Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(4-Methoxybenzylidene)-semicarbazide

Zu-Pei Liang,[a]* Jian Li,[a] Hong-Liang Wang[b] and Hui-Qin Wang[a]

[a]Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China, and [b]Weifang Examination Centre of Boilers and Pressure Vessels, Weifang 261057, People's Republic of China
Correspondence e-mail: zupeiliang@yahoo.com.cn

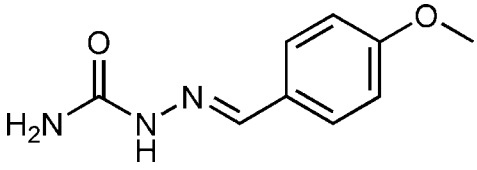
Received 11 May 2007; accepted 11 May 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.131; data-to-parameter ratio = 12.1.

In the crystal structure of the title compound, $C_9H_{11}N_3O_2$, the almost planar molecules interact by way of N—H···O hydrogen bonds.

Related literature

For a related structure, see Tai *et al.* (2007).



Experimental

Crystal data

$C_9H_{11}N_3O_2$
M_r = 193.21
Monoclinic, $P2_1/c$
a = 13.811 (11) Å
b = 5.443 (4) Å
c = 12.912 (10) Å
β = 97.933 (13)°
V = 961.4 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.979, T_{max} = 0.990
4626 measured reflections
1698 independent reflections
1080 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.131
S = 1.03
1698 reflections
140 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O2^{i}	0.899 (9)	2.037 (10)	2.929 (3)	171.4 (19)
N2—H2A···O2^{ii}	0.893 (9)	2.053 (10)	2.940 (3)	172.2 (19)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Doctoral Foundation of Weifang University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2409).

References

Bruker (1997). *SADABS* (Version 2.01), *SMART* (Version 5.044), *SAINT* (Version 5.01) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tai, X.-S., Hao, M.-Y., Yin, J. & Liang, Z.-P. (2007). *Acta Cryst.* E**63**, o1725–o1726.

supplementary materials

Acta Cryst. (2007). E**63**, o2939 [**doi:10.1107/S1600536807023240**]

(*E*)-1-(4-Methoxybenzylidene)semicarbazide

Z.-P. Liang, J. Li, H.-L. Wang and H.-Q. Wang

Comment

In this paper, the structure of the title compound, (I), is reported. The bond lengths and angles agree with those in a similar compound (*E*)-1-(4-Hydroxybenzylidene)semicarbazide hemihydrate (Tai *et al.*, 2007). The molecule is essentially planar, with a maximum deviation from the mean plane of 0.033 (4) Å for the non-hydrogen atoms. The crystal structure is stabilized by N—H···O hydrogen bonds (Fig. 2 and Table 2).

Experimental

A mixture of 4-methoxybenzaldehyde (0.01 mol) and semicarbazide hydrochloride (0.01 mol) in ethanol (10 ml) was refluxed for 1 h. After cooling, filtration and drying, the title compound was obtained. 10 mg of this compound was dissolved in ethanol (12 ml), and the solution was then allowed to evaporate at room temperature; light yellow single crystals of (I) were formed after 8 d.

Refinement

The N-bound H atoms were located in difference maps: their positions were refined with the restraint N—H = 0.89 (1) Å and their U_{iso} values were freely refined. The C-bound H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

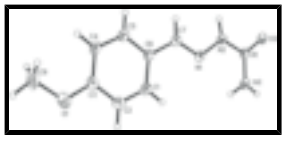


Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids (arbitrary spheres for the H atoms).

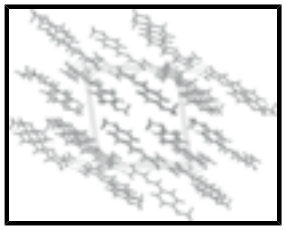


Fig. 2. The crystal packing of (I), viewed along the *b* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)-1-(4-Methoxybenzylidene)semicarbazide

Crystal data

$C_9H_{11}N_3O_2$ F_{000} = 408

M_r = 193.21	D_x = 1.335 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1284 reflections
a = 13.811 (11) Å	θ = 3.0–24.6º
b = 5.443 (4) Å	μ = 0.10 mm^{-1}
c = 12.912 (10) Å	T = 294 (2) K
β = 97.933 (13)º	Block, light yellow
V = 961.4 (13) Å^3	0.22 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	1698 independent reflections
Radiation source: fine-focus sealed tube	1080 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 294(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −16→15
T_{min} = 0.979, T_{max} = 0.990	k = −4→6
4626 measured reflections	l = −15→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.131	$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.0049P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.004
1698 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
140 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 calculat-

ing *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}
O1	0.41747 (12)	1.0943 (3)	0.72911 (13)	0.0620 (5)
O2	−0.00139 (11)	0.1030 (2)	1.13605 (10)	0.0443 (4)
N1	0.14797 (13)	0.4422 (3)	0.98921 (12)	0.0376 (5)
N2	0.08994 (13)	0.2555 (3)	1.01916 (13)	0.0408 (5)
N3	0.07790 (15)	0.4668 (3)	1.17129 (13)	0.0435 (5)
C1	0.26768 (16)	0.8148 (4)	0.90565 (16)	0.0426 (6)
H1	0.2498	0.8496	0.9709	0.051*
C2	0.32882 (17)	0.9721 (4)	0.86267 (17)	0.0463 (6)
H2	0.3522	1.1117	0.8994	0.056*
C3	0.35624 (16)	0.9253 (4)	0.76477 (17)	0.0420 (6)
C4	0.32198 (15)	0.7166 (4)	0.71071 (16)	0.0444 (6)
H4	0.3401	0.6826	0.6455	0.053*
C5	0.26042 (16)	0.5587 (4)	0.75450 (15)	0.0418 (6)
H5	0.2373	0.4189	0.7177	0.050*
C6	0.23206 (16)	0.6034 (3)	0.85243 (15)	0.0356 (5)
C7	0.16913 (15)	0.4259 (3)	0.89599 (15)	0.0377 (5)
H7	0.1434	0.2958	0.8542	0.045*
C8	0.05186 (15)	0.2709 (3)	1.11155 (14)	0.0345 (5)
C9	0.4390 (2)	1.0650 (6)	0.62403 (19)	0.0804 (10)
H9A	0.4745	0.9148	0.6189	0.121*
H9B	0.4779	1.2010	0.6064	0.121*
H9C	0.3790	1.0597	0.5766	0.121*
H3A	0.0485 (14)	0.501 (4)	1.2275 (12)	0.062 (7)*
H2A	0.0686 (14)	0.140 (3)	0.9726 (13)	0.047 (6)*
H3B	0.1138 (15)	0.586 (3)	1.1490 (16)	0.064 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0656 (13)	0.0628 (11)	0.0611 (11)	−0.0215 (8)	0.0210 (9)	0.0056 (8)
O2	0.0615 (11)	0.0435 (8)	0.0312 (8)	−0.0086 (7)	0.0183 (7)	0.0022 (6)
N1	0.0484 (12)	0.0398 (9)	0.0264 (9)	−0.0026 (8)	0.0117 (8)	0.0017 (7)
N2	0.0585 (13)	0.0401 (10)	0.0272 (9)	−0.0102 (9)	0.0179 (9)	−0.0034 (8)
N3	0.0603 (14)	0.0452 (11)	0.0274 (10)	−0.0055 (10)	0.0146 (9)	−0.0046 (8)
C1	0.0552 (15)	0.0431 (12)	0.0306 (11)	0.0027 (11)	0.0101 (11)	−0.0006 (9)
C2	0.0548 (16)	0.0401 (12)	0.0442 (13)	−0.0058 (11)	0.0080 (11)	−0.0054 (10)
C3	0.0430 (15)	0.0428 (12)	0.0417 (13)	−0.0028 (10)	0.0107 (11)	0.0085 (9)
C4	0.0513 (15)	0.0548 (14)	0.0295 (12)	−0.0043 (11)	0.0137 (10)	0.0023 (10)
C5	0.0497 (15)	0.0458 (12)	0.0311 (12)	−0.0046 (10)	0.0099 (10)	−0.0024 (9)
C6	0.0421 (14)	0.0383 (11)	0.0269 (11)	0.0020 (9)	0.0067 (10)	0.0038 (8)
C7	0.0451 (14)	0.0412 (11)	0.0275 (11)	−0.0006 (9)	0.0081 (10)	0.0010 (8)
C8	0.0443 (13)	0.0360 (11)	0.0239 (10)	0.0055 (9)	0.0072 (9)	0.0046 (8)

C9	0.086 (2)	0.104 (2)	0.0543 (18)	−0.0391 (18)	0.0217 (16)	0.0196 (15)

Geometric parameters (Å, °)

O1—C3	1.372 (2)	C2—C3	1.392 (3)
O1—C9	1.437 (3)	C2—H2	0.9300
O2—C8	1.241 (2)	C3—C4	1.382 (3)
N1—C7	1.280 (3)	C4—C5	1.383 (3)
N1—N2	1.382 (2)	C4—H4	0.9300
N2—C8	1.371 (3)	C5—C6	1.396 (3)
N2—H2A	0.893 (9)	C5—H5	0.9300
N3—C8	1.336 (3)	C6—C7	1.463 (3)
N3—H3A	0.899 (9)	C7—H7	0.9300
N3—H3B	0.886 (9)	C9—H9A	0.9600
C1—C2	1.372 (3)	C9—H9B	0.9600
C1—C6	1.395 (3)	C9—H9C	0.9600
C1—H1	0.9300		
C3—O1—C9	117.38 (18)	C5—C4—H4	120.3
C7—N1—N2	115.36 (17)	C4—C5—C6	121.8 (2)
C8—N2—N1	120.35 (16)	C4—C5—H5	119.1
C8—N2—H2A	119.9 (14)	C6—C5—H5	119.1
N1—N2—H2A	118.7 (13)	C1—C6—C5	117.78 (19)
C8—N3—H3A	121.1 (13)	C1—C6—C7	122.91 (19)
C8—N3—H3B	120.9 (13)	C5—C6—C7	119.29 (18)
H3A—N3—H3B	116.4 (14)	N1—C7—C6	122.71 (19)
C2—C1—C6	120.7 (2)	N1—C7—H7	118.6
C2—C1—H1	119.7	C6—C7—H7	118.6
C6—C1—H1	119.7	O2—C8—N3	124.21 (18)
C1—C2—C3	120.8 (2)	O2—C8—N2	119.26 (17)
C1—C2—H2	119.6	N3—C8—N2	116.50 (19)
C3—C2—H2	119.6	O1—C9—H9A	109.5
O1—C3—C4	124.45 (19)	O1—C9—H9B	109.5
O1—C3—C2	116.09 (19)	H9A—C9—H9B	109.5
C4—C3—C2	119.46 (19)	O1—C9—H9C	109.5
C3—C4—C5	119.4 (2)	H9A—C9—H9C	109.5
C3—C4—H4	120.3	H9B—C9—H9C	109.5
C7—N1—N2—C8	−171.16 (19)	C2—C1—C6—C5	−0.2 (3)
C6—C1—C2—C3	0.4 (3)	C2—C1—C6—C7	178.11 (19)
C9—O1—C3—C4	6.8 (3)	C4—C5—C6—C1	0.2 (3)
C9—O1—C3—C2	−173.7 (2)	C4—C5—C6—C7	−178.21 (19)
C1—C2—C3—O1	179.93 (19)	N2—N1—C7—C6	−178.17 (17)
C1—C2—C3—C4	−0.5 (3)	C1—C6—C7—N1	−5.3 (3)
O1—C3—C4—C5	180.0 (2)	C5—C6—C7—N1	173.0 (2)
C2—C3—C4—C5	0.5 (3)	N1—N2—C8—O2	178.81 (18)
C3—C4—C5—C6	−0.3 (3)	N1—N2—C8—N3	−3.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N3—H3A···O2^{i}	0.899 (9)	2.037 (10)	2.929 (3)	171.4 (19)
N2—H2A···O2^{ii}	0.893 (9)	2.053 (10)	2.940 (3)	172.2 (19)

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

Fig. 1

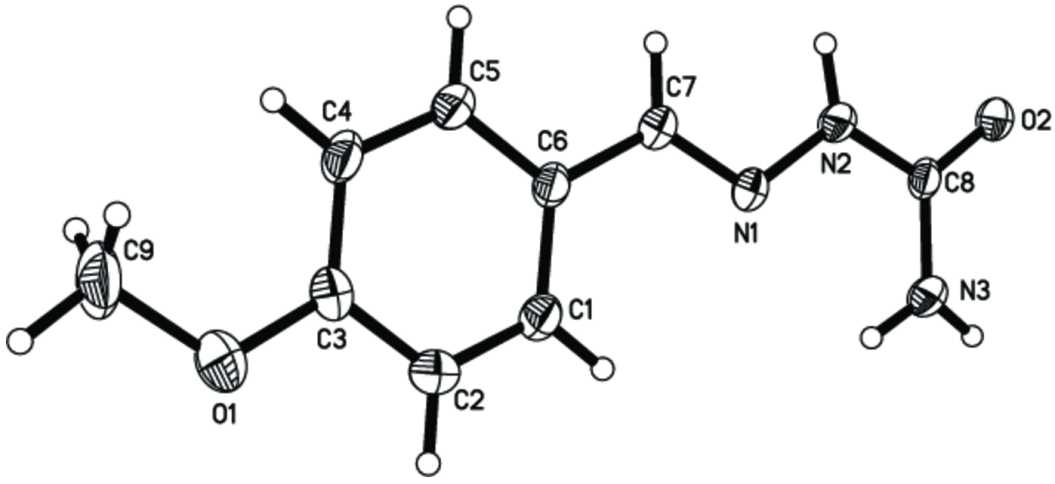


Fig. 2