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2-(3-Benzoylthioureido)ethanoic acid dimethyl sulfoxide solvate

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In the title compound, $C_{10}H_{10}N_2O_3S\cdot C_2H_6SO$, the ethanoic acid fragment and benzene ring make dihedral angles of 3.61 (9) and 20.77 (9)°, respectively, with the central thiourea N_2CS group. In the crystal structure, an intermolecular O—H···O hydrogen bond exists between the hydroxy group of the ethanoic acid fragment and the O atom of the dimethyl sulfoxide solvent molecule.

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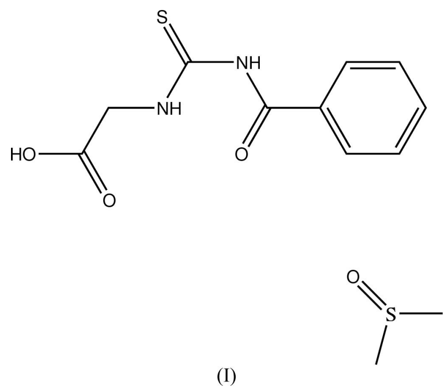
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.077
Data-to-parameter ratio = 14.8

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

Comment

The reaction of benzoyl isothiocyanate with β-alanine was found to give the benzoylthiourea derivative of the amino acid 3-(3-benzoylthioureido)propionic acid (Yusof & Yamin, 2003). In the same way, 2-(3-benzoylthioureido)ethanoic acid has been obtained from the reaction with glycine and recrystallized from DMSO (dimethyl sulfoxide) to provide the title compound, (I). The molecule of 2-(3-benzoylthioureido)ethanoic acid maintains the *cis–trans* configuration with respect to the positions of the ethanoic acid and benzoyl groups relative to the thiono C═S group across the C8—N2 and C8—N1 bonds, respectively (Fig. 1). The geometric dimensions of the molecule (Table 1) are in normal ranges (Allen *et al.*, 1987) and correspond to those observed in 3-(3-benzoylthioureido)propionic acid (Yusof & Yamin, 2003). The central thiourea S1/N1/N2/C8 and ethanoic acid C9/C10/O2/O3 fragments are essentially planar, with maximum deviations of 0.012 (2) Å (for atom N1) and 0.037 (2) Å (for atom O3), respectively. The central thiourea fragment makes dihedral angles of 3.61 (9) and 20.77 (9)° with the ethanoic acid fragment and phenyl ring, respectively. The dihedral angle between the phenyl ring and ethanoic acid fragment is 17.25 (11)°. There are two intramolecular N—H···O hydrogen bonds (Table 2), resulting in the formation of pseudo-five- and six-membered rings, N2—H2*A*—O2—C10—C9 and N2—H2*A*—O1—C7—N1—C8, respectively (Fig. 1).



(I)

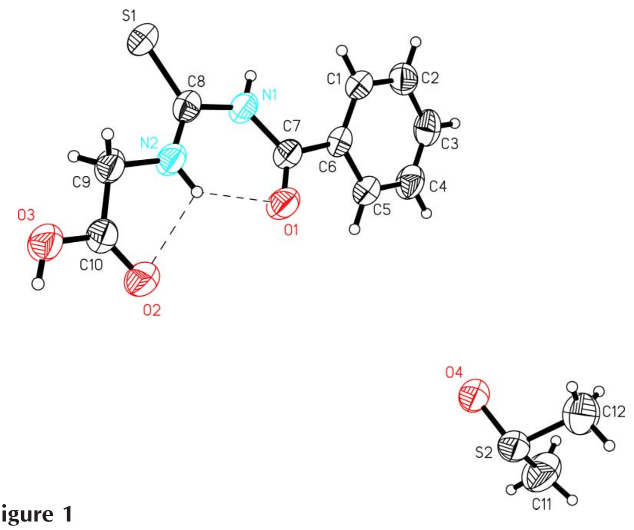


Figure 1
View of (I), with 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

In the crystal structure, each thiourea molecule interacts with the dimethylsulfoxide solvent molecule *via* an intermolecular O—H$\cdots$O hydrogen bond (Table 2) between the hydroxy group of the ethanoic acid fragment and the O atom of the dimethyl sulfoxide solvent molecule (Fig. 2).

Experimental

The title compound, (I), was synthesized following the known procedure for the synthesis of benzoylthiourea derivatives (Yusof & Yamin, 2003). Yellow crystals were obtained by recrystallization from DMSO (m.p. 481–482 K). Microelemental analysis found: C 44.9, H 4.76, N 8.69, O 9.4, S 19.6%; calculated: C 45.45, H 5.00, N 8.84, O 10.11, S 20.28%.

Crystal data

$C_{10}H_{10}N_2O_3S\cdot C_2H_6OS$
$M_r = 316.39$
Monoclinic, $P2_1$
$a = 10.074$ (2) Å
$b = 5.6289$ (12) Å
$c = 13.969$ (3) Å
$\beta = 110.074$ (4)°
$V = 744.0$ (3) Å^3
$Z = 2$

$D_x = 1.412$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 734 reflections
$\theta = 1.6$–26.0°
$\mu = 0.37$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.44 × 0.34 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.854$, $T_{max} = 0.940$
4157 measured reflections

2725 independent reflections
2537 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 26.0°$
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 6$
$l = -15 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.077$
$S = 1.07$
2725 reflections
184 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 0.1167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Absolute structure: Flack (1983), with 1117 Friedel pairs
Flack parameter: 0.06 (7)

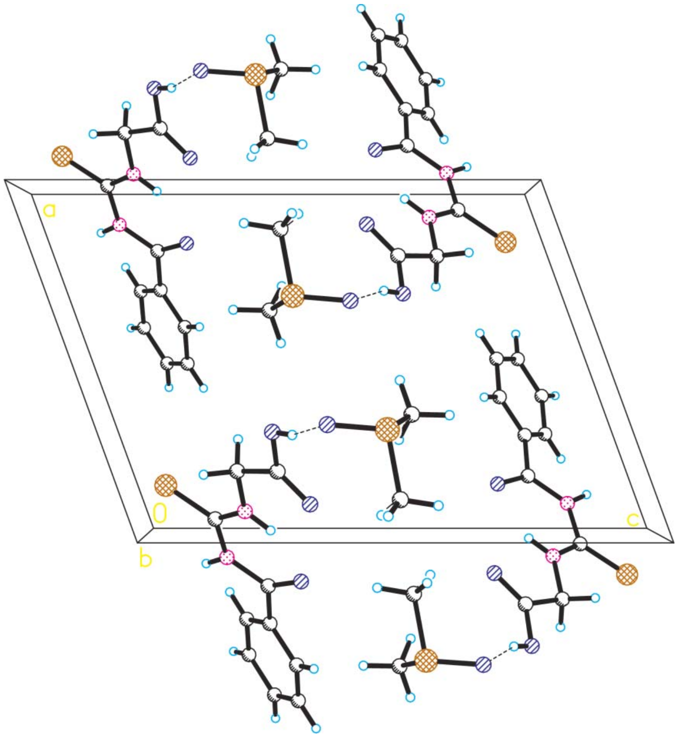


Figure 2
Packing diagram, viewed down the *b* axis. Dashed lines denote O—H$\cdots$O hydrogen bonds.

Table 1
Selected interatomic distances (Å).

S1—C8	1.670 (2)	N1—C7	1.381 (3)
O1—C7	1.219 (3)	N1—C8	1.389 (3)
O2—C10	1.206 (3)	N2—C8	1.311 (3)
O3—C10	1.294 (3)	N2—C9	1.444 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$A\cdots$O1	0.86	1.97	2.645 (3)	134
N2—H2$A\cdots$O2	0.86	2.35	2.700 (3)	105
O3—H3$A\cdots$O4^{i}	0.82	1.76	2.562 (3)	164

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

After their location in a difference Fourier map, all H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å, O—H = 0.82 Å and N—H = 0.86 Å, and with U_{iso}(H) = 1.2–1.5U_{eq}(parent atom).

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, 2003).

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