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4-Ethyl-1-(4-methoxybenzylidene)thiosemicarbazide

Yu-Feng Li[a] and Fang-Fang Jian[b]*

[a]Microscale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China, and [b]Microscale Science Institute, Weifang University, Weifang 261061, People's Republic of China Correspondence e-mail: liyufeng8111@163.com

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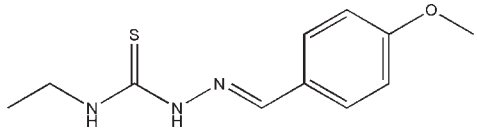
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.053; wR factor = 0.160; data-to-parameter ratio = 20.1.

In the title compound, $C_{11}H_{15}N_3OS$, the dihedral angle between the aromatic ring and the thiourea unit is 4.28 (7)° and an intramolecular N—H···N hydrogen bond generates an S(5) ring. In the crystal, molecules are linked into (001) sheets by N—H···S hydrogen bonds.

Related literature

For background to the reactions and properties of thiosemicarbazones, see: Casas *et al.* (2000); Lobana *et al.* (2009); Quiroga & Ranninger (2004). For a related structure, see: Li & Jian (2010).



Experimental

Crystal data

$C_{11}H_{15}N_3OS$
M_r = 237.32
Orthorhombic, *Pbca*
a = 13.066 (3) Å
b = 10.128 (2) Å
c = 19.224 (4) Å
V = 2543.9 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
22633 measured reflections
2912 independent reflections
2302 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.160
S = 1.05
2912 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···S1^{i}	0.86	2.60	3.4080 (17)	156
N3—H3A···N2	0.86	2.26	2.634 (2)	106
N3—H3A···S1^{ii}	0.86	2.78	3.4670 (17)	137

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5492).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Casas, J. S., Garcia-T, M. S. & Sordo, J. (2000). *Coord. Chem. Rev.* **209**, 197–261.
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* E**66**, o1399.
Lobana, T. S., Khanna, S., Hundal, G., Kaur, P., Thakur, B., Attri, S. & Butcher, R. J. (2009). *Polyhedron*, **28**, 1583–1593.
Quiroga, A. G. & Ranninger, C. N. (2004). *Coord. Chem. Rev.* **248**, 119–133.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o1714 [doi:10.1107/S1600536810022919]

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S1. Comment

Thiosemicarbazones have attracted much attention because they can be utilized as effective ligands to form the compounds with antitumoral drugs. (Quiroga & Ranninger, 2004).They are important versatile coordination agents which have been reported to be coordination compounds (Casas *et al.*, 2000) (Lobana *et al.*, 2009). As part of our search for new thiosemicarbazones compounds we synthesized the title compound (I), and describe its structure here. The dihedral angle between the benzene ring and the thiourea unit is [4.28 (7)°]. Intermolecular N—H···S hydrogen bonds generate chains.

Bond lengths and angles agree with those observed in a related compound (Li & Jian, 2010).

S2. Experimental

A mixture of 4-methoxybenzaldehyde (0.1 mol) and 4-ethylthiosemicarbazide (0.1 mol) was stirred in refluxing ethanol (20 ml) for 2 h to afford the title compound (0.086 mol, yield 86%). Colourless blocks of (I) were obtained by recrystallization from ethanol at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.97 Å, and with U_{iso}(H) = 1.2–1.5U_{eq}(C).

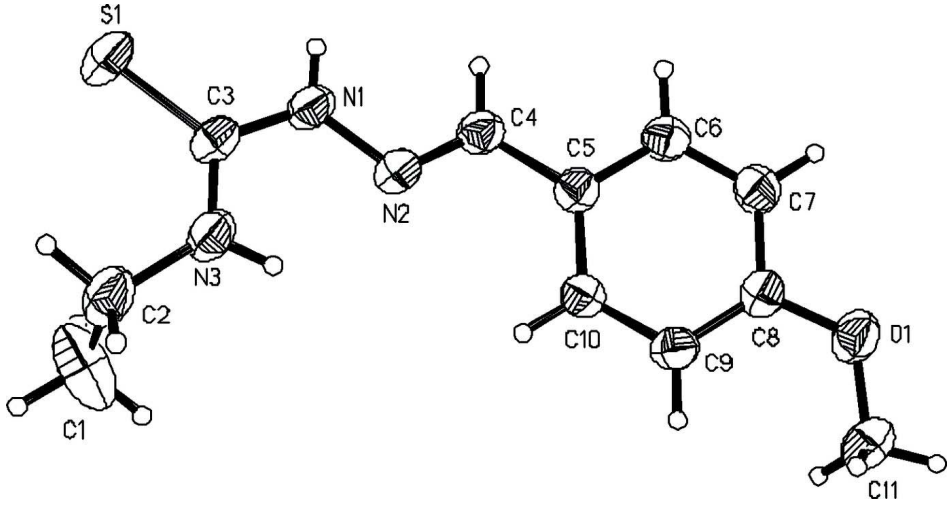

Figure 1

The structure of (I) showing 30% probability displacement ellipsoids.

4-Ethyl-1-(4-methoxybenzylidene)thiosemicarbazide

Crystal data

$C_{11}H_{15}N_3OS$
$M_r = 237.32$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.066$ (3) Å
$b = 10.128$ (2) Å
$c = 19.224$ (4) Å
$V = 2543.9$ (9) Å^3
$Z = 8$
$F(000) = 1008$
$D_x = 1.239$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2302 reflections
$\theta = 2.8$–$25.3°$
$\mu = 0.24$ mm^{-1}
$T = 293$ K
Block, colorless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
22633 measured reflections
2912 independent reflections
2302 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -16 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.160$
$S = 1.05$
2912 reflections
145 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0997P)^2 + 0.2868P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.12047 (4)	1.04218 (4)	0.57631 (3)	0.0768 (2)
N1	0.09565 (11)	0.83299 (14)	0.49802 (8)	0.0648 (4)
H1A	0.0548	0.8818	0.4742	0.078*
N2	0.11117 (10)	0.70368 (14)	0.47913 (8)	0.0598 (3)
C9	0.12596 (13)	0.30355 (18)	0.41061 (9)	0.0637 (4)
H9A	0.1545	0.2367	0.4375	0.076*

C3	0.14420 (12)	0.88334 (15)	0.55374 (9)	0.0596 (4)
N3	0.21067 (12)	0.80566 (14)	0.58550 (9)	0.0719 (4)
H3A	0.2271	0.7331	0.5651	0.086*
O1	0.09603 (11)	0.15651 (14)	0.31258 (7)	0.0803 (4)
C5	0.07418 (12)	0.53144 (16)	0.39807 (8)	0.0576 (4)
C10	0.11699 (13)	0.42999 (18)	0.43678 (9)	0.0621 (4)
H10A	0.1403	0.4476	0.4815	0.075*
C4	0.06596 (13)	0.66625 (17)	0.42425 (9)	0.0626 (4)
H4A	0.0262	0.7267	0.3999	0.075*
C8	0.09189 (12)	0.27751 (17)	0.34373 (9)	0.0621 (4)
C6	0.04087 (15)	0.5032 (2)	0.33114 (10)	0.0719 (5)
H6A	0.0115	0.5696	0.3044	0.086*
C11	0.14227 (19)	0.05082 (19)	0.34942 (13)	0.0866 (6)
H11A	0.1397	−0.0278	0.3216	0.130*
H11B	0.1061	0.0363	0.3922	0.130*
H11C	0.2123	0.0725	0.3594	0.130*
C2	0.25846 (16)	0.8338 (2)	0.65277 (13)	0.0910 (7)
H2B	0.2628	0.9285	0.6594	0.109*
H2C	0.3275	0.7984	0.6532	0.109*
C7	0.05071 (16)	0.3784 (2)	0.30398 (10)	0.0769 (5)
H7A	0.0296	0.3616	0.2586	0.092*
C1	0.1984 (3)	0.7743 (4)	0.71090 (15)	0.1455 (13)
H1B	0.2311	0.7943	0.7544	0.218*
H1C	0.1952	0.6803	0.7049	0.218*
H1D	0.1304	0.8100	0.7108	0.218*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0653 (3)	0.0441 (3)	0.1211 (5)	0.00297 (16)	−0.0100 (2)	−0.0109 (2)
N1	0.0687 (8)	0.0469 (7)	0.0786 (9)	0.0078 (6)	−0.0085 (7)	0.0005 (6)
N2	0.0596 (7)	0.0479 (7)	0.0720 (8)	0.0022 (5)	−0.0015 (6)	−0.0004 (6)
C9	0.0672 (9)	0.0567 (10)	0.0673 (9)	0.0013 (7)	−0.0052 (7)	0.0071 (7)
C3	0.0526 (7)	0.0447 (8)	0.0814 (10)	−0.0024 (6)	0.0014 (7)	−0.0002 (7)
N3	0.0716 (9)	0.0512 (8)	0.0929 (10)	0.0102 (6)	−0.0169 (8)	−0.0137 (7)
O1	0.0857 (8)	0.0647 (8)	0.0905 (9)	0.0036 (6)	−0.0126 (7)	−0.0171 (7)
C5	0.0515 (8)	0.0583 (9)	0.0632 (8)	0.0022 (6)	−0.0032 (6)	−0.0007 (6)
C10	0.0695 (10)	0.0594 (10)	0.0575 (8)	−0.0005 (7)	−0.0070 (7)	0.0017 (7)
C4	0.0604 (9)	0.0585 (9)	0.0688 (9)	0.0067 (7)	−0.0063 (7)	0.0016 (7)
C8	0.0547 (8)	0.0613 (10)	0.0702 (9)	−0.0008 (7)	−0.0022 (7)	−0.0071 (7)
C6	0.0762 (11)	0.0698 (10)	0.0697 (10)	0.0143 (9)	−0.0188 (8)	0.0012 (8)
C11	0.0946 (14)	0.0568 (11)	0.1083 (16)	0.0024 (9)	0.0065 (13)	−0.0021 (10)
C2	0.0766 (12)	0.0677 (12)	0.1285 (18)	0.0107 (9)	−0.0402 (13)	−0.0265 (11)
C7	0.0856 (12)	0.0772 (12)	0.0679 (10)	0.0109 (9)	−0.0231 (9)	−0.0102 (9)
C1	0.120 (2)	0.232 (4)	0.0849 (16)	−0.025 (2)	−0.0161 (16)	−0.032 (2)

Geometric parameters (Å, º)

S1—C3	1.6948 (17)	C10—H10A	0.9300
N1—C3	1.345 (2)	C4—H4A	0.9300
N1—N2	1.3742 (19)	C8—C7	1.385 (3)
N1—H1A	0.8600	C6—C7	1.373 (3)
N2—C4	1.267 (2)	C6—H6A	0.9300
C9—C10	1.381 (2)	C11—H11A	0.9600
C9—C8	1.386 (3)	C11—H11B	0.9600
C9—H9A	0.9300	C11—H11C	0.9600
C3—N3	1.321 (2)	C2—C1	1.492 (4)
N3—C2	1.464 (2)	C2—H2B	0.9700
N3—H3A	0.8600	C2—H2C	0.9700
O1—C8	1.365 (2)	C7—H7A	0.9300
O1—C11	1.419 (3)	C1—H1B	0.9600
C5—C10	1.387 (2)	C1—H1C	0.9600
C5—C6	1.388 (2)	C1—H1D	0.9600
C5—C4	1.459 (2)		
C3—N1—N2	120.14 (13)	C7—C8—C9	119.75 (16)
C3—N1—H1A	119.9	C7—C6—C5	120.86 (16)
N2—N1—H1A	119.9	C7—C6—H6A	119.6
C4—N2—N1	115.87 (14)	C5—C6—H6A	119.6
C10—C9—C8	119.16 (16)	O1—C11—H11A	109.5
C10—C9—H9A	120.4	O1—C11—H11B	109.5
C8—C9—H9A	120.4	H11A—C11—H11B	109.5
N3—C3—N1	116.89 (14)	O1—C11—H11C	109.5
N3—C3—S1	124.54 (13)	H11A—C11—H11C	109.5
N1—C3—S1	118.51 (12)	H11B—C11—H11C	109.5
C3—N3—C2	124.95 (15)	N3—C2—C1	111.03 (19)
C3—N3—H3A	117.5	N3—C2—H2B	109.4
C2—N3—H3A	117.5	C1—C2—H2B	109.4
C8—O1—C11	118.37 (16)	N3—C2—H2C	109.4
C10—C5—C6	118.11 (16)	C1—C2—H2C	109.4
C10—C5—C4	122.54 (15)	H2B—C2—H2C	108.0
C6—C5—C4	119.32 (15)	C6—C7—C8	120.37 (16)
C9—C10—C5	121.73 (16)	C6—C7—H7A	119.8
C9—C10—H10A	119.1	C8—C7—H7A	119.8
C5—C10—H10A	119.1	C2—C1—H1B	109.5
N2—C4—C5	122.20 (15)	C2—C1—H1C	109.5
N2—C4—H4A	118.9	H1B—C1—H1C	109.5
C5—C4—H4A	118.9	C2—C1—H1D	109.5
O1—C8—C7	115.83 (15)	H1B—C1—H1D	109.5
O1—C8—C9	124.41 (16)	H1C—C1—H1D	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S1^{i}	0.86	2.60	3.4080 (17)	156
N3—H3*A*···N2	0.86	2.26	2.634 (2)	106
N3—H3*A*···S1^{ii}	0.86	2.78	3.4670 (17)	137

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