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1-(2-Hydroxyethyl)-3-phenylthiourea

Antar A. Abdelhamid,^a Shaaban K. Mohamed,^a Mehmet Akkurt,^{b*} Kuldip Singh^c and Herman Potgieter^d

^aChemistry and Environmental Science Division, School of Science & The Environment, Manchester Metropolitan University, M1 5GD, England, ^bDepartment of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, ^cDepartment of Chemistry, University of Leicester, Leicester, England, and ^dSchool of Research, Enterprise & Innovation, Manchester Metropolitan University, M1 5GD, England

Correspondence e-mail: akkurt@erciyes.edu.tr

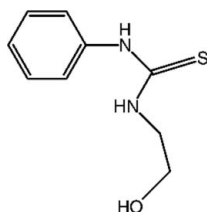
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.110; data-to-parameter ratio = 17.3.

The title compound, $\text{C}_9\text{H}_{12}\text{N}_2\text{OS}$, was obtained unexpectedly in a multicomponent reaction of an equimolar ratio of phenyl isothiocyanate, malononitrile and aminoethanol. The $-\text{C}(\text{H}_2)-\text{N}(\text{H})-(\text{C}=\text{S})-\text{N}(\text{H})-$ methylthiourea-methane group is almost normal to the phenyl ring, with a dihedral angle of $71.13(9)^\circ$. The $\text{N}-\text{C}-\text{C}-\text{O}$ torsion angle is $72.8(2)^\circ$. In the crystal, molecules are connected by $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{S}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For the biological activity of thioureas, see: Kilcigil & Altanlar (2006); Struga *et al.* (2007); Desai *et al.* (2007); Patel *et al.* (2007); Arslan *et al.* (2006); Katritzky & Gordeev (1991). For standard bond lengths, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$\text{C}_9\text{H}_{12}\text{N}_2\text{OS}$
 $M_r = 196.28$
 Tetragonal, $I4_1/a$
 $a = 26.170(4)$ Å
 $c = 5.7775(16)$ Å
 $V = 3956.8(16)$ Å³

$Z = 16$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 150$ K
 $0.27 \times 0.09 \times 0.08$ mm

Data collection

Bruker APEX 2K CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.926$, $T_{\max} = 0.977$

15367 measured reflections
 2056 independent reflections
 1540 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.095$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.110$
 $S = 0.98$
 2056 reflections

119 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1
 Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{S1}^{\text{i}}$	0.86	2.54	3.3676 (18)	163
$\text{O1}-\text{H1B}\cdots\text{S1}^{\text{ii}}$	0.82	2.40	3.2137 (18)	169
$\text{N2}-\text{H2A}\cdots\text{O1}^{\text{iii}}$	0.86	2.15	2.875 (2)	142

Symmetry codes: (i) $-x + 1, -y, -z + 1$; (ii) $x, y, z - 1$; (iii) $y + \frac{1}{4}, -x + \frac{3}{4}, -z - \frac{1}{4}$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2342).

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1-(2-Hydroxyethyl)-3-phenylthiourea

Antar A. Abdelhamid, Shaaban K. Mohamed, Mehmet Akkurt, Kuldip Singh and Herman Potgieter

S1. Comment

Substituted thioureas are important organic compounds prompting the interest of chemists due to their broad spectrum of biological activities such as anti-*HIV*, antiviral, *HDL*-elevating, antibacterial, analgesic properties (Kilcigil & Altanlar, 2006; Struga *et al.*, 2007; Desai *et al.*, 2007; Patel *et al.*, 2007) and acting as fungicides (Arslan *et al.*, 2006). Industrially, thioureas act as corrosion inhibitors, antioxidant, and are polymer components (Katritzky & Gordeev, 1991). The title compound has been obtained as an unexpected product from our multicomponent reaction techniques of phenylisothiocyanate, malononitrile and amino ethanol under conventional heat.

In the title compound **I** (Fig. 1), the methylthiourea-methane group ($-C8(H_2)-N2(H)-(C7=S1)-N1(H)-$) makes a dihedral angle of $71.13 (9)^\circ$ with the C1-C6 phenyl ring. The N2-C8-C9-O1 torsion angle is $72.8 (2)^\circ$. The bond lengths and angles in **I** are in the normal range (Allen *et al.*, 1987).

Intramolecular C8-H8A \cdots S1 contact help to stabilize the molecular conformation of **I**, generating a C(5) loop (Bernstein *et al.*, 1995; Etter *et al.*, 1990). The crystal packing is stabilized by N-H \cdots O, O-H \cdots S and N-H \cdots O intermolecular hydrogen bonds, forming a three-dimensional network (Table 1, Fig. 2).

S2. Experimental

The 1-(2-hydroxyethyl)-3-phenylthiourea has been formed as an unexpected product from a multicomponent reaction of an equimolar ratio of phenylisothiocyanate, malononitrile and amino ethanol. The reaction mixture was heated at 374 K in dioxane (30 ml) for 3 h, then cooled at room temperature to afford a solid precipitate. The product was filtered off, washed with cold ethanol and recrystallized from ethanol. Colourless needles in a spiky shape have been isolated on slow evaporation of a diluted ethanol of the product (yield 39%, m.p. 393 K).

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with C-H = 0.93 \AA for aromatic, C-H = 0.97 \AA for methylene, O-H = 0.82 \AA for hydroxyl and N-H = 0.86 \AA for amine H atoms, and with $U_{iso}(H) = 1.2(1.5)U_{eq}(C,N,O)$.

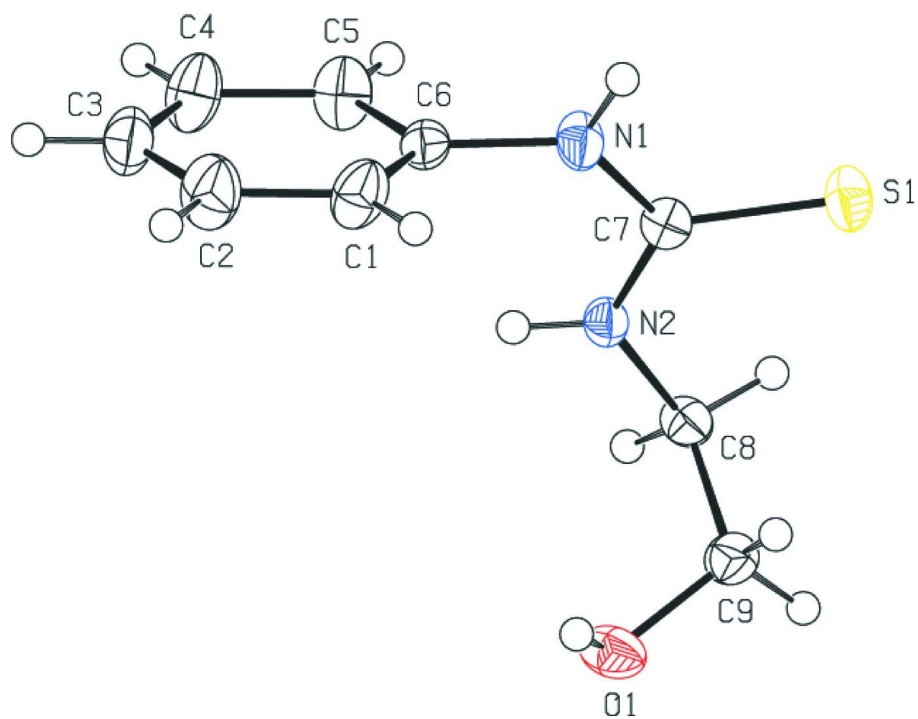


Figure 1

The molecular structure of **I**, showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

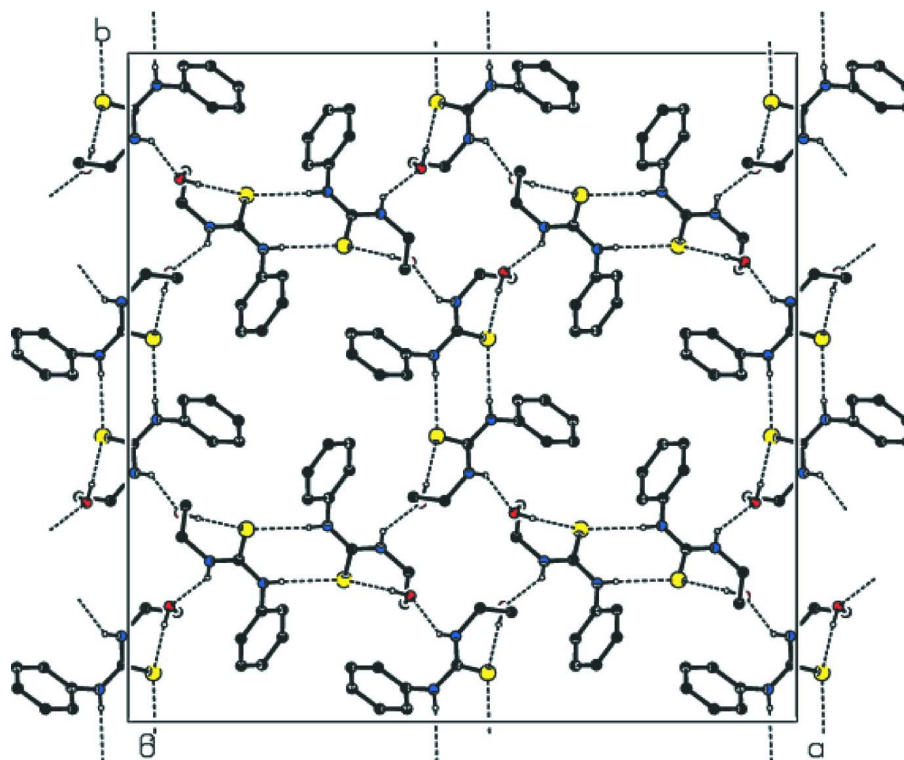


Figure 2

View of the crystal packing and hydrogen bonding of **I** down the *a* axis. H atoms not involved in hydrogen bonds have been omitted for clarity.

1-(2-Hydroxyethyl)-3-phenylthiourea

Crystal data

$C_9H_{12}N_2OS$

$M_r = 196.28$

Tetragonal, $I4_1/a$

Hall symbol: $-I\ 4ad$

$a = 26.170(4)\ \text{\AA}$

$c = 5.7775(16)\ \text{\AA}$

$V = 3956.8(16)\ \text{\AA}^3$

$Z = 16$

$F(000) = 1664$

$D_x = 1.318\ \text{Mg m}^{-3}$

Melting point: 393 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 800 reflections

$\theta = 3.5\text{--}28.2^\circ$

$\mu = 0.29\ \text{mm}^{-1}$

$T = 150\ \text{K}$

Needle, colourless

$0.27 \times 0.09 \times 0.08\ \text{mm}$

Data collection

Bruker APEX 2K CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.926$, $T_{\max} = 0.977$

15367 measured reflections

2056 independent reflections

1540 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.095$

$\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 1.6^\circ$

$h = -32 \rightarrow 32$

$k = -32 \rightarrow 32$

$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.048$	H-atom parameters constrained
$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2]$
$S = 0.98$	where $P = (F_o^2 + 2F_c^2)/3$
2056 reflections	$(\Delta/\sigma)_{\max} = 0.001$
119 parameters	$\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All s.u.'s are estimated from the variances of the (full) variance-covariance matrix. The cell s.u.'s are taken into account in the estimation of distances, angles and torsion angles.

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating R -factor *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.53773 (2)	0.07294 (2)	0.52460 (10)	0.0281 (2)
O1	0.56205 (6)	0.17228 (6)	-0.1675 (3)	0.0322 (5)
N1	0.45696 (7)	0.04810 (6)	0.2790 (3)	0.0269 (6)
N2	0.49000 (6)	0.12770 (6)	0.1987 (3)	0.0215 (5)
C1	0.41711 (9)	0.01902 (9)	-0.0757 (4)	0.0352 (8)
C2	0.37649 (9)	0.01894 (10)	-0.2287 (4)	0.0416 (9)
C3	0.33488 (9)	0.04979 (9)	-0.1906 (4)	0.0365 (8)
C4	0.33371 (9)	0.08094 (10)	-0.0010 (4)	0.0394 (9)
C5	0.37390 (9)	0.08096 (9)	0.1544 (4)	0.0346 (8)
C6	0.41558 (8)	0.05020 (8)	0.1157 (4)	0.0237 (7)
C7	0.49204 (8)	0.08449 (8)	0.3204 (4)	0.0216 (7)
C8	0.52638 (8)	0.16964 (8)	0.2207 (4)	0.0231 (7)
C9	0.57332 (8)	0.16375 (9)	0.0697 (4)	0.0282 (7)
H1	0.44530	-0.00190	-0.10200	0.0420*
H1A	0.45960	0.02050	0.35900	0.0320*
H1B	0.55420	0.14510	-0.22890	0.0480*
H2	0.37740	-0.00210	-0.35820	0.0500*
H2A	0.46550	0.13110	0.10050	0.0260*
H3	0.30760	0.04950	-0.29360	0.0440*
H4	0.30580	0.10220	0.02370	0.0470*
H5	0.37270	0.10170	0.28490	0.0410*
H8A	0.53710	0.17210	0.38100	0.0280*
H8B	0.50930	0.20130	0.18060	0.0280*
H9A	0.59920	0.18790	0.11950	0.0340*
H9B	0.58700	0.12960	0.08840	0.0340*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0314 (3)	0.0206 (3)	0.0322 (3)	-0.0001 (2)	-0.0121 (3)	0.0010 (2)
O1	0.0448 (10)	0.0284 (9)	0.0234 (9)	-0.0100 (8)	0.0032 (8)	-0.0016 (7)
N1	0.0285 (10)	0.0191 (9)	0.0332 (11)	-0.0045 (8)	-0.0107 (8)	0.0074 (8)
N2	0.0214 (9)	0.0219 (9)	0.0211 (10)	-0.0023 (7)	-0.0036 (7)	0.0015 (7)
C1	0.0253 (12)	0.0417 (15)	0.0385 (14)	0.0047 (11)	-0.0008 (11)	-0.0120 (12)
C2	0.0388 (15)	0.0516 (16)	0.0343 (15)	0.0003 (12)	-0.0058 (12)	-0.0151 (12)
C3	0.0292 (13)	0.0402 (15)	0.0402 (15)	-0.0034 (11)	-0.0118 (11)	0.0027 (12)
C4	0.0291 (13)	0.0384 (14)	0.0508 (17)	0.0090 (11)	-0.0089 (12)	-0.0073 (12)
C5	0.0362 (14)	0.0298 (13)	0.0377 (15)	0.0049 (11)	-0.0063 (11)	-0.0102 (11)
C6	0.0233 (11)	0.0201 (11)	0.0278 (12)	-0.0053 (9)	-0.0038 (9)	0.0052 (9)
C7	0.0233 (11)	0.0191 (11)	0.0223 (12)	0.0014 (9)	0.0007 (9)	-0.0033 (9)
C8	0.0291 (12)	0.0188 (11)	0.0214 (12)	-0.0039 (9)	-0.0015 (9)	-0.0001 (9)
C9	0.0267 (12)	0.0325 (13)	0.0255 (13)	-0.0068 (10)	-0.0002 (10)	-0.0011 (10)

Geometric parameters (Å, °)

S1—C7	1.707 (2)	C4—C5	1.383 (3)
O1—C9	1.420 (3)	C5—C6	1.374 (3)
O1—H1B	0.8200	C8—C9	1.515 (3)
N1—C6	1.437 (3)	C1—H1	0.9300
N1—C7	1.344 (3)	C2—H2	0.9300
N2—C7	1.333 (3)	C3—H3	0.9300
N2—C8	1.459 (3)	C4—H4	0.9300
N1—H1A	0.8600	C5—H5	0.9300
N2—H2A	0.8600	C8—H8A	0.9700
C1—C2	1.383 (3)	C8—H8B	0.9700
C1—C6	1.375 (3)	C9—H9A	0.9700
C2—C3	1.373 (3)	C9—H9B	0.9700
C3—C4	1.366 (3)		
C9—O1—H1B	109.00	C2—C1—H1	120.00
C6—N1—C7	127.18 (17)	C6—C1—H1	120.00
C7—N2—C8	124.50 (17)	C1—C2—H2	120.00
C6—N1—H1A	116.00	C3—C2—H2	120.00
C7—N1—H1A	116.00	C2—C3—H3	120.00
C8—N2—H2A	118.00	C4—C3—H3	120.00
C7—N2—H2A	118.00	C3—C4—H4	120.00
C2—C1—C6	119.5 (2)	C5—C4—H4	120.00
C1—C2—C3	120.4 (2)	C4—C5—H5	120.00
C2—C3—C4	119.8 (2)	C6—C5—H5	120.00
C3—C4—C5	120.3 (2)	N2—C8—H8A	109.00
C4—C5—C6	119.9 (2)	N2—C8—H8B	109.00
N1—C6—C1	118.90 (19)	C9—C8—H8A	109.00
C1—C6—C5	120.1 (2)	C9—C8—H8B	109.00
N1—C6—C5	120.9 (2)	H8A—C8—H8B	108.00

S1—C7—N1	118.42 (16)	O1—C9—H9A	109.00
N1—C7—N2	118.68 (19)	O1—C9—H9B	109.00
S1—C7—N2	122.89 (16)	C8—C9—H9A	109.00
N2—C8—C9	113.75 (18)	C8—C9—H9B	109.00
O1—C9—C8	111.81 (17)	H9A—C9—H9B	108.00
C6—N1—C7—S1	179.93 (17)	C2—C1—C6—N1	177.1 (2)
C7—N1—C6—C1	111.2 (3)	C2—C1—C6—C5	0.1 (3)
C7—N1—C6—C5	-71.9 (3)	C1—C2—C3—C4	0.3 (4)
C6—N1—C7—N2	-0.8 (3)	C2—C3—C4—C5	-1.0 (4)
C7—N2—C8—C9	86.7 (3)	C3—C4—C5—C6	1.2 (4)
C8—N2—C7—S1	1.1 (3)	C4—C5—C6—N1	-177.6 (2)
C8—N2—C7—N1	-178.12 (19)	C4—C5—C6—C1	-0.7 (3)
C6—C1—C2—C3	0.1 (4)	N2—C8—C9—O1	72.8 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots S1 ⁱ	0.86	2.54	3.3676 (18)	163
O1—H1B \cdots S1 ⁱⁱ	0.82	2.40	3.2137 (18)	169
N2—H2A \cdots O1 ⁱⁱⁱ	0.86	2.15	2.875 (2)	142
C8—H8A \cdots S1	0.97	2.72	3.094 (2)	103

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x, y, z-1$; (iii) $y+1/4, -x+3/4, -z-1/4$.