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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.033
wR factor = 0.097
Data-to-parameter ratio = 19.1

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

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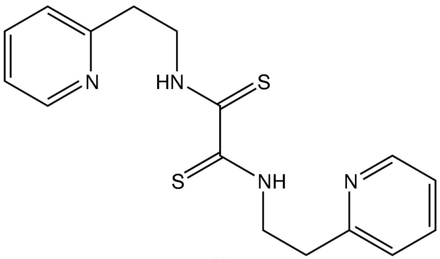
The title compound, $C_{16}H_{18}N_4S_2$, has a crystallographic inversion centre at the mid-point of the central C—C bond. The plane of the dithiooxamide fragment is approximately perpendicular to the plane of the dimethylene bridge [dihedral angle = 84.8 (1)°], the plane of which is in turn normal to the pyridine ring plane [dihedral angle = 89.80 (8)°]. An intermolecular N—H···N hydrogen bond involving the amide NH group and the pyridine N atom [N···N = 3.0503 (18) Å and N—H···N = 145.8 (14)°] links the molecules into chains extending along the *c* axis.

Comment

The title compound 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 title compound, (I), was synthesized according to the method of Cui *et al.* (2004); its molecular structure is shown in Fig. 1. The molecule of (I) has a crystallographic inversion centre at the mid-point of the C1—C1*A* bond. The dithiooxamide plane, defined by atoms N1, C1 and S1 and their symmetry-related counterparts, is approximately orthogonal to the mean plane of the dimethylene bridge, N1/C2–C4 [the dihedral angle is 84.8 (1)°], which in turn is almost orthogonal to the pyridine plane, N2/C4–C8 [dihedral angle 89.80 (8)°]. The pyridine plane forms a small dihedral angle of 6.2 (2)° with the central dithiooxamide plane.



(I)

An intermolecular N1—H1···N2^{ii} bond [symmetry code: (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; Table 2] links the molecules of (I) into chains extending along the *c* axis. The packing of the molecules in the crystal structure of (I) is shown in Fig. 2.

Experimental

The title compound was prepared as an orange powder, with a melting point of 431 K, according to the procedure reported by Cui *et*

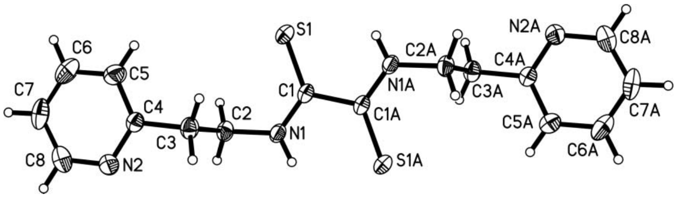


Figure 1
View of the molecule, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as circles of arbitrary size. [Symmetry code: (A) $1 - x, -y, 1 - z$].

al. (2004) (yield 80%). Single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{16}H_{18}N_4S_2$
$M_r = 330.46$
Monoclinic, $P2_1/c$
$a = 10.4499$ (18) Å
$b = 6.0036$ (10) Å
$c = 13.688$ (2) Å
$\beta = 103.995$ (2)°
$V = 833.2$ (2) Å^3
$Z = 2$
$D_x = 1.317$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1570 reflections
$\theta = 3.2$–27.1°
$\mu = 0.32$ mm^{-1}
$T = 293$ (2) K
Prism, orange
$0.28 \times 0.24 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.902$, $T_{\max} = 0.938$
5351 measured reflections
1982 independent reflections
1491 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 27.9°$
$h = -13 \rightarrow 10$
$k = -7 \rightarrow 7$
$l = -15 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.097$
$S = 1.11$
1982 reflections
104 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6591 (13)	C2—C3	1.520 (2)
N1—C1	1.3112 (18)	C3—C4	1.498 (2)
N1—C2	1.4511 (17)	C4—C5	1.377 (2)
N2—C4	1.3256 (19)	C5—C6	1.367 (3)
N2—C8	1.338 (2)	C6—C7	1.362 (3)
C1—C1$^{\rm i}$	1.530 (2)	C7—C8	1.366 (3)
C1—N1—C2	124.12 (12)	N2—C4—C5	121.91 (14)
C4—N2—C8	117.21 (14)	N2—C4—C3	117.04 (13)
N1—C1—C1$^{\rm i}$	114.32 (14)	C5—C4—C3	121.01 (13)
N1—C1—S1	124.50 (10)	C6—C5—C4	119.75 (17)
C1$^{\rm i}$—C1—S1	121.18 (13)	C7—C6—C5	119.06 (17)
N1—C2—C3	112.12 (12)	C6—C7—C8	117.93 (17)
C4—C3—C2	110.79 (12)	N2—C8—C7	124.15 (18)
C2—N1—C1—C1$^{\rm i}$	−179.72 (13)	C2—C3—C4—N2	89.18 (16)
C2—N1—C1—S1	0.23 (19)	C2—C3—C4—C5	−88.52 (18)
C1—N1—C2—C3	−84.01 (17)	C3—C4—C5—C6	177.57 (16)
N1—C2—C3—C4	175.80 (12)	C4—C5—C6—C7	0.3 (3)
C8—N2—C4—C5	−0.3 (2)	C6—C7—C8—N2	−0.1 (3)
C8—N2—C4—C3	−177.96 (13)		

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

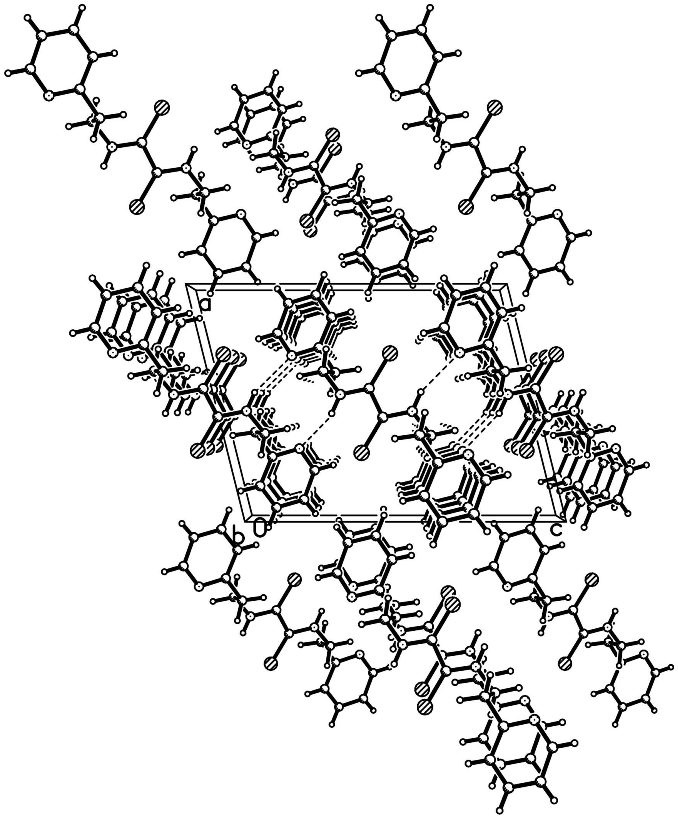


Figure 2
Packing of the molecules in the crystal structure of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$N2$^{\rm ii}$	0.85 (1)	2.31 (1)	3.0503 (18)	146 (1)

Symmetry code: (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

The H atom of the NH group was located in a difference Fourier map and refined isotropically, subject to the restraint N—H = 0.85 (1) Å. All C-bound H atoms were positioned geometrically and included in the refinement in the riding-model approximation (C—H = 0.93–0.97 Å). $U_{\rm iso}$(H) values were set to $1.2U_{\rm eq}$(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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