Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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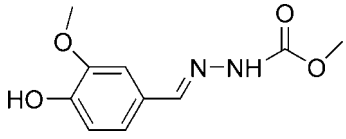
Received 27 June 2008; accepted 28 June 2008

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.113; data-to-parameter ratio = 12.9.

The molecule of the title compound, $C_{10}H_{12}N_2O_4$, adopts a *trans* configuration with respect to the C=N double bond. The dihedral angle between the benzene ring and the hydrazinecarboxylate mean plane is 36.54 (6)°. The molecules are linked into a two-dimensional network by intermolecular O—H···O, N—H···O and O—H···N hydrogen bonds, and by aromatic π–π stacking interactions [ring-centroid separation 3.7689 (9) Å].

Related literature

For a related structure, see: Cheng (2008).



Experimental

Crystal data

$C_{10}H_{12}N_2O_4$
M_r = 224.22
Monoclinic, $P2_1/c$
a = 9.4718 (10) Å
b = 11.0983 (11) Å
c = 10.3220 (11) Å
β = 98.272 (4)°
V = 1073.77 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 123 (2) K
0.29 × 0.27 × 0.26 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.965, T_{max} = 0.968
11214 measured reflections
1887 independent reflections
1590 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.113
S = 1.02
1887 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.84	2.21	2.6695 (15)	114
O1—H1···O3^{i}	0.84	2.34	3.0286 (16)	139
O1—H1···N1^{i}	0.84	2.57	3.2640 (17)	140
N2—H2*B*···O3^{ii}	0.88	2.19	3.0124 (16)	155

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, -y-\frac{1}{2}, z+\frac{1}{2}$.

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*.

The author acknowledges financial support from Zhejiang Police College, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2754).

References

Bruker (2002). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, X.-W. (2008). *Acta Cryst.* E**64**, o1302.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o1397 [doi:10.1107/S1600536808019788]

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

Xiang-Wei Cheng

S1. Comment

As part of our ongoig studies of benzaldehydehydrazone derivatives (Cheng, 2008), we now report the synthesis and structure of the title compound, (I).

The title molecule (Fig. 1) adopts a *trans* configuration with respect to the C═N bond. The dihedral angle between the benzene ring and the C9/C10//N1/N2/O3/O4 plane is 36.54 (6)°. Otherwise, the bond lengths and angles for (I) agree with those observed for (*E*)-methyl*N*′-(4-hydroxybenzylidene)hydrazinecarboxylate (Cheng, 2008).

In the crystal, the molecules are linked into a two-dimensional network by intramolecular O—H···O and intermolecular O—H···O, N—H···O, O—H···N hydrogen bonds (Table 1). Additionally, neighbouring aromatic rings interact by π–π stacking [centroid separation = 3.7689 (9) Å].

S2. Experimental

4-Hydroxy-3-methoxybenzaldehyde (1.52 g, 0.01 mol) and methyl hydrazinecarboxylate (0.9 g, 0.01 mol) were dissolved in stirred methanol (15 ml) and left for 2.5 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 90% yield. Colourless blocks of (I) were obtained by slow evaporation of a ethanol solution at room temperature (m.p. 480–482 K).

S3. Refinement

The H atoms were placed geometrically (O—H = 0.84 Å, N—H = 0.88 Å and C—H = 0.95 or 0.98 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5U_{eq}(carrier).

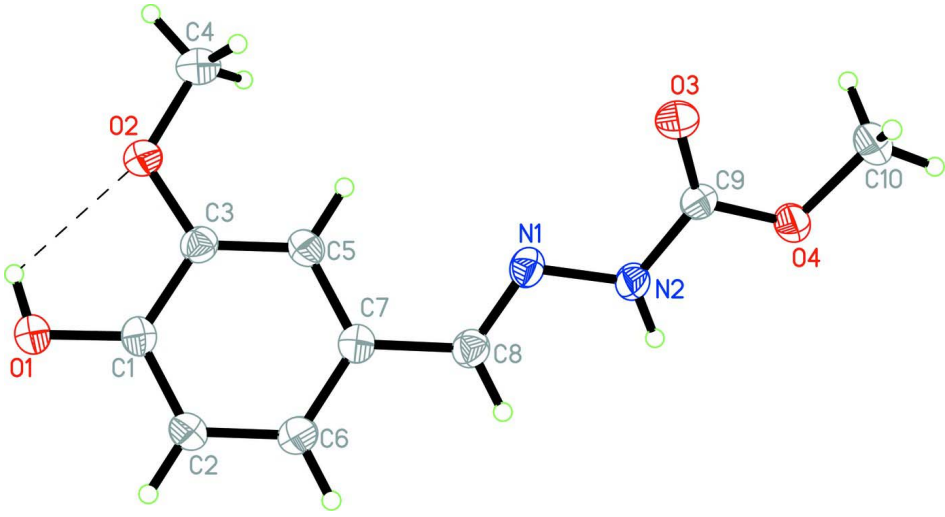


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids for the non-hydrogen atoms.

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

Crystal data

$C_{10}H_{12}N_2O_4$
$M_r = 224.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.4718\ (10)$ Å
$b = 11.0983\ (11)$ Å
$c = 10.3220\ (11)$ Å
$\beta = 98.272\ (4)°$
$V = 1073.77\ (19)$ Å^3
$Z = 4$
$F(000) = 472$
$D_x = 1.387$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1887 reflections
$\theta = 2.2–25.0°$
$\mu = 0.11$ mm^{-1}
$T = 123$ K
Block, colourless
0.29 × 0.27 × 0.26 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.965$, $T_{max} = 0.968$
11214 measured reflections
1887 independent reflections
1590 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -11\rightarrow10$
$k = -13\rightarrow13$
$l = -11\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.113$
$S = 1.02$
1887 reflections
146 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
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 0.1817P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.010$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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

Extinction coefficient: 0.036 (5)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.46841 (16)	0.22471 (12)	0.90488 (15)	0.0408 (4)
C2	0.57541 (17)	0.23855 (13)	1.00956 (16)	0.0453 (4)
H2A	0.5760	0.3073	1.0645	0.054*
C3	0.46883 (16)	0.12325 (13)	0.82397 (14)	0.0405 (4)
C4	0.3402 (2)	0.01177 (17)	0.64655 (17)	0.0596 (5)
H4A	0.2550	0.0191	0.5809	0.089*
H4B	0.3309	−0.0589	0.7016	0.089*
H4C	0.4246	0.0026	0.6025	0.089*
C5	0.57560 (15)	0.03861 (13)	0.84778 (14)	0.0406 (4)
H5	0.5761	−0.0291	0.7916	0.049*
C6	0.68260 (16)	0.15212 (14)	1.03504 (15)	0.0439 (4)
H6	0.7554	0.1618	1.1079	0.053*
C7	0.68361 (15)	0.05223 (13)	0.95482 (14)	0.0390 (4)
C8	0.79364 (16)	−0.04036 (13)	0.98484 (15)	0.0425 (4)
H8	0.8497	−0.0414	1.0689	0.051*
C9	0.96010 (15)	−0.28026 (12)	0.85416 (14)	0.0381 (4)
C10	1.0957 (2)	−0.45396 (18)	0.82782 (19)	0.0681 (6)
H10A	1.1524	−0.5152	0.8804	0.102*
H10B	1.1562	−0.4113	0.7734	0.102*
H10C	1.0166	−0.4929	0.7715	0.102*
N1	0.81595 (13)	−0.11952 (11)	0.90087 (12)	0.0409 (3)
N2	0.91622 (13)	−0.20662 (11)	0.94426 (12)	0.0434 (3)
H2B	0.9502	−0.2137	1.0279	0.052*
O1	0.36374 (12)	0.30938 (9)	0.88270 (11)	0.0543 (3)
H1	0.3060	0.2900	0.8164	0.081*
O2	0.35509 (13)	0.11728 (10)	0.72612 (11)	0.0571 (4)
O3	0.93369 (12)	−0.26695 (10)	0.73659 (10)	0.0508 (3)
O4	1.03976 (12)	−0.36926 (10)	0.91342 (10)	0.0520 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0423 (8)	0.0368 (7)	0.0437 (9)	0.0025 (6)	0.0071 (6)	0.0028 (6)

C2	0.0511 (9)	0.0373 (8)	0.0469 (9)	0.0004 (6)	0.0045 (7)	−0.0050 (6)
C3	0.0410 (8)	0.0445 (8)	0.0360 (8)	0.0010 (6)	0.0055 (6)	0.0012 (6)
C4	0.0648 (11)	0.0618 (10)	0.0485 (10)	0.0017 (9)	−0.0038 (8)	−0.0113 (8)
C5	0.0432 (8)	0.0401 (8)	0.0398 (8)	0.0016 (6)	0.0101 (6)	−0.0025 (6)
C6	0.0431 (8)	0.0458 (8)	0.0415 (9)	−0.0022 (7)	0.0018 (6)	0.0014 (6)
C7	0.0387 (8)	0.0403 (7)	0.0390 (8)	−0.0002 (6)	0.0095 (6)	0.0040 (6)
C8	0.0431 (8)	0.0455 (8)	0.0387 (8)	0.0030 (6)	0.0056 (6)	0.0025 (6)
C9	0.0352 (7)	0.0434 (8)	0.0349 (8)	−0.0014 (6)	0.0029 (6)	0.0006 (6)
C10	0.0767 (13)	0.0687 (12)	0.0567 (11)	0.0307 (10)	0.0019 (9)	−0.0149 (9)
N1	0.0393 (7)	0.0442 (7)	0.0393 (7)	0.0059 (5)	0.0064 (5)	0.0070 (5)
N2	0.0465 (7)	0.0500 (7)	0.0328 (7)	0.0132 (6)	0.0033 (5)	0.0023 (5)
O1	0.0565 (7)	0.0465 (6)	0.0559 (7)	0.0137 (5)	−0.0053 (5)	−0.0084 (5)
O2	0.0551 (7)	0.0610 (7)	0.0501 (7)	0.0157 (5)	−0.0093 (5)	−0.0147 (5)
O3	0.0589 (7)	0.0593 (7)	0.0336 (7)	0.0073 (5)	0.0046 (5)	0.0001 (5)
O4	0.0612 (7)	0.0534 (7)	0.0402 (6)	0.0199 (5)	0.0026 (5)	−0.0030 (5)

Geometric parameters (Å, º)

C1—O1	1.3613 (17)	C6—H6	0.9500
C1—C2	1.380 (2)	C7—C8	1.465 (2)
C1—C3	1.402 (2)	C8—N1	1.2729 (19)
C2—C6	1.394 (2)	C8—H8	0.9500
C2—H2A	0.9500	C9—O3	1.2123 (18)
C3—O2	1.3682 (18)	C9—O4	1.3366 (17)
C3—C5	1.376 (2)	C9—N2	1.3479 (19)
C4—O2	1.4256 (19)	C10—O4	1.4421 (19)
C4—H4A	0.9800	C10—H10A	0.9800
C4—H4B	0.9800	C10—H10B	0.9800
C4—H4C	0.9800	C10—H10C	0.9800
C5—C7	1.402 (2)	N1—N2	1.3837 (16)
C5—H5	0.9500	N2—H2B	0.8800
C6—C7	1.385 (2)	O1—H1	0.8400
O1—C1—C2	119.35 (13)	C6—C7—C8	120.08 (13)
O1—C1—C3	121.23 (13)	C5—C7—C8	120.50 (13)
C2—C1—C3	119.42 (13)	N1—C8—C7	121.53 (13)
C1—C2—C6	120.28 (14)	N1—C8—H8	119.2
C1—C2—H2A	119.9	C7—C8—H8	119.2
C6—C2—H2A	119.9	O3—C9—O4	124.73 (13)
O2—C3—C5	125.38 (13)	O3—C9—N2	125.25 (13)
O2—C3—C1	114.15 (13)	O4—C9—N2	110.01 (12)
C5—C3—C1	120.45 (13)	O4—C10—H10A	109.5
O2—C4—H4A	109.5	O4—C10—H10B	109.5
O2—C4—H4B	109.5	H10A—C10—H10B	109.5
H4A—C4—H4B	109.5	O4—C10—H10C	109.5
O2—C4—H4C	109.5	H10A—C10—H10C	109.5
H4A—C4—H4C	109.5	H10B—C10—H10C	109.5
H4B—C4—H4C	109.5	C8—N1—N2	115.80 (12)

C3—C5—C7	120.09 (13)	C9—N2—N1	117.75 (12)
C3—C5—H5	120.0	C9—N2—H2B	121.1
C7—C5—H5	120.0	N1—N2—H2B	121.1
C7—C6—C2	120.37 (14)	C1—O1—H1	109.5
C7—C6—H6	119.8	C3—O2—C4	117.85 (12)
C2—C6—H6	119.8	C9—O4—C10	115.75 (12)
C6—C7—C5	119.38 (13)		
O1—C1—C2—C6	179.17 (14)	C3—C5—C7—C8	176.91 (13)
C3—C1—C2—C6	−0.3 (2)	C6—C7—C8—N1	−165.42 (14)
O1—C1—C3—O2	−1.5 (2)	C5—C7—C8—N1	16.9 (2)
C2—C1—C3—O2	177.96 (14)	C7—C8—N1—N2	−175.85 (12)
O1—C1—C3—C5	179.93 (14)	O3—C9—N2—N1	10.3 (2)
C2—C1—C3—C5	−0.6 (2)	O4—C9—N2—N1	−170.91 (11)
O2—C3—C5—C7	−177.24 (13)	C8—N1—N2—C9	−169.52 (13)
C1—C3—C5—C7	1.1 (2)	C5—C3—O2—C4	4.0 (2)
C1—C2—C6—C7	0.7 (2)	C1—C3—O2—C4	−174.49 (14)
C2—C6—C7—C5	−0.1 (2)	O3—C9—O4—C10	−0.3 (2)
C2—C6—C7—C8	−177.83 (13)	N2—C9—O4—C10	−179.09 (14)
C3—C5—C7—C6	−0.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.84	2.21	2.6695 (15)	114
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