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(E)-Methyl N'-(2-hydroxy-3-methoxybenzylidene)hydrazinecarboxylate

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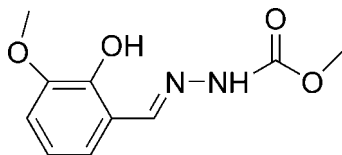
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Key indicators: single-crystal X-ray study; $T = 223$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.029; wR factor = 0.075; data-to-parameter ratio = 7.1.

The title compound, $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_4$, adopts a *trans* configuration with respect to the $\text{C}=\text{N}$ double bond. The non-H atoms of the molecule are essentially coplanar, with a maximum deviation of 0.015 (2) Å. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ interaction is observed. In the crystal structure, the molecules are linked into a two-dimensional network parallel to the *ac* plane by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds involving the methoxy O atom and by two $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds involving the carbonyl O atom. In addition, an intermolecular $\text{C}-\text{H}\cdots\pi$ interaction is observed.

Related literature

For general background to the properties of benzaldehyde-hydrazone derivatives, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987); Borg *et al.* (1999). For the use of metal complexes of Schiff bases as model compounds of active centres in various proteins and enzymes, see: Kahwa *et al.* (1986); Santos *et al.* (2001). For a related structure, see: Shang *et al.* (2007).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_4$
 $M_r = 224.22$
 Monoclinic, Cc

$a = 11.4348$ (13) Å
 $b = 14.8717$ (18) Å
 $c = 6.3508$ (8) Å

$\beta = 98.538$ (4)°
 $V = 1068.0$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.11$ mm⁻¹
 $T = 223$ K
 $0.24 \times 0.22 \times 0.17$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.975$, $T_{\max} = 0.985$

5851 measured reflections
 1049 independent reflections
 948 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.075$
 $S = 1.11$
 1049 reflections
 148 parameters

2 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.11$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.93	2.645 (2)	145
$\text{N2}-\text{H2}\cdots\text{O2}^{\text{i}}$	0.86	2.42	3.022 (2)	127
$\text{C5}-\text{H5}\cdots\text{O3}^{\text{ii}}$	0.93	2.49	3.320 (2)	149
$\text{C7}-\text{H7}\cdots\text{O3}^{\text{iii}}$	0.93	2.45	3.291 (2)	150
$\text{C3}-\text{H3}\cdots\text{Cg1}^{\text{iii}}$	0.93	2.85	3.606 (2)	139

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x, -y + 1, z - \frac{1}{2}$. Cg1 is the centroid of the C1–C6 ring.

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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2816).

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supplementary materials

Acta Cryst. (2009). E65, o1548 [doi:10.1107/S1600536809021631]

(*E*)-Methyl *N'*-(2-hydroxy-3-methoxybenzylidene)hydrazinecarboxylate

L.-P. Lv, W.-B. Yu, W.-W. Li, Y.-Z. Zhang and X.-C. Hu

Comment

Benzaldehydehydrazone derivatives have attracted much attention due to their pharmacological activity (Parashar *et al.*, 1988) and their photochromic properties (Hadjoudis *et al.*, 1987). They are important intermediates of 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many interesting properties (Borg *et al.*, 1999). Metal complexes based on Schiff bases have received considerable attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). We report here the crystal structure of the title compound.

The title molecule adopts a *trans* configuration with respect to the C=N bond. The non-hydrogen atoms of the molecule are essentially coplanar, with a maximum deviation of 0.015 (2) Å for atom C(7). The bond lengths and angles are comparable to those observed for methyl*N'*-[(*E*)-4-methoxybenzylidene]hydrazinecarboxylate (Shang *et al.*, 2007). An intramolecular O—H⋯N interaction is observed.

In the crystal structure, the molecules are linked into a two-dimensional network parallel to the *ac* plane by N—H⋯O and C—H⋯O hydrogen bonds (Table 1 and Fig.2). In addition, C—H⋯ π interactions are observed.

Experimental

2-Hydroxy-3-methoxybenzaldehyde (1.52 g, 0.01 mol) and methyl hydrazinecarboxylate (0.90g, 0.01 mol) were dissolved in stirred methanol (20 ml) and left for 3.5 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 90% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 415–418 K).

Refinement

H atoms were positioned geometrically (O-H = 0.82 Å, N-H = 0.86 Å and C-H = 0.93 or 0.96 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ and $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Figures

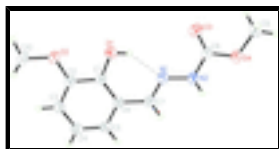


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

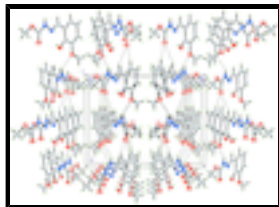


Fig. 2. Crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

(E)-Methyl N¹-(2-hydroxy-3-methoxybenzylidene)hydrazinecarboxylate

Crystal data

C₁₀H₁₂N₂O₄

M_r = 224.22

Monoclinic, *Cc*

Hall symbol: C -2yc

a = 11.4348 (13) Å

b = 14.8717 (18) Å

c = 6.3508 (8) Å

β = 98.538 (4)°

V = 1068.0 (2) Å³

Z = 4

*F*₀₀₀ = 472

D_x = 1.394 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 1049 reflections

θ = 2.3–26.0°

μ = 0.11 mm⁻¹

T = 223 K

Block, colourless

0.24 × 0.22 × 0.17 mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 223 K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2002)

T_{min} = 0.975, *T_{max}* = 0.985

5851 measured reflections

1049 independent reflections

948 reflections with *I* > 2σ(*I*)

R_{int} = 0.021

θ_{max} = 26.0°

θ_{min} = 2.3°

h = -14→14

k = -16→18

l = -7→7

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.029

wR (*F*²) = 0.075

S = 1.11

1049 reflections

148 parameters

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0409*P*)² + 0.1415*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.003

Δρ_{max} = 0.11 e Å⁻³

Δρ_{min} = -0.13 e Å⁻³

2 restraints

Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.11401 (17)	0.33419 (13)	0.6468 (3)	0.0412 (5)
C2	0.14440 (19)	0.38079 (15)	0.4705 (4)	0.0446 (5)
C3	0.0578 (2)	0.42357 (16)	0.3309 (4)	0.0513 (6)
H3	0.0779	0.4550	0.2149	0.062*
C4	-0.0596 (2)	0.4195 (2)	0.3646 (4)	0.0584 (6)
H4	-0.1176	0.4484	0.2704	0.070*
C5	-0.0906 (2)	0.37377 (16)	0.5339 (4)	0.0528 (6)
H5	-0.1696	0.3711	0.5531	0.063*
C6	-0.00420 (19)	0.33056 (15)	0.6795 (3)	0.0436 (5)
C7	-0.04116 (18)	0.28326 (16)	0.8601 (3)	0.0469 (5)
H7	-0.1203	0.2836	0.8788	0.056*
C8	0.07691 (19)	0.15569 (15)	1.3010 (3)	0.0451 (5)
C9	0.1022 (3)	0.0689 (2)	1.6128 (5)	0.0690 (8)
H9A	0.1535	0.0304	1.5467	0.103*
H9B	0.0569	0.0334	1.6977	0.103*
H9C	0.1487	0.1118	1.7019	0.103*
C10	0.2979 (3)	0.4277 (2)	0.2805 (5)	0.0767 (9)
H10A	0.2597	0.4021	0.1495	0.115*
H10B	0.3821	0.4231	0.2868	0.115*
H10C	0.2760	0.4898	0.2874	0.115*
N1	0.03469 (15)	0.24138 (13)	0.9926 (3)	0.0462 (4)
N2	-0.00459 (16)	0.19813 (14)	1.1600 (3)	0.0502 (5)
H2	-0.0782	0.1981	1.1743	0.060*
O1	0.20402 (14)	0.29467 (12)	0.7790 (3)	0.0553 (4)
H1	0.1776	0.2697	0.8770	0.083*
O2	0.26239 (14)	0.38043 (11)	0.4552 (3)	0.0561 (4)
O3	0.18042 (15)	0.15245 (13)	1.2939 (3)	0.0660 (5)
O4	0.02356 (14)	0.11544 (12)	1.4512 (3)	0.0572 (5)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0391 (10)	0.0391 (12)	0.0450 (11)	0.0002 (8)	0.0049 (9)	-0.0029 (10)
C2	0.0418 (10)	0.0435 (12)	0.0494 (12)	-0.0029 (8)	0.0099 (9)	-0.0048 (9)
C3	0.0525 (13)	0.0533 (14)	0.0481 (14)	-0.0039 (10)	0.0069 (10)	0.0079 (11)
C4	0.0442 (11)	0.0696 (17)	0.0587 (15)	0.0017 (10)	-0.0016 (10)	0.0162 (12)
C5	0.0377 (10)	0.0573 (15)	0.0632 (15)	0.0004 (10)	0.0066 (10)	0.0057 (11)
C6	0.0419 (10)	0.0420 (12)	0.0477 (12)	-0.0026 (9)	0.0087 (8)	-0.0025 (9)
C7	0.0417 (11)	0.0477 (14)	0.0526 (13)	-0.0010 (9)	0.0114 (10)	-0.0009 (10)
C8	0.0430 (12)	0.0476 (13)	0.0466 (12)	-0.0036 (9)	0.0130 (9)	-0.0022 (10)
C9	0.0701 (16)	0.0767 (18)	0.0617 (17)	0.0157 (14)	0.0148 (13)	0.0164 (14)
C10	0.0583 (15)	0.098 (2)	0.081 (2)	-0.0028 (14)	0.0332 (14)	0.0230 (16)
N1	0.0457 (9)	0.0486 (11)	0.0459 (9)	-0.0044 (8)	0.0120 (8)	0.0006 (9)
N2	0.0388 (9)	0.0593 (12)	0.0544 (12)	-0.0024 (8)	0.0135 (8)	0.0107 (9)
O1	0.0429 (8)	0.0631 (10)	0.0591 (10)	0.0033 (8)	0.0050 (7)	0.0137 (8)
O2	0.0433 (8)	0.0628 (9)	0.0653 (10)	0.0004 (7)	0.0185 (7)	0.0103 (8)
O3	0.0413 (9)	0.0889 (13)	0.0689 (11)	0.0005 (8)	0.0122 (7)	0.0093 (10)
O4	0.0499 (8)	0.0641 (11)	0.0596 (10)	0.0070 (7)	0.0151 (7)	0.0183 (8)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.361 (2)	C8—O3	1.192 (3)
C1—C6	1.399 (3)	C8—O4	1.347 (3)
C1—C2	1.404 (3)	C8—N2	1.349 (3)
C2—O2	1.367 (3)	C9—O4	1.438 (3)
C2—C3	1.382 (3)	C9—H9A	0.96
C3—C4	1.392 (3)	C9—H9B	0.96
C3—H3	0.93	C9—H9C	0.96
C4—C5	1.363 (4)	C10—O2	1.423 (3)
C4—H4	0.93	C10—H10A	0.96
C5—C6	1.405 (3)	C10—H10B	0.96
C5—H5	0.93	C10—H10C	0.96
C6—C7	1.460 (3)	N1—N2	1.374 (2)
C7—N1	1.277 (3)	N2—H2	0.86
C7—H7	0.93	O1—H1	0.82
O1—C1—C6	123.41 (19)	O3—C8—N2	125.9 (2)
O1—C1—C2	116.80 (17)	O4—C8—N2	109.70 (18)
C6—C1—C2	119.79 (19)	O4—C9—H9A	109.5
O2—C2—C3	125.2 (2)	O4—C9—H9B	109.5
O2—C2—C1	114.75 (19)	H9A—C9—H9B	109.5
C3—C2—C1	120.00 (19)	O4—C9—H9C	109.5
C2—C3—C4	119.8 (2)	H9A—C9—H9C	109.5
C2—C3—H3	120.1	H9B—C9—H9C	109.5
C4—C3—H3	120.1	O2—C10—H10A	109.5
C5—C4—C3	120.9 (2)	O2—C10—H10B	109.5
C5—C4—H4	119.6	H10A—C10—H10B	109.5

C3—C4—H4	119.6	O2—C10—H10C	109.5
C4—C5—C6	120.5 (2)	H10A—C10—H10C	109.5
C4—C5—H5	119.8	H10B—C10—H10C	109.5
C6—C5—H5	119.8	C7—N1—N2	118.03 (16)
C1—C6—C5	119.0 (2)	C8—N2—N1	117.36 (17)
C1—C6—C7	122.28 (19)	C8—N2—H2	121.3
C5—C6—C7	118.7 (2)	N1—N2—H2	121.3
N1—C7—C6	120.31 (18)	C1—O1—H1	109.5
N1—C7—H7	119.8	C2—O2—C10	116.9 (2)
C6—C7—H7	119.8	C8—O4—C9	114.74 (19)
O3—C8—O4	124.4 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

<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	0.82	1.93	2.645 (2)	145
N2—H2 \cdots O2 ⁱ	0.86	2.42	3.022 (2)	127
C5—H5 \cdots O3 ⁱⁱ	0.93	2.49	3.320 (2)	149
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C3—H3 \cdots Cg1 ⁱⁱⁱ	0.93	2.85	3.606 (2)	139

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

Fig. 1

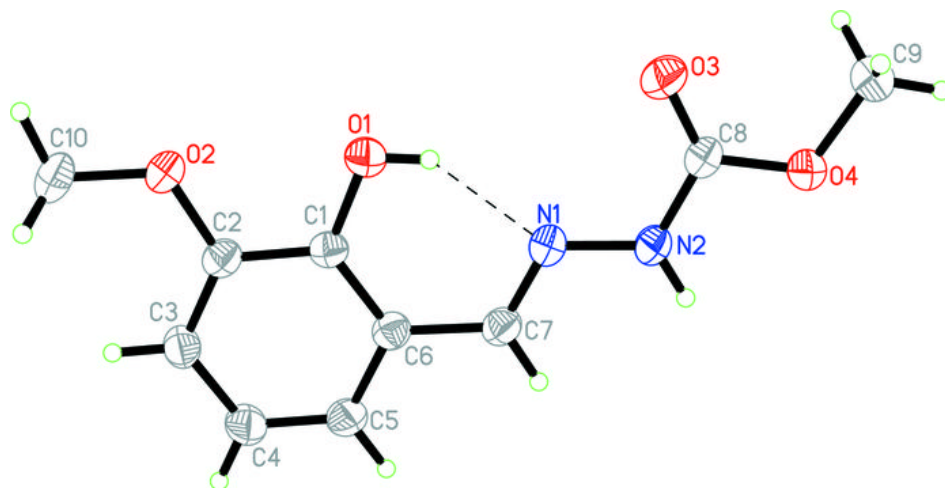


Fig. 2

