Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-Methyl *N*′-(2-hydroxybenzylidene)-hydrazinecarboxylate at 123 K

Rong Sun and Xiang-Wei Cheng*

Zhejiang Police College Experience Center, Zhejiang Police College, Hangzhou 310053, People's Republic of China
Correspondence e-mail: zpccxw@126.com

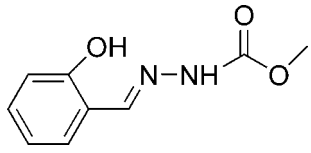
Received 9 July 2008; accepted 16 July 2008

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.120; data-to-parameter ratio = 13.1.

In the title molecule, $C_9H_{10}N_2O_3$, the hydrazinecarboxylic acid mean plane and the benzene ring form a dihedral angle of 11.1 (1)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into chains extending along the *b* axis. An intramolecular O—H···N hydrogen bond is also present.

Related literature

For applications of benzaldehydehydrazone derivatives, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987); Borg *et al.* (1999). For related structures, see: Cheng (2008).



Experimental

Crystal data

$C_9H_{10}N_2O_3$
M_r = 194.19
Orthorhombic, *Pbca*
a = 9.3998 (17) Å
b = 9.0945 (16) Å
c = 22.319 (4) Å
V = 1908.0 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 123 (2) K
0.27 × 0.24 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.965, T_{max} = 0.968
18306 measured reflections
1679 independent reflections
1427 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.120
S = 1.05
1679 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 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
O1—H1···N1	0.84	1.89	2.6234 (16)	145
N2—H2···O2^{i}	0.88	2.02	2.8881 (16)	169

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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*.

We acknowledge financial support from Zhejiang Police College, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2431).

References

Borg, S., Vollinga, R. C., Labarre, M., Payza, K., Terenius, L. & Luthman, K. (1999). *J. Med. Chem.* **42**, 4331–4342.
Bruker (2002). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, X.-W. (2008). *Acta Cryst.* E**64**, o1396.
Hadjoudis, E., Vittorakis, M. & Moustakali-Mavridis, J. (1987). *Tetrahedron*, **43**, 1345–1360.
Parashar, R. K., Sharma, R. C., Kumar, A. & Mohanm, G. (1988). *Inorg. Chim. Acta*, **151**, 201–208.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, o1563 [**doi:10.1107/S1600536808022332**]

(*E*)-Methyl *N*'-(2-hydroxybenzylidene)hydrazinecarboxylate at 123 K

R. Sun and X.-W. Cheng

Comment

Benzaldehydehydrazone derivatives have received considerable attentions for a long time due to their pharmacological activity (Parashar *et al.*, 1988) and their photochromic properties (Hadjoudis *et al.*, 1987). Meanwhile, it's an important intermidiate of 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many useful properties (Borg *et al.*, 1999). As a further investigation of this type of derivatives, the crystal structure of the title compound, $C_9H_{10}N_2O_3$, is described here.

The title molecule (Fig. 1) adopts a *trans* configuration with respect to the C=N bond. Intramolecular O—H···N hydrogen bond (Table 1) influences the molecular conformation. The hydrazine carboxylic acid methyl ester group is slightly twisted away from the attached ring. The dihedral angle between the C1-C6 ring and the C8/C9/N1/N2/O2/O3 plane is 11.1 (1)°. The bond lengths and angles agree with those observed for (*E*)-Methyl *N*'-(4-hydroxybenzylidene) hydrazinecarboxylate (Cheng, 2008).

In the crystal, intermolecular N–H···O (Table 1) hydrogen bonds link the molecules into chains extended along *b* axis.

Experimental

2-hydroxy benzaldehyde (1.22 g, 0.01 mol) and methyl hydrazinecarboxylate (0.90 g, 0.01 mol) were dissolved in stirred methanol (20 ml) and left for 3 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 90% yield. Crystals suitable for X-ray analysis were obtained by slow evaporation of a ethanol solution at room temperature (m.p. 465–468 K).

Refinement

All H atoms were positioned geometrically (N—H 0.88 Å, O—H 0.84 Å, C—H 0.95-0.98 Å) and refined in the riding model approximation,, with U_{iso}(H) = 1.2–1.5U_{eq} of the parent atom.

Figures

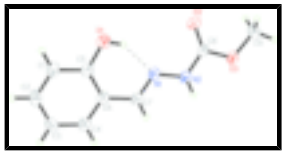


Fig. 1. Molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering. Dashed line denotes intramolecular hydrogen bond.

(*E*)-Methyl N'-(2-hydroxybenzylidene)hydrazinecarboxylate

Crystal data

$C_9H_{10}N_2O_3$	F_{000} = 816
M_r = 194.19	D_x = 1.352 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1679 reflections
a = 9.3998 (17) Å	θ = 2.0–25.0º
b = 9.0945 (16) Å	μ = 0.10 mm^{-1}
c = 22.319 (4) Å	*T* = 123 (2) K
V = 1908.0 (6) Å^3	Block, colourless
Z = 8	0.27 × 0.24 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1679 independent reflections
Radiation source: fine-focus sealed tube	1427 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 123(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	*h* = −10→11
T_{min} = 0.965, T_{max} = 0.968	*k* = −10→10
18306 measured reflections	*l* = −26→26

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.4536P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.120	$(\Delta/\sigma)_{max}$ = 0.028
S = 1.05	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
1679 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
128 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.018 (2)
Secondary atom site location: difference Fourier map	

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}
O3	0.37197 (12)	0.41375 (11)	−0.07538 (5)	0.0513 (3)
O2	0.36215 (12)	0.62287 (11)	−0.02044 (5)	0.0576 (4)
O1	0.16149 (12)	0.69567 (13)	0.13213 (5)	0.0625 (4)
H1	0.1906	0.6525	0.1012	0.094*
N2	0.22056 (13)	0.42471 (13)	−0.00032 (5)	0.0480 (4)
H2	0.1929	0.3365	−0.0116	0.058*
N1	0.16212 (12)	0.49157 (13)	0.04907 (5)	0.0452 (3)
C6	−0.00199 (15)	0.48908 (16)	0.13011 (6)	0.0429 (4)
C8	0.32185 (15)	0.49856 (15)	−0.03107 (6)	0.0431 (4)
C5	0.05028 (15)	0.61971 (16)	0.15591 (6)	0.0459 (4)
C7	0.06038 (16)	0.42561 (15)	0.07632 (6)	0.0461 (4)
H7	0.0255	0.3347	0.0613	0.055*
C1	−0.01226 (17)	0.67565 (18)	0.20764 (7)	0.0558 (4)
H1A	0.0236	0.7634	0.2251	0.067*
C4	−0.11726 (17)	0.41942 (18)	0.15784 (7)	0.0527 (4)
H4	−0.1537	0.3311	0.1411	0.063*
C2	−0.12633 (18)	0.6040 (2)	0.23379 (7)	0.0568 (5)
H2A	−0.1685	0.6431	0.2690	0.068*
C3	−0.17925 (17)	0.4760 (2)	0.20895 (7)	0.0573 (4)
H3	−0.2577	0.4272	0.2269	0.069*
C9	0.47848 (19)	0.4811 (2)	−0.11312 (7)	0.0617 (5)
H9A	0.5081	0.4111	−0.1441	0.093*
H9B	0.5610	0.5087	−0.0887	0.093*
H9C	0.4386	0.5690	−0.1321	0.093*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0596 (7)	0.0459 (6)	0.0484 (6)	0.0025 (5)	0.0093 (5)	−0.0070 (4)
O2	0.0665 (7)	0.0388 (6)	0.0674 (7)	−0.0022 (5)	0.0054 (5)	−0.0070 (5)
O1	0.0605 (7)	0.0593 (7)	0.0678 (7)	−0.0134 (5)	0.0108 (5)	−0.0181 (6)
N2	0.0567 (8)	0.0389 (7)	0.0484 (7)	−0.0020 (5)	0.0070 (6)	−0.0121 (5)

N1	0.0476 (7)	0.0430 (7)	0.0451 (7)	0.0051 (5)	0.0001 (5)	−0.0081 (5)
C6	0.0434 (8)	0.0435 (8)	0.0419 (8)	0.0047 (6)	−0.0069 (6)	0.0001 (6)
C8	0.0478 (8)	0.0366 (7)	0.0448 (8)	0.0075 (6)	−0.0035 (6)	−0.0040 (6)
C5	0.0457 (8)	0.0472 (8)	0.0449 (8)	0.0032 (6)	−0.0052 (6)	−0.0023 (6)
C7	0.0491 (8)	0.0406 (8)	0.0487 (8)	−0.0001 (6)	−0.0032 (6)	−0.0054 (6)
C1	0.0603 (10)	0.0578 (10)	0.0491 (8)	0.0048 (8)	−0.0057 (7)	−0.0124 (7)
C4	0.0526 (9)	0.0508 (9)	0.0548 (9)	−0.0005 (7)	−0.0012 (7)	−0.0002 (7)
C2	0.0586 (9)	0.0722 (11)	0.0395 (8)	0.0159 (8)	−0.0004 (7)	−0.0001 (7)
C3	0.0530 (9)	0.0651 (10)	0.0536 (9)	0.0043 (8)	0.0057 (7)	0.0105 (8)
C9	0.0589 (10)	0.0734 (11)	0.0528 (9)	0.0014 (8)	0.0107 (7)	0.0036 (8)

Geometric parameters (Å, °)

O3—C8	1.3397 (17)	C5—C1	1.392 (2)
O3—C9	1.4445 (19)	C7—H7	0.9500
O2—C8	1.2158 (18)	C1—C2	1.384 (2)
O1—C5	1.3607 (18)	C1—H1A	0.9500
O1—H1	0.8400	C4—C3	1.380 (2)
N2—C8	1.3522 (19)	C4—H4	0.9500
N2—N1	1.3736 (16)	C2—C3	1.383 (3)
N2—H2	0.8800	C2—H2A	0.9500
N1—C7	1.2823 (19)	C3—H3	0.9500
C6—C4	1.399 (2)	C9—H9A	0.9800
C6—C5	1.409 (2)	C9—H9B	0.9800
C6—C7	1.455 (2)	C9—H9C	0.9800
C8—O3—C9	115.49 (12)	C2—C1—C5	120.34 (15)
C5—O1—H1	109.5	C2—C1—H1A	119.8
C8—N2—N1	117.97 (12)	C5—C1—H1A	119.8
C8—N2—H2	121.0	C3—C4—C6	121.58 (15)
N1—N2—H2	121.0	C3—C4—H4	119.2
C7—N1—N2	118.15 (12)	C6—C4—H4	119.2
C4—C6—C5	118.10 (14)	C3—C2—C1	120.42 (15)
C4—C6—C7	119.81 (13)	C3—C2—H2A	119.8
C5—C6—C7	122.09 (14)	C1—C2—H2A	119.8
O2—C8—O3	124.74 (14)	C4—C3—C2	119.56 (16)
O2—C8—N2	125.61 (14)	C4—C3—H3	120.2
O3—C8—N2	109.65 (12)	C2—C3—H3	120.2
O1—C5—C1	117.55 (13)	O3—C9—H9A	109.5
O1—C5—C6	122.45 (13)	O3—C9—H9B	109.5
C1—C5—C6	120.00 (14)	H9A—C9—H9B	109.5
N1—C7—C6	120.40 (13)	O3—C9—H9C	109.5
N1—C7—H7	119.8	H9A—C9—H9C	109.5
C6—C7—H7	119.8	H9B—C9—H9C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.84	1.89	2.6234 (16)	145
N2—H2···O2^{i}	0.88	2.02	2.8881 (16)	169

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

Fig. 1