Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis(benzamidothiocarbonyl)hydrazine dimethyl sulfoxide disolvate

Bohari M. Yamin* and M. Sukeri M. Yusof

School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail: bohari@pkrisc.cc.ukm.my

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.135
Data-to-parameter ratio = 18.5

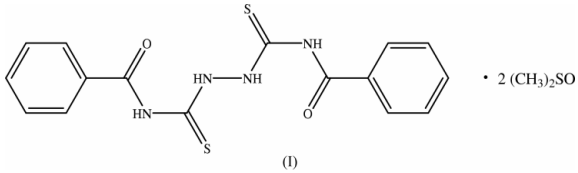
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 February 2003
Accepted 14 February 2003
Online 28 February 2003

In the title compound, $C_{16}H_{14}N_4O_2S_2\cdot 2C_2H_6OS$, the molecule is discrete and centrosymmetric. The benzoylthiuourea and benzoyl groups are *cis* and *trans*, respectively, to the thiourea S atom across the C—N bonds. In the crystal structure, the molecule is linked to the solvent DMSO molecules by N—H···O hydrogen bonds.

Comment

Benzoylthiourea derivatives can be synthesized from the reaction between benzoylisothiocyanate and amine compounds. Consequently, with a diamine, a bis(benzoylthiourea) derivative can be obtained. The title compound, (I), is a simple analogue of the derivative when hydrazine is used. The molecular structure and bond dimensions of (I) are in agreement with other benzoylthiourea derivatives, such as *N*-phenyl-*N*′-benzoylthiourea (Yamin & Yusof, 2003; Usman *et al.*, 2002; Kaminsky *et al.*, 2001). However, the molecule is centrosymmetric about the mid-point of the N2—N2^{i} bond (Fig. 1 and Table 1; symmetry codes are defined in Table 1). The benzoylthiourea and benzoyl groups are *cis* and *trans* to atom S1 across the C—N bonds. The central bis-thiourea fragment, C6 to C6*A*, and the phenyl moieties are planar. The whole molecule looks like a wing with a dihedral angle between the central thiourea and each phenyl ring of 35.27 (9)°.



(I)

The presence of intramolecular hydrogen bonding, N2—H2*A*···O1 and N2—H2*A*···S1^{i}, results in the formation of two pseudo-six-membered (N2/C8/N1/C7/O1/H2*A*) and five-membered rings (S1/C8/N2/N2A/H2*A*^{i}) (Table 2). In the crystal structure, the molecule is linked to the solvent dimethyl sulfoxide (DMSO) molecules by N1—H1*A*···O2^{ii} intermolecular hydrogen bonds (Fig. 2 and Table 2).

Experimental

A solution of hydrazine (0.50 g, 9.9 mmol) in acetone (50 ml) was added dropwise to 50 ml of an acetone solution containing an equimolar amount of benzoylisothiocyanate in a two-neck round-bottomed flask. The solution was refluxed for about 2 h and then cooled in ice. The white precipitate was filtered off and washed with ethanol–distilled water, then dried in a vacuum (yield 80%). Recrystallization from DMSO yielded single crystals of (I) suitable for X-ray analysis.

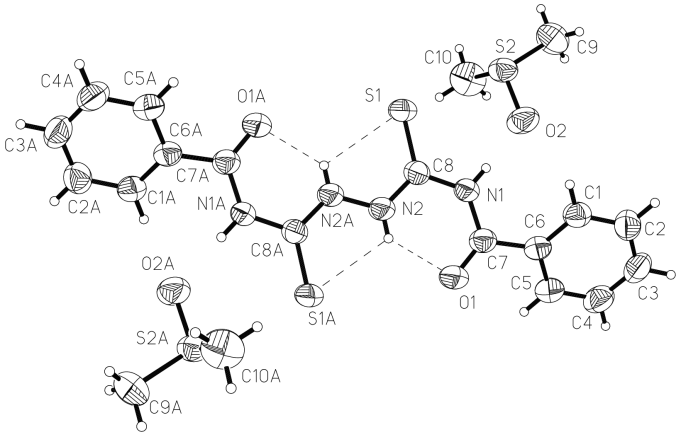


Figure 1
The molecular structure of the title compound, (I), shown with 50% probability displacement ellipsoids. Atoms labelled with a suffix 'A' are generated by the symmetry code $-x, -y, -z$.

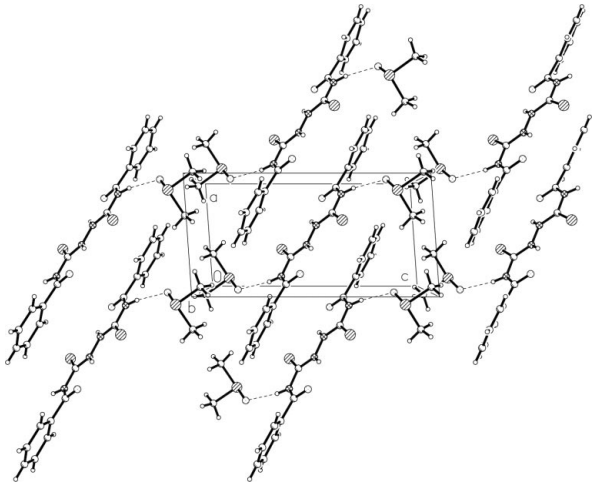


Figure 2
Packing diagram of (I), viewed down the *b* axis. The dashed lines denote N—H···O hydrogen bonds.

Crystal data

$C_{16}H_{14}N_4O_2S_2{\cdot}2C_2H_6OS$
$M_r = 514.70$
Monoclinic, $P2_1/n$
$a = 6.4127\ (11)$ Å
$b = 15.486\ (3)$ Å
$c = 12.805\ (2)$ Å
$\beta = 93.971\ (3)^\circ$
$V = 1268.6\ (4)$ Å^3
$Z = 2$
$D_x = 1.347$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1464 reflections
$\theta = 2.1$–27.5°
$\mu = 0.41$ mm^{-1}
$T = 273\ (2)$ K
Block, colourless
0.56 × 0.34 × 0.33 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.804$, $T_{max} = 0.877$
7458 measured reflections
2862 independent reflections
2008 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.5^\circ$
$h = -6 \rightarrow 8$
$k = -20 \rightarrow 18$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.135$
$S = 1.03$
2862 reflections
155 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.1638P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C8—N2	1.323 (3)	C9—S2	1.780 (3)
C8—N1	1.386 (3)	C10—S2	1.764 (3)
C8—S1	1.664 (2)	N2—N2^{i}	1.372 (4)
C7—O1	1.224 (3)	O2—S2	1.5026 (19)
C7—N1	1.380 (3)		
N2—C8—N1	116.0 (2)	N1—C8—S1	120.94 (18)
N2—C8—S1	123.1 (2)	O1—C7—N1	122.5 (2)

Symmetry codes: (i) $-x, -y, -z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.78 (3)	2.01 (3)	2.593 (3)	132 (2)
N2—H2A···S1^{i}	0.78 (3)	2.53 (3)	2.920 (2)	113 (2)
N1—H1A···O2^{ii}	0.84 (3)	2.01 (3)	2.843 (3)	169 (3)

Symmetry codes: (i) $-x, -y, -z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

H atoms bonded to N atoms were located from difference maps and refined isotropically. H atoms bonded to C atoms were fixed geometrically at their ideal positions and allowed to ride on the parent atoms, with C—H = 0.97 Å.

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, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

The authors thank the Malaysian Government and Universiti Kebangsaan Malaysia for research grant IRPA No. 09-02-02-0163.

References

Kaminsky, W., Golberg, K. I. & West, D. X. (2001). *J. Mol. Struct.* **605**, 9–15.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Usman, A., Razak, I. A., Satar, S., Kadir, M. A., Yamin, B. M & Fun, H.-K. (2002). *Acta Cryst.* E**58**, o656–o658.
Yamin, B. M. & Yusof, M. S. M. (2003). *Acta Cryst*, E**59**, o151–o152.