

## 4-(2-Hydroxybenzylidene)-3-methylisoxazol-5(4H)-one

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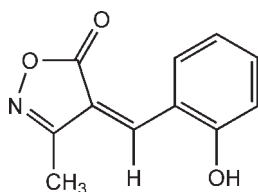
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 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.125; data-to-parameter ratio = 12.2.

The molecular skeleton of the title molecule,  $\text{C}_{11}\text{H}_9\text{NO}_3$ , is approximately planar (r.m.s. deviation = 0.0056 Å); the two rings form a dihedral angle of 6.5 (1)°. In the crystal structure, intermolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bonds involving the H atom of the hydroxy group and the N atom of the isoxazole ring link molecules into chains running along the  $c$  axis.

### Related literature

For the biological activity of arylmethylene isoxazolone derivatives, see: Ishioka *et al.* (2002); Liu *et al.* (2005). For related structures, see: Cocivera *et al.* (1976); Villemain *et al.* (1993); Zhang *et al.* (2008).



### Experimental

#### Crystal data

$\text{C}_{11}\text{H}_9\text{NO}_3$   
 $M_r = 203.19$   
 Monoclinic,  $P2_1/c$   
 $a = 8.0172$  (12) Å  
 $b = 6.8620$  (9) Å  
 $c = 17.535$  (2) Å  
 $\beta = 99.962$  (2)°

$V = 950.1$  (2) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.43 \times 0.30 \times 0.28$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.956$ ,  $T_{\max} = 0.971$   
 4598 measured reflections  
 1669 independent reflections  
 1067 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.125$   
 $S = 1.03$   
 1669 reflections  
 137 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.33$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.18$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H3}\cdots\text{N1}^i$	0.82	2.07	2.852 (2)	159

 Symmetry code: (i)  $x, -y + \frac{3}{2}, z + \frac{1}{2}$ 

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); 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) and DIAMOND (Brandenburg, 2004); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2639).

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**supplementary materials**

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## 4-(2-Hydroxybenzylidene)-3-methylisoxazol-5(4H)-one

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### Comment

Arylmethylene isoxazolone derivatives are effective anti-psychotics in the treatment of depression and schizophrenia. The study about arylmethylene isoxazolone derivatives mainly concentrates in the biological activities (Ishioka *et al.*, 2002; Liu *et al.*, 2005). However, structural studies of them have rarely been reported. As a part of our investigation on arylmethylene isoxazolone derivatives, we report here the structure of the title compound, (I), synthesized by three component condensation reaction of methyl acetoacetate, hydroxylamine with salicylaldehyde in aqueous media under ultrasonic irradiation.

In (I) (Fig. 1), all bond lengths and angles agree well with those reported for the related compounds (Cocivera *et al.*, 1976; Villemin *et al.*, 1993; Zhang *et al.*, 2008). The molecular structure adopts a *Z*-configuration about the C2=C5 double bond. The hydroxy groups and the isoxazol groups are involved in intermolecular O—H...N hydrogen bonds (Table 1), which link the molecules into chain structure, hydroxy O atom in the molecule acts as hydrogen-bond donor to isoxazol N atom in the neighbouring molecule, so forming a chain structure.

### Experimental

A mixture of methyl acetoacetate (4 mol), hydroxylamine hydrochloride (4 mmol), and pyridine(4 mmol) in distilled water(10 ml) was irradiated in the water bath of an ultrasonic cleaner for 10 min., then salicylaldehyde(4 mmol) was slowly added to the mixture. The resulting mixture was irradiated in the water bath of an ultrasonic cleaner for 1.5 h. The solution was left at room temperature overnight, the obtained mushy solution was filtered and the solid was washed with cold water and ethanol. The crude product was recrystallized from ethanol to afford the desired product as a yellow solid. Single crystal of (I) were obtained by slow evaporation of aqueous ethanol(95%) solution at ambient temperature after 7 d. Elemental analysis, calculated for C<sub>11</sub> H<sub>9</sub> N O<sub>3</sub>: C 65.02, H 4.46, N 6.89%; found: C 65.09, H 4.49, N 6.92%.

### Refinement

All hydrogen atoms were geometrically positioned (C—H = 0.93–0.96 Å, O—H = 0.82 Å) and allowed to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C}, \text{O})$ .

### Figures

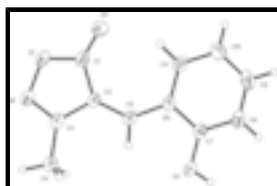


Fig. 1. The molecular structure of the title complex. Displacement ellipsoids are drawn at the 30% probability level.

## 4-(2-Hydroxybenzylidene)-3-methylisoxazol-5(4H)-one

### Crystal data

$C_{11}H_9NO_3$	$F_{000} = 424$
$M_r = 203.19$	$D_x = 1.420 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 1182 reflections
$a = 8.0172 (12) \text{ \AA}$	$\theta = 2.4\text{--}22.5^\circ$
$b = 6.8620 (9) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 17.535 (2) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 99.962 (2)^\circ$	Prism, yellow
$V = 950.1 (2) \text{ \AA}^3$	$0.43 \times 0.30 \times 0.28 \text{ mm}$
$Z = 4$	

### Data collection

Bruker SMART CCD area-detector diffractometer	1669 independent reflections
Radiation source: fine-focus sealed tube	1067 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.034$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.956$ , $T_{\text{max}} = 0.971$	$k = -8 \rightarrow 7$
4598 measured reflections	$l = -19 \rightarrow 20$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.2598P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
1669 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
137 parameters	$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
	Extinction correction: none

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.1424 (3)	0.7335 (3)	0.29966 (10)	0.0495 (6)
O1	0.2203 (2)	0.5472 (2)	0.29343 (8)	0.0573 (5)
O2	0.3282 (3)	0.3078 (3)	0.37132 (10)	0.0668 (6)
O3	0.1516 (2)	0.6467 (2)	0.64469 (8)	0.0579 (5)
H3	0.1416	0.6505	0.6904	0.087*
C1	0.2599 (3)	0.4642 (4)	0.36509 (12)	0.0474 (6)
C2	0.2013 (3)	0.5995 (3)	0.41967 (12)	0.0372 (6)
C3	0.1320 (3)	0.7584 (3)	0.37120 (12)	0.0404 (6)
C4	0.0543 (3)	0.9391 (3)	0.39499 (13)	0.0541 (7)
H4A	0.0185	1.0195	0.3504	0.081*
H4B	0.1358	1.0085	0.4316	0.081*
H4C	-0.0418	0.9065	0.4184	0.081*
C5	0.2050 (3)	0.5923 (3)	0.49738 (12)	0.0388 (6)
H5	0.1541	0.6992	0.5165	0.047*
C6	0.2713 (3)	0.4536 (3)	0.55603 (11)	0.0377 (5)
C7	0.2432 (3)	0.4893 (3)	0.63213 (12)	0.0406 (6)
C8	0.3061 (3)	0.3630 (4)	0.69154 (13)	0.0483 (6)
H8	0.2866	0.3871	0.7414	0.058*
C9	0.3967 (3)	0.2032 (4)	0.67709 (14)	0.0567 (7)
H9	0.4394	0.1194	0.7175	0.068*
C10	0.4262 (3)	0.1639 (4)	0.60287 (15)	0.0572 (7)
H10	0.4876	0.0543	0.5934	0.069*
C11	0.3640 (3)	0.2882 (3)	0.54405 (13)	0.0481 (6)
H11	0.3841	0.2615	0.4944	0.058*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0615 (14)	0.0559 (13)	0.0319 (11)	0.0024 (11)	0.0107 (9)	0.0008 (9)
O1	0.0808 (13)	0.0632 (11)	0.0295 (9)	0.0095 (10)	0.0140 (8)	-0.0030 (8)
O2	0.0956 (15)	0.0587 (12)	0.0470 (11)	0.0183 (11)	0.0150 (10)	-0.0060 (9)
O3	0.0894 (14)	0.0585 (11)	0.0271 (8)	0.0131 (10)	0.0142 (8)	-0.0018 (7)
C1	0.0577 (17)	0.0501 (15)	0.0351 (13)	-0.0025 (13)	0.0100 (10)	-0.0013 (11)
C2	0.0395 (14)	0.0437 (13)	0.0291 (11)	-0.0061 (10)	0.0074 (9)	-0.0026 (10)
C3	0.0402 (14)	0.0482 (14)	0.0331 (12)	-0.0072 (11)	0.0072 (10)	-0.0003 (10)
C4	0.0650 (18)	0.0524 (15)	0.0454 (14)	0.0074 (14)	0.0109 (12)	0.0061 (12)
C5	0.0410 (14)	0.0423 (13)	0.0343 (12)	-0.0040 (11)	0.0101 (10)	-0.0030 (10)

## supplementary materials

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C6	0.0385 (13)	0.0441 (13)	0.0304 (11)	-0.0061 (11)	0.0056 (9)	0.0018 (10)
C7	0.0434 (14)	0.0433 (13)	0.0349 (12)	-0.0046 (11)	0.0060 (9)	-0.0011 (10)
C8	0.0493 (16)	0.0619 (16)	0.0338 (13)	-0.0047 (13)	0.0078 (11)	0.0089 (12)
C9	0.0494 (17)	0.0666 (18)	0.0532 (16)	0.0012 (14)	0.0067 (12)	0.0243 (13)
C10	0.0520 (17)	0.0590 (16)	0.0622 (17)	0.0088 (14)	0.0144 (13)	0.0145 (14)
C11	0.0470 (15)	0.0562 (15)	0.0438 (14)	0.0009 (13)	0.0151 (11)	-0.0003 (12)

### *Geometric parameters (Å, °)*

N1—C3	1.283 (3)	C5—C6	1.434 (3)
N1—O1	1.435 (2)	C5—H5	0.9300
O1—C1	1.366 (3)	C6—C11	1.392 (3)
O2—C1	1.201 (3)	C6—C7	1.413 (3)
O3—C7	1.346 (3)	C7—C8	1.382 (3)
O3—H3	0.8200	C8—C9	1.363 (3)
C1—C2	1.467 (3)	C8—H8	0.9300
C2—C5	1.359 (3)	C9—C10	1.389 (4)
C2—C3	1.434 (3)	C9—H9	0.9300
C3—C4	1.479 (3)	C10—C11	1.364 (3)
C4—H4A	0.9600	C10—H10	0.9300
C4—H4B	0.9600	C11—H11	0.9300
C4—H4C	0.9600		
C3—N1—O1	107.22 (17)	C6—C5—H5	113.4
C1—O1—N1	109.63 (16)	C11—C6—C7	117.43 (19)
C7—O3—H3	109.5	C11—C6—C5	125.0 (2)
O2—C1—O1	119.1 (2)	C7—C6—C5	117.5 (2)
O2—C1—C2	134.3 (2)	O3—C7—C8	121.2 (2)
O1—C1—C2	106.7 (2)	O3—C7—C6	118.35 (19)
C5—C2—C3	124.1 (2)	C8—C7—C6	120.4 (2)
C5—C2—C1	132.6 (2)	C9—C8—C7	120.1 (2)
C3—C2—C1	103.26 (18)	C9—C8—H8	120.0
N1—C3—C2	113.2 (2)	C7—C8—H8	120.0
N1—C3—C4	119.3 (2)	C8—C9—C10	120.9 (2)
C2—C3—C4	127.5 (2)	C8—C9—H9	119.6
C3—C4—H4A	109.5	C10—C9—H9	119.6
C3—C4—H4B	109.5	C11—C10—C9	119.2 (3)
H4A—C4—H4B	109.5	C11—C10—H10	120.4
C3—C4—H4C	109.5	C9—C10—H10	120.4
H4A—C4—H4C	109.5	C10—C11—C6	122.0 (2)
H4B—C4—H4C	109.5	C10—C11—H11	119.0
C2—C5—C6	133.2 (2)	C6—C11—H11	119.0
C2—C5—H5	113.4		
C3—N1—O1—C1	-1.5 (3)	C1—C2—C5—C6	2.1 (4)
N1—O1—C1—O2	-179.2 (2)	C2—C5—C6—C11	4.8 (4)
N1—O1—C1—C2	1.5 (2)	C2—C5—C6—C7	-176.4 (2)
O2—C1—C2—C5	0.0 (5)	C11—C6—C7—O3	-178.9 (2)
O1—C1—C2—C5	179.1 (2)	C5—C6—C7—O3	2.2 (3)
O2—C1—C2—C3	179.9 (3)	C11—C6—C7—C8	-0.1 (3)
O1—C1—C2—C3	-1.0 (2)	C5—C6—C7—C8	-179.0 (2)

O1—N1—C3—C2	0.9 (3)	O3—C7—C8—C9	179.2 (2)
O1—N1—C3—C4	-179.32 (19)	C6—C7—C8—C9	0.3 (4)
C5—C2—C3—N1	180.0 (2)	C7—C8—C9—C10	-0.5 (4)
C1—C2—C3—N1	0.0 (3)	C8—C9—C10—C11	0.3 (4)
C5—C2—C3—C4	0.2 (4)	C9—C10—C11—C6	-0.1 (4)
C1—C2—C3—C4	-179.7 (2)	C7—C6—C11—C10	-0.1 (3)
C3—C2—C5—C6	-177.8 (2)	C5—C6—C11—C10	178.7 (2)

*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>
O3—H3 $\cdots$ N1 <sup>i</sup>	0.82	2.07	2.852 (2)	159

Symmetry codes: (i)  $x, -y+3/2, z+1/2$ .

Fig. 1

