Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-(4-Ethoxyphenyl)thiourea

Yan-Fang Lin, Guang-Xiang Zhong, Feng Xu and Wei-Xiao Hu*

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310032, People's Republic of China
Correspondence e-mail: huyang@mail.hz.zj.cn

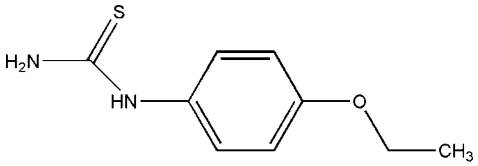
Received 13 July 2007; accepted 16 July 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.094; data-to-parameter ratio = 17.5.

In the title molecule, $C_9H_{12}N_2OS$, the benzene ring and mean plane of the thiourea fragment [–N—C(=S)—N] make a dihedral angle of 59.19 (3)°. In the crystal structure, weak intermolecular N—H···S and N—H···O hydrogen bonds link the molecules into a three-dimensional network.

Related literature

For the synthesis of the title compound, see: Liu *et al.* (1994). For details of the pharmacological properties of thiazolone derivatives, see: Mane & Ingle (1983).



Experimental

Crystal data

$C_9H_{12}N_2OS$
M_r = 196.27
Monoclinic, $C2/c$
a = 14.8450 (19) Å
b = 8.2482 (11) Å
c = 16.860 (2) Å
β = 97.489 (1)°
V = 2046.8 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 (2) K
0.30 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.921, T_{max} = 0.949
6278 measured reflections
2313 independent reflections
2082 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.094
S = 1.06
2313 reflections
132 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1X···S1^{i}	0.855 (13)	2.523 (14)	3.3594 (11)	166.0 (14)
N2—H2X···O1^{ii}	0.874 (14)	2.386 (16)	3.1105 (15)	140.5 (14)
N2—H2Y···S1^{iii}	0.836 (14)	2.578 (14)	3.4008 (13)	168.3 (15)

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, -y+\frac{5}{2}, -z+1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2005); 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, 2005); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2277).

References

Bruker (2004). *APEX2*. Version 1.027. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, B., Gao, H. Q. & Zhou, X. J. (1994). *Hua Xue Tong Bao*, **5**, 42–43.
Mane, R. A. & Ingle, D. B. (1983). *Indian J. Chem. Sect. B*. **22**, 690–692.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

doi:10.1107/S1600536807034654
© 2007 International Union of Crystallography

supplementary materials

Acta Cryst. (2007). E**63**, o3549 [**doi:10.1107/S1600536807034654**]

N-(4-Ethoxyphenyl)thiourea

Y.-F. Lin, G.-X. Zhong, F. Xu and W.-X. Hu

Comment

Thiazolone derivatives have potential antimicrobial and antitumour properties (Mane *et al.*, 1983). The title compound, (*p*-ethoxyphenyl)thiourea (I), is an important intermediate in the synthesis of thiazolone derivatives. In our work, we present its crystal structure.

In (I) (Fig. 1), the benzene ring is twisted out of –N1—C9(=S1)—N2 and –O1—C7—C8 planes by the diherdral angles of 59.19 (3)° and 12.26 (3)°, respectively. In the crystal, the weak intermolecular N—H···S and N—H···O hydrogen bonds link the molecules into three-dimensional hydrogen-bonding network (Table 1, Fig. 2).

Experimental

A mixture of 4-Ethoxyaniline(13.7 g,0.1 mol), 36% aqueous HCl(10.1 g,0.1 mol) and ammonium thiocyanate(7.6 g,0.1 mol) was refluxed in water(30 ml) for 4 hrs, then a white precipitate was observed and filtered. The solid was recrystallized from water to give the pure product. The pure product was dissolved in water evaporated gradually at room temperature to afford single crystals of (I). (m.p. 450–451 K). ^{1}HNMR($CDCl_3$)σp.p.m.:7.15(d,2*H*,J=8.4 Hz),6.92(d,2*H*,J=8.4 Hz), 4.03(q,2*H*,CH_2), 1.42(t,3*H*,CH_3). MS.(m/*z*,%).196(*M*,100),163 (35),154 (70),108 (95),80 (35),60 (25).

Refinement

The atoms H1X, H2X and H2Y were located in difference Fourier maps and refined isotropically with the N—H bond restraint of 0.86 (2) Å. Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined to fit the electron density [U_{iso}(H) = 1.5U_{eq}(C)]. Other H atoms were placed in calculated positions, with C—H = 0.93 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

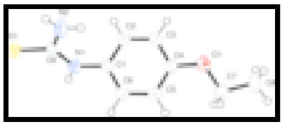


Fig. 1. The molecular structure of (I), shown with 30% probability displacement ellipsoids.

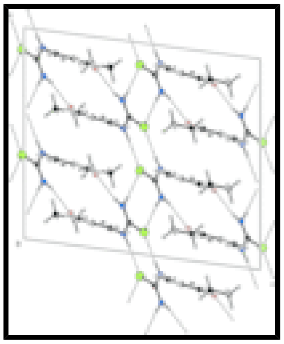


Fig. 2. Crystal structure of (I), viewed down the *b* axis. N—H···O and N—H···S hydrogen bondings are shown as dashed lines.

N-(4-Ethoxyphenyl)thiourea

Crystal data

$C_9H_{12}N_2OS$

$M_r = 196.27$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 14.8450$ (19) Å

$b = 8.2482$ (11) Å

$c = 16.860$ (2) Å

$\beta = 97.4890$ (10)°

$V = 2046.8$ (4) Å^3

$Z = 8$

$F_{000} = 832$

$D_x = 1.274$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3819 reflections

$\theta = 2.8–28.1$°

$\mu = 0.28$ mm^{-1}

$T = 293$ (2) K

Prismatic, colourless

0.30 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

φ and ω scans

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

$T_{\min} = 0.921$, $T_{\max} = 0.949$

6278 measured reflections

2313 independent reflections

2082 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\max} = 27.5$°

$\theta_{\min} = 2.4$°

$h = -17 \rightarrow 19$

$k = -10 \rightarrow 10$

$l = -19 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.094$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.628P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
2313 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
132 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
3 restraints	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0053 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness 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 threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.60518 (2)	1.19209 (4)	0.507891 (19)	0.04179 (14)
O1	0.64641 (7)	0.45160 (12)	0.19824 (6)	0.0506 (3)
N1	0.58619 (7)	0.92414 (14)	0.42155 (7)	0.0415 (3)
H1X	0.5372 (10)	0.9124 (19)	0.4426 (9)	0.047 (4)*
N2	0.71911 (8)	1.06437 (17)	0.41752 (8)	0.0525 (3)
H2X	0.7399 (11)	0.9898 (19)	0.3879 (10)	0.058 (5)*
H2Y	0.7562 (10)	1.1336 (19)	0.4382 (10)	0.052 (4)*
C1	0.60403 (8)	0.80089 (15)	0.36640 (7)	0.0365 (3)
C2	0.61645 (9)	0.84100 (16)	0.28852 (8)	0.0411 (3)
H2	0.6153	0.9492	0.2729	0.049*
C3	0.63050 (9)	0.72167 (16)	0.23422 (7)	0.0415 (3)
H3	0.6393	0.7497	0.1824	0.050*
C4	0.63154 (9)	0.56007 (15)	0.25676 (7)	0.0387 (3)
C5	0.61802 (10)	0.51855 (17)	0.33402 (8)	0.0475 (3)
H5	0.6179	0.4102	0.3493	0.057*
C6	0.60467 (10)	0.63975 (17)	0.38853 (8)	0.0458 (3)
H6	0.5961	0.6120	0.4405	0.055*
C7	0.62819 (11)	0.28480 (17)	0.21063 (10)	0.0515 (4)
H7A	0.5677	0.2719	0.2256	0.062*
H7B	0.6717	0.2415	0.2533	0.062*
C8	0.63544 (13)	0.1967 (2)	0.13409 (10)	0.0625 (4)
H8A	0.5950	0.2450	0.0916	0.094*
H8B	0.6193	0.0849	0.1398	0.094*
H8C	0.6967	0.2035	0.1218	0.094*

C9	0.63966 (8)	1.05019 (15)	0.44530 (7)	0.0360 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0422 (2)	0.0416 (2)	0.0448 (2)	−0.01078 (12)	0.01772 (14)	−0.01294 (12)
O1	0.0699 (7)	0.0407 (5)	0.0453 (5)	−0.0050 (5)	0.0236 (5)	−0.0091 (4)
N1	0.0395 (6)	0.0443 (6)	0.0442 (6)	−0.0121 (5)	0.0187 (5)	−0.0130 (5)
N2	0.0418 (6)	0.0594 (8)	0.0607 (7)	−0.0188 (6)	0.0236 (5)	−0.0276 (6)
C1	0.0340 (6)	0.0407 (6)	0.0359 (6)	−0.0081 (5)	0.0090 (5)	−0.0083 (5)
C2	0.0492 (7)	0.0360 (6)	0.0389 (6)	−0.0021 (5)	0.0085 (5)	−0.0001 (5)
C3	0.0510 (7)	0.0435 (7)	0.0309 (6)	−0.0015 (5)	0.0096 (5)	0.0005 (5)
C4	0.0412 (6)	0.0394 (6)	0.0369 (6)	−0.0037 (5)	0.0105 (5)	−0.0062 (5)
C5	0.0652 (9)	0.0365 (7)	0.0434 (7)	−0.0064 (6)	0.0165 (6)	0.0000 (5)
C6	0.0595 (8)	0.0458 (7)	0.0349 (6)	−0.0098 (6)	0.0159 (6)	−0.0013 (5)
C7	0.0559 (8)	0.0419 (7)	0.0601 (9)	−0.0089 (6)	0.0204 (7)	−0.0099 (6)
C8	0.0717 (11)	0.0540 (9)	0.0624 (10)	−0.0081 (8)	0.0116 (8)	−0.0210 (7)
C9	0.0368 (6)	0.0392 (6)	0.0334 (6)	−0.0065 (5)	0.0096 (4)	−0.0037 (5)

Geometric parameters (Å, °)

S1—C9	1.6989 (12)	C3—C4	1.3855 (18)
O1—C4	1.3708 (15)	C3—H3	0.9300
O1—C7	1.4228 (17)	C4—C5	1.3866 (18)
N1—C9	1.3374 (16)	C5—C6	1.3894 (19)
N1—C1	1.4255 (15)	C5—H5	0.9300
N1—H1X	0.855 (13)	C6—H6	0.9300
N2—C9	1.3297 (16)	C7—C8	1.497 (2)
N2—H2X	0.874 (14)	C7—H7A	0.9700
N2—H2Y	0.836 (14)	C7—H7B	0.9700
C1—C6	1.3802 (19)	C8—H8A	0.9599
C1—C2	1.3895 (18)	C8—H8B	0.9599
C2—C3	1.3785 (18)	C8—H8C	0.9599
C2—H2	0.9300		

C4—O1—C7	118.29 (10)	C4—C5—H5	120.2
C9—N1—C1	126.59 (10)	C6—C5—H5	120.2
C9—N1—H1X	117.9 (11)	C1—C6—C5	120.71 (12)
C1—N1—H1X	115.5 (11)	C1—C6—H6	119.6
C9—N2—H2X	122.4 (11)	C5—C6—H6	119.6
C9—N2—H2Y	118.6 (11)	O1—C7—C8	107.86 (13)
H2X—N2—H2Y	117.1 (16)	O1—C7—H7A	110.1
C6—C1—C2	119.18 (11)	C8—C7—H7A	110.1
C6—C1—N1	120.38 (11)	O1—C7—H7B	110.1
C2—C1—N1	120.36 (11)	C8—C7—H7B	110.1
C3—C2—C1	120.52 (12)	H7A—C7—H7B	108.4
C3—C2—H2	119.7	C7—C8—H8A	109.5
C1—C2—H2	119.7	C7—C8—H8B	109.5
C2—C3—C4	120.13 (11)	H8A—C8—H8B	109.5

C2—C3—H3	119.9	C7—C8—H8C	109.5
C4—C3—H3	119.9	H8A—C8—H8C	109.5
O1—C4—C3	115.32 (11)	H8B—C8—H8C	109.5
O1—C4—C5	124.86 (12)	N2—C9—N1	118.82 (11)
C3—C4—C5	119.83 (12)	N2—C9—S1	120.74 (10)
C4—C5—C6	119.62 (12)	N1—C9—S1	120.42 (9)

C9—N1—C1—C6	123.61 (15)	O1—C4—C5—C6	179.21 (13)
C9—N1—C1—C2	−59.64 (19)	C3—C4—C5—C6	−0.8 (2)
C6—C1—C2—C3	−0.9 (2)	C2—C1—C6—C5	0.4 (2)
N1—C1—C2—C3	−177.68 (12)	N1—C1—C6—C5	177.14 (13)
C1—C2—C3—C4	0.6 (2)	C4—C5—C6—C1	0.5 (2)
C7—O1—C4—C3	−165.98 (13)	C4—O1—C7—C8	171.20 (13)
C7—O1—C4—C5	14.0 (2)	C1—N1—C9—N2	−1.7 (2)
C2—C3—C4—O1	−179.75 (12)	C1—N1—C9—S1	176.65 (10)
C2—C3—C4—C5	0.3 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1X···S1^{i}	0.855 (13)	2.523 (14)	3.3594 (11)	166.0 (14)
N2—H2X···O1^{ii}	0.874 (14)	2.386 (16)	3.1105 (15)	140.5 (14)
N2—H2Y···S1^{iii}	0.836 (14)	2.578 (14)	3.4008 (13)	168.3 (15)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iii) −*x*+3/2, −*y*+5/2, −*z*+1.

Fig. 1

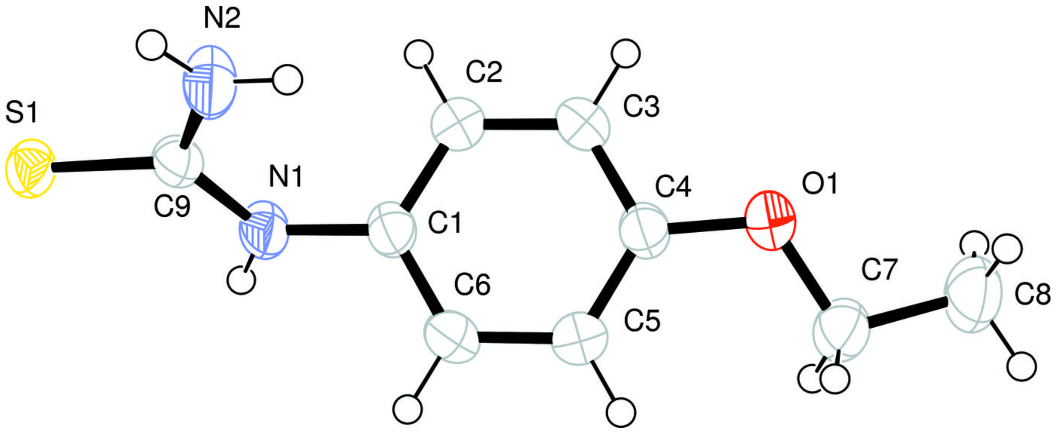


Fig. 2