

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

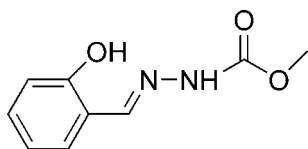
Received 9 July 2008; accepted 16 July 2008

 Key indicators: single-crystal X-ray study;  $T = 123$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.120; data-to-parameter ratio = 13.1.

In the title molecule,  $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_3$ , the hydrazinecarboxylic acid mean plane and the benzene ring form a dihedral angle of  $11.1(1)^\circ$ . In the crystal structure, intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules into chains extending along the  $b$  axis. An intramolecular  $\text{O}-\text{H}\cdots\text{N}$  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

 $\text{C}_9\text{H}_{10}\text{N}_2\text{O}_3$   
 $M_r = 194.19$   
 Orthorhombic,  $Pbca$ 
 $a = 9.3998(17)$  Å  
 $b = 9.0945(16)$  Å  
 $c = 22.319(4)$  Å

 $V = 1908.0(6)$  Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation

 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 123(2)$  K  
 $0.27 \times 0.24 \times 0.23$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.026$ 

### 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<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.19$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.84	1.89	2.6234 (16)	145
$\text{N2}-\text{H2}\cdots\text{O2}^{\dagger}$	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

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## supporting information

*Acta Cryst.* (2008). E64, o1563 [doi:10.1107/S1600536808022332]

**(E)-Methyl N'-(2-hydroxybenzylidene)hydrazinecarboxylate at 123 K****Rong Sun and Xiang-Wei Cheng****S1. Comment**

Benzaldehydhydrazone 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 intermediate 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<sub>9</sub>H<sub>10</sub>N<sub>2</sub>O<sub>3</sub>, 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).

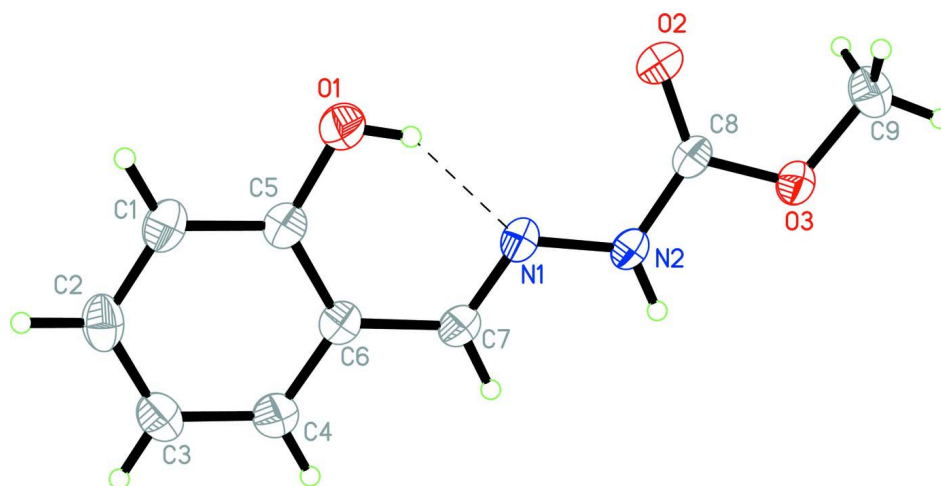
In the crystal, intermolecular N—H···O (Table 1) hydrogen bonds link the molecules into chains extended along *b* axis.

**S2. 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).

**S3. 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  $U_{\text{iso}}(\text{H}) = 1.2-1.5U_{\text{eq}}$  of the parent atom.

**Figure 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

$C_9H_{10}N_2O_3$

$M_r = 194.19$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 9.3998$  (17) Å

$b = 9.0945$  (16) Å

$c = 22.319$  (4) Å

$V = 1908.0$  (6) Å<sup>3</sup>

$Z = 8$

$F(000) = 816$

$D_x = 1.352$  Mg m<sup>-3</sup>

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

Cell parameters from 1679 reflections

$\theta = 2.0$ – $25.0^\circ$

$\mu = 0.10$  mm<sup>-1</sup>

$T = 123$  K

Block, colourless

$0.27 \times 0.24 \times 0.23$  mm

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2002)

$T_{\min} = 0.965$ ,  $T_{\max} = 0.968$

18306 measured reflections

1679 independent reflections

1427 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 1.8^\circ$

$h = -10 \rightarrow 11$

$k = -10 \rightarrow 10$

$l = -26 \rightarrow 26$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.120$

$S = 1.05$

1679 reflections

128 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

$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.4536P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.028$

$\Delta\rho_{\max} = 0.19$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.19$  e Å<sup>-3</sup>

Extinction correction: *SHELXL97* (Sheldrick, 2008),  $F_c^* = kFc[1 + 0.001xFe^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.018 (2)

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{iso}^*/U_{eq}$
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 ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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, °)*

<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>
O1—H1 $\cdots$ N1	0.84	1.89	2.6234 (16)	145

N2—H2 $\cdots$ O2 <sup>i</sup>	0.88	2.02	2.8881 (16)	169
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Symmetry code: (i)  $-x+1/2, y-1/2, z$ .