter-molecule disposed on a mirror plane with a second methyl group generated by reflection, and with the other half generated by twofold rotation symmetry, the axis passing through the inversion centre at the mid-point of the $\text{C}1-\text{C}1^i$ bond [Fig. 1 and Table 1; symmetry code: (i) $-x, -y, -z$]. There are intramolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds, as detailed in Table 2.

Experimental

Compound (I) was prepared according to the literature procedure of Hurd *et al.* (1961). Ethanol solutions of one molar equivalent of dithiooxamide and two molar equivalents of *N,N*-dimethylaminopropylamine were mixed and stirred at room temperature for 8 h to obtain (I) as an orange powder (yield 34%; m.p. 316–317 K). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a petroleum ether solution of (I). IR (KBr): ν 2943, 2853, 2815, 1519, 1460, 1380, 1245, 1160, 922, 880, 762 cm^{-1} .

Crystal data

C₁₂H₂₆N₄S₂
M_r = 290.49
 Tetragonal, *P*4₂/*m*bc
a = 14.4361 (19) Å
c = 8.2304 (15) Å
V = 1715.2 (4) Å³
Z = 4
D_x = 1.125 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 2361 reflections
 θ = 2.8–26.2°
 μ = 0.30 mm⁻¹
T = 294 (2) K
 Block, orange
 0.24 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.921, *T_{max}* = 0.959
 8983 measured reflections

953 independent reflections
 599 reflections with *I* > 2σ(*I*)
R_{int} = 0.046
 θ_{max} = 26.4°
h = -18 → 17
k = -18 → 12
l = -8 → 10

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.045
wR(*F*²) = 0.151
S = 1.04
 953 reflections
 57 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 1.1916P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 $\Delta\rho_{max} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.24 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.067 (6)

Table 1

Selected geometric parameters (Å, °).

S1–C1	1.665 (4)	N2–C4	1.459 (5)
N1–C1	1.306 (5)	N2–C5	1.425 (4)
N1–C2	1.450 (5)		
S1–C1–N1	124.5 (3)	C1–N1–C2	124.3 (3)
S1–C1–C1 ⁱ	121.7 (4)	C4–N2–C5	110.6 (3)
N1–C1–C1 ⁱ	113.8 (4)	C5–N2–C5 ⁱⁱ	111.2 (5)

Symmetry codes: (i) -*x*, -*y*, -*z* + 1; (ii) *x*, *y*, -*z* + 1.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N1–H1A...N2	0.88	2.17	2.851 (4)	134
N1–H1A...S1 ⁱ	0.88	2.41	2.938 (3)	118

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

Atom C3 is disordered over the mirror plane and was refined anisotropically with an occupancy factor of 0.50. All H atoms were positioned geometrically and refined using the riding-model approximation, with N–H = 0.88 Å and C–H = 0.96 Å, and with *U*_{iso}(H) = 1.2*U*_{eq}(N and methylene C) or 1.5*U*_{eq}(methyl C).

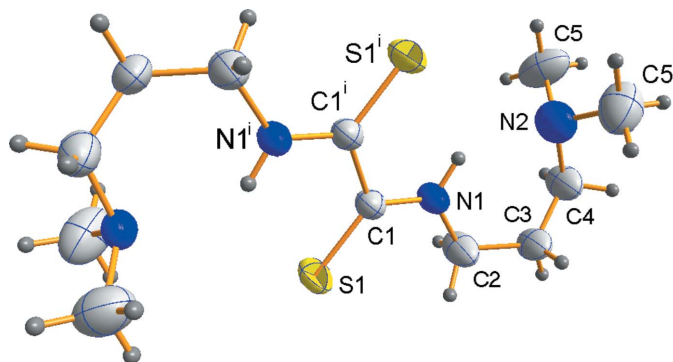


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) -*x*, -*y*, -*z*; (ii) *x*, *y*, 1 - *z*.]

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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