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Methyl 2-[(*E*)-3-hydroxy-4-methoxybenzylidene]hydrazinecarboxylate

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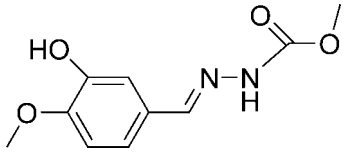
Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.098; data-to-parameter ratio = 13.0.

The title compound, $C_{10}H_{12}N_2O_4$, adopts a *trans* configuration with respect to the C=N bond. The hydrazinecarboxylate group is twisted from the benzene ring by 6.62 (5)° and an intramolecular O—H···O hydrogen bond occurs. In the crystal structure, molecules are linked into a two-dimensional network parallel to (100) by O—H···O, N—H···O and C—H···O hydrogen bonds. In addition, weak C—H···π interactions are observed.

Related literature

For properties of benzaldehydehydrazone derivatives, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987); Borg *et al.* (1999). For Schiff base metal complexes, see: Kahwa *et al.* (1986); Santos *et al.* (2001). For a related structure, see: Shang *et al.* (2007).



Experimental

Crystal data

$C_{10}H_{12}N_2O_4$
M_r = 224.22
Monoclinic, $P2_1/c$
a = 7.7223 (12) Å
b = 9.2106 (14) Å
c = 15.092 (2) Å
β = 100.944 (6)°
V = 1054.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 223 K
0.18 × 0.16 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.978, T_{max} = 0.982
5767 measured reflections
1944 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.05
1944 reflections
149 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.82	2.28	2.6871 (13)	112
O2—H2···O3^{i}	0.82	2.20	2.9303 (13)	148
N2—H2A···O3^{ii}	0.86	2.44	3.1951 (15)	147
C8—H8···O3^{ii}	0.93	2.51	3.3185 (16)	146
C10—H10A···*Cg*1^{iii}	0.96	2.87	3.6878 (18)	143

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x+1, -y-\frac{1}{2}, z-\frac{1}{2}$. *Cg*1 is the centroid of the C2–C7 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: CI2804).

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Methyl 2-[(*E*)-3-hydroxy-4-methoxybenzylidene]hydrazinecarboxylate

Lu-Ping Lv, Tie-Ming Yu, Wen-Bo Yu, Wei-Wei Li and Xian-Chao Hu

S1. 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 (Fig. 1).

The title molecule adopts a *trans* configuration with respect to the C═N bond. The hydrazinecarboxylate group is twisted from the benzene ring by 6.62 (5)°. 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···O interaction is observed.

In the crystal structure, the molecules are linked into a two-dimensional network parallel to the (100) by O—H···O, N—H···O and C—H···O hydrogen bonds (Table 1 and Fig.2). In addition, a C—H···π interaction is observed.

S2. Experimental

3-Hydroxy-4-methoxy-benzaldehyde (1.52 g, 0.01 mol) and methyl hydrazinecarboxylate (0.90 g, 0.01 mol) were dissolved in stirred methanol (25 ml) and left for 4 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 75% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 398–401 K).

S3. Refinement

H atoms were positioned geometrically (N-H = 0.86 Å and C-H = 0.93 or 0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{eq}(C_{methyl}).

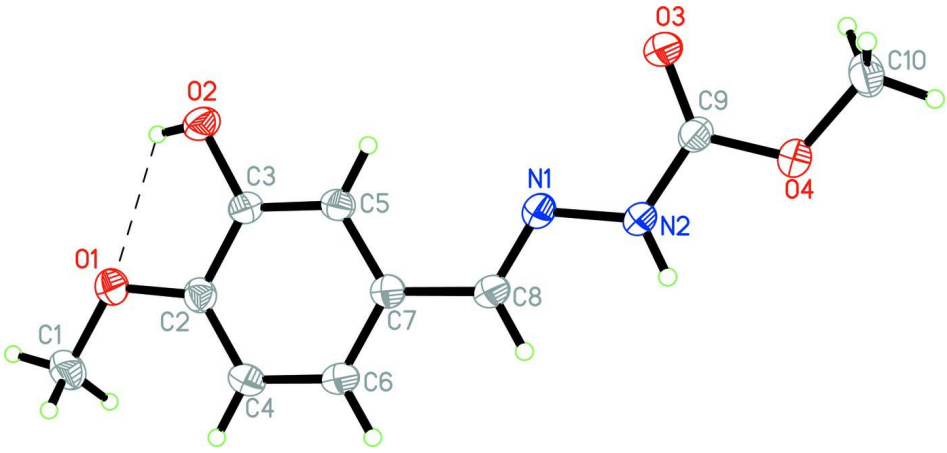

Figure 1

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

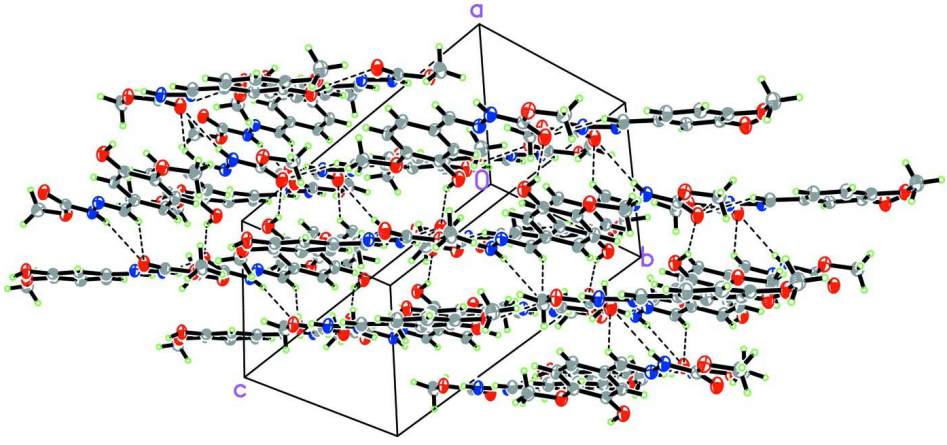

Figure 2

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

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Crystal data

$C_{10}H_{12}N_2O_4$
$M_r = 224.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.7223\ (12)$ Å
$b = 9.2106\ (14)$ Å
$c = 15.092\ (2)$ Å
$\beta = 100.944\ (6)°$
$V = 1054.0\ (3)$ Å^3
$Z = 4$

$F(000) = 472$
$D_x = 1.413$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1944 reflections
$\theta = 2.6–25.5°$
$\mu = 0.11$ mm^{-1}
$T = 223$ K
Block, colourless
0.18 × 0.16 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.978, T_{max} = 0.982
5767 measured reflections
1944 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.5°, θ_{min} = 2.6°
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.098
S = 1.05
1944 reflections
149 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
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.1597P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.035 (4)

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}
C9	1.14900 (17)	0.19752 (13)	0.79259 (8)	0.0395 (3)
C8	0.89788 (18)	0.11117 (13)	0.58309 (9)	0.0435 (3)
H8	0.8814	0.0133	0.5941	0.052*
C3	0.78128 (16)	0.37223 (13)	0.39017 (8)	0.0386 (3)
C5	0.84592 (17)	0.31881 (13)	0.47483 (8)	0.0392 (3)
H5	0.9053	0.3802	0.5193	0.047*
C7	0.82305 (17)	0.17208 (13)	0.49464 (8)	0.0399 (3)
C2	0.68958 (16)	0.28075 (14)	0.32243 (8)	0.0401 (3)
C6	0.73162 (19)	0.08300 (14)	0.42746 (9)	0.0475 (3)
H6	0.7149	−0.0143	0.4401	0.057*
C4	0.66479 (18)	0.13636 (14)	0.34190 (9)	0.0466 (3)
H4	0.6035	0.0753	0.2978	0.056*
C10	1.3093 (2)	0.17575 (17)	0.94154 (9)	0.0564 (4)
H10A	1.3936	0.2432	0.9261	0.085*
H10B	1.3697	0.1025	0.9810	0.085*

H10C	1.2281	0.2263	0.9714	0.085*
C1	0.5523 (2)	0.25850 (18)	0.16765 (10)	0.0575 (4)
H1A	0.6295	0.1791	0.1614	0.086*
H1B	0.5317	0.3150	0.1132	0.086*
H1C	0.4422	0.2214	0.1787	0.086*
O1	0.63158 (13)	0.34761 (10)	0.24138 (6)	0.0500 (3)
O4	1.21426 (13)	0.10865 (10)	0.86081 (6)	0.0513 (3)
O3	1.17310 (14)	0.32803 (9)	0.79258 (6)	0.0529 (3)
O2	0.80571 (14)	0.51620 (9)	0.37432 (6)	0.0520 (3)
H2	0.7894	0.5307	0.3197	0.078*
N1	0.98497 (14)	0.18969 (11)	0.64511 (7)	0.0420 (3)
N2	1.05406 (15)	0.12010 (11)	0.72457 (7)	0.0442 (3)
H2A	1.0369	0.0287	0.7307	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C9	0.0475 (7)	0.0337 (6)	0.0380 (7)	0.0026 (5)	0.0096 (5)	0.0032 (5)
C8	0.0531 (8)	0.0325 (6)	0.0440 (7)	−0.0037 (5)	0.0067 (6)	0.0023 (5)
C3	0.0427 (7)	0.0331 (6)	0.0406 (7)	0.0000 (5)	0.0091 (5)	−0.0006 (5)
C5	0.0441 (7)	0.0354 (6)	0.0372 (6)	−0.0039 (5)	0.0057 (5)	−0.0032 (5)
C7	0.0425 (7)	0.0369 (6)	0.0397 (7)	−0.0015 (5)	0.0067 (5)	0.0002 (5)
C2	0.0412 (7)	0.0415 (7)	0.0369 (7)	0.0025 (5)	0.0060 (5)	−0.0007 (5)
C6	0.0571 (8)	0.0335 (6)	0.0497 (8)	−0.0070 (6)	0.0045 (6)	−0.0007 (5)
C4	0.0518 (8)	0.0417 (7)	0.0430 (7)	−0.0061 (6)	0.0003 (6)	−0.0079 (5)
C10	0.0626 (9)	0.0631 (9)	0.0391 (8)	−0.0074 (7)	−0.0015 (6)	0.0067 (6)
C1	0.0618 (9)	0.0646 (9)	0.0411 (8)	−0.0054 (7)	−0.0030 (6)	−0.0032 (7)
O1	0.0606 (6)	0.0474 (5)	0.0377 (5)	0.0009 (4)	−0.0018 (4)	0.0008 (4)
O4	0.0664 (6)	0.0407 (5)	0.0416 (5)	−0.0009 (4)	−0.0027 (4)	0.0066 (4)
O3	0.0776 (7)	0.0341 (5)	0.0440 (5)	−0.0049 (4)	0.0038 (5)	0.0007 (4)
O2	0.0772 (7)	0.0348 (5)	0.0415 (5)	−0.0048 (4)	0.0053 (5)	0.0044 (4)
N1	0.0517 (6)	0.0348 (5)	0.0380 (6)	0.0009 (4)	0.0049 (5)	0.0046 (4)
N2	0.0602 (7)	0.0300 (5)	0.0393 (6)	−0.0015 (5)	0.0015 (5)	0.0040 (4)

Geometric parameters (Å, º)

C9—O3	1.2164 (15)	C6—C4	1.3864 (19)
C9—O4	1.3369 (15)	C6—H6	0.93
C9—N2	1.3469 (16)	C4—H4	0.93
C8—N1	1.2698 (16)	C10—O4	1.4371 (16)
C8—C7	1.4621 (17)	C10—H10A	0.96
C8—H8	0.93	C10—H10B	0.96
C3—O2	1.3669 (14)	C10—H10C	0.96
C3—C5	1.3717 (17)	C1—O1	1.4241 (16)
C3—C2	1.4078 (17)	C1—H1A	0.96
C5—C7	1.4025 (17)	C1—H1B	0.96
C5—H5	0.93	C1—H1C	0.96
C7—C6	1.3885 (18)	O2—H2	0.82

C2—O1	1.3668 (15)	N1—N2	1.3750 (14)
C2—C4	1.3831 (19)	N2—H2A	0.86
O3—C9—O4	124.74 (12)	C2—C4—H4	120.1
O3—C9—N2	125.75 (11)	C6—C4—H4	120.1
O4—C9—N2	109.51 (10)	O4—C10—H10A	109.5
N1—C8—C7	121.11 (11)	O4—C10—H10B	109.5
N1—C8—H8	119.4	H10A—C10—H10B	109.5
C7—C8—H8	119.4	O4—C10—H10C	109.5
O2—C3—C5	118.24 (11)	H10A—C10—H10C	109.5
O2—C3—C2	121.38 (11)	H10B—C10—H10C	109.5
C5—C3—C2	120.37 (11)	O1—C1—H1A	109.5
C3—C5—C7	120.40 (11)	O1—C1—H1B	109.5
C3—C5—H5	119.8	H1A—C1—H1B	109.5
C7—C5—H5	119.8	O1—C1—H1C	109.5
C6—C7—C5	118.74 (12)	H1A—C1—H1C	109.5
C6—C7—C8	119.87 (11)	H1B—C1—H1C	109.5
C5—C7—C8	121.36 (11)	C2—O1—C1	117.31 (11)
O1—C2—C4	126.04 (11)	C9—O4—C10	116.53 (10)
O1—C2—C3	114.48 (11)	C3—O2—H2	109.5
C4—C2—C3	119.47 (11)	C8—N1—N2	116.16 (10)
C4—C6—C7	121.22 (12)	C9—N2—N1	118.88 (10)
C4—C6—H6	119.4	C9—N2—H2A	120.6
C7—C6—H6	119.4	N1—N2—H2A	120.6
C2—C4—C6	119.79 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.82	2.28	2.6871 (13)	112
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