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Methyl (*E*)-*N*′-[1-(2,4-dihydroxyphenyl)-ethylidene]hydrazinecarboxylate

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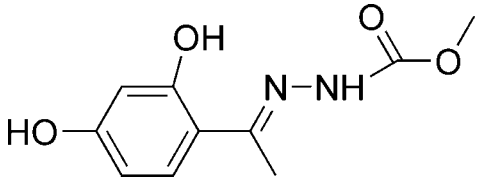
Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.131; data-to-parameter ratio = 12.2.

The molecule of the title compound, $C_{10}H_{12}N_2O_4$, adopts a *trans* configuration with respect to the C=N bond. The dihedral angle between the benzene ring and the methyl hydrazinecarboxylate plane is 3.01 (6)°. An intramolecular O—H···N hydrogen bond is observed. In the crystal, molecules are linked into a two-dimensional network parallel to $(10\bar{1})$ by O—H···O, N—H···O and C—H···O hydrogen bonds.

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 a related structure, see: Lv *et al.* (2008).



Experimental

Crystal data

$C_{10}H_{12}N_2O_4$
M_r = 224.22
Monoclinic, $P2_1/n$
a = 10.714 (5) Å
b = 8.700 (4) Å
c = 11.682 (6) Å
β = 107.872 (6)°
V = 1036.3 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 223 K
0.24 × 0.23 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*, Bruker, 2002)
T_{min} = 0.977, T_{max} = 0.980
5096 measured reflections
1815 independent reflections
1355 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.131
S = 1.05
1815 reflections
149 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 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···N1	0.82	1.84	2.557 (2)	145
O1—H1···O3^{i}	0.82	1.98	2.791 (2)	170
N2—H2B···O2^{ii}	0.86	2.35	3.185 (2)	164
C2—H2A···O3^{i}	0.93	2.42	3.141 (3)	135

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

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: CI2854).

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Methyl (*E*)-*N*′-[1-(2,4-dihydroxyphenyl)ethylidene]hydrazinecarboxylate

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

S1. Comment

Benzaldehydehydrazone derivatives have received considerable attention for a long time, due to their pharmacological activities (Parashar *et al.*, 1988) and their photochromic properties (Hadjoudis *et al.*, 1987). They are important intermidiates for 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many properties (Borg *et al.*, 1999). As a further investigation of this type of derivatives, we report herein the crystal structure of the title compound.

The title molecule (Fig. 1) adopts a trans configuration with respect to the C═N double bond. The bond lengths and angles are comparable to those observed for (E)-methyl N′-[1-(4-methoxyphenyl)ethylidene]hydrazinecarboxylate (Lv *et al.*, 2008). The dihedral angle between benzene (C1-C6) and O3/O4/N1/N2/C7-C10 planes is 3.01 (6)°. An intramolecular O2—H2···N1 hydrogen bond is observed.

In the crystal structure, intermolecular O—H···O, N—H···O and C–H···O hydrogen bonds (Table 1) link the molecules into a two-dimensional network parallel to the $(10\bar{1})$ (Fig. 2).

S2. Experimental

2,4-Dihydroxy-acetophenone (1.52 g, 0.01 mol) and methyl hydrazinecarboxylate (0.90 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 (yield 93%, m.p. 475-478 K). Single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

S3. Refinement

H atoms were positioned geometrically (N-H = 0.86 Å, O-H = 0.82Å and C-H =0.93 and 0.96 Å) and constrained to ride on their parent atoms with $U_{iso}(H) = xU_{eq}(C,N)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms. A rotating group model was used for the methyl groups.

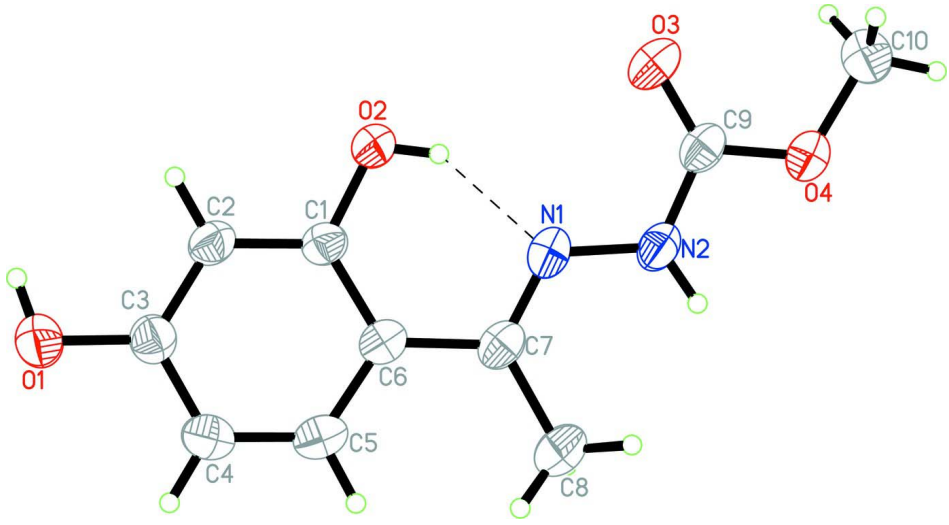

Figure 1

The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. The dashed line indicates a hydrogen bond.

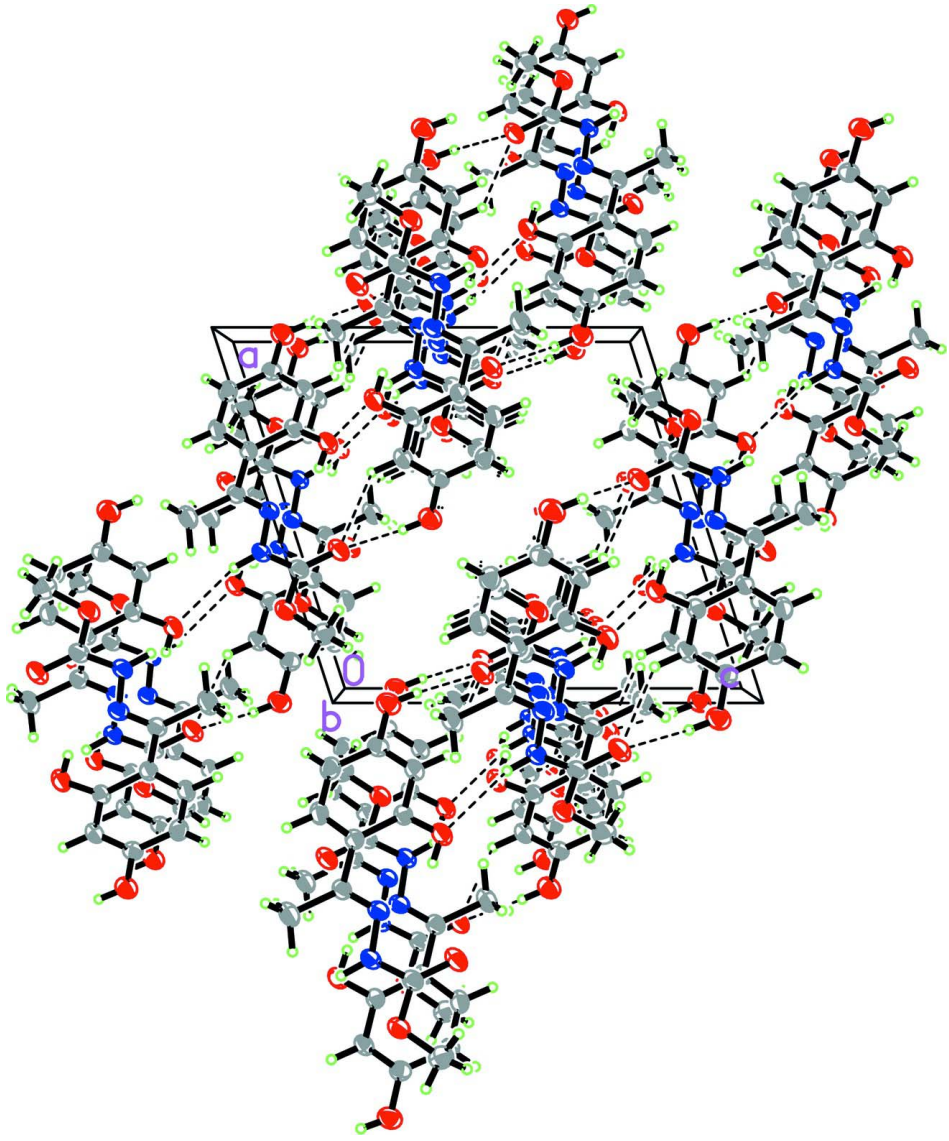

Figure 2

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

Methyl (*E*)-*N*′-[1-(2,4-dihydroxyphenyl)ethylidene]hydrazinecarboxylate

Crystal data

$C_{10}H_{12}N_2O_4$
$M_r = 224.22$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.714\ (5)$ Å
$b = 8.700\ (4)$ Å
$c = 11.682\ (6)$ Å
$\beta = 107.872\ (6)°$
$V = 1036.3\ (9)$ Å^3
$Z = 4$

$F(000) = 472$
$D_x = 1.437$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1915 reflections
$\theta = 2.3–25.0°$
$\mu = 0.11$ mm^{-1}
$T = 223$ K
Block, colourless
0.24 × 0.23 × 0.19 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} = 0.977, T_{max} = 0.980
5096 measured reflections
1815 independent reflections
1355 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −12→12
k = −10→10
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.131
S = 1.05
1815 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
$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 0.2997P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.025
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O4	0.75341 (14)	0.02754 (18)	0.04159 (13)	0.0531 (4)
O2	0.30274 (15)	0.34157 (19)	−0.17412 (12)	0.0549 (5)
H2	0.3710	0.2990	−0.1355	0.082*
O1	−0.00973 (15)	0.70770 (19)	−0.13812 (14)	0.0615 (5)
H1	−0.0390	0.6873	−0.2100	0.092*
O3	0.59381 (15)	0.1012 (2)	−0.12508 (13)	0.0592 (5)
C6	0.32217 (19)	0.4395 (2)	0.02586 (17)	0.0403 (5)
C7	0.44242 (19)	0.3525 (2)	0.08526 (18)	0.0426 (5)
C2	0.1513 (2)	0.5234 (2)	−0.15377 (18)	0.0461 (5)
H2A	0.1139	0.5203	−0.2369	0.055*
C9	0.64512 (19)	0.1050 (2)	−0.01725 (18)	0.0443 (5)
C1	0.26013 (19)	0.4342 (2)	−0.09982 (17)	0.0404 (5)
C3	0.0972 (2)	0.6171 (2)	−0.08632 (19)	0.0458 (5)
C5	0.2630 (2)	0.5360 (3)	0.09052 (19)	0.0501 (6)
H5	0.3002	0.5422	0.1735	0.060*
C4	0.1528 (2)	0.6219 (3)	0.03705 (19)	0.0522 (6)

H4	0.1160	0.6829	0.0836	0.063*
C10	0.8151 (2)	−0.0586 (3)	−0.0320 (2)	0.0615 (7)
H10A	0.7509	−0.1217	−0.0874	0.092*
H10B	0.8830	−0.1225	0.0184	0.092*
H10C	0.8526	0.0113	−0.0760	0.092*
C8	0.5058 (2)	0.3622 (3)	0.21894 (19)	0.0646 (7)
H8A	0.5988	0.3485	0.2374	0.097*
H8B	0.4708	0.2833	0.2577	0.097*
H8C	0.4882	0.4611	0.2471	0.097*
N2	0.60110 (16)	0.1850 (2)	0.06181 (15)	0.0469 (5)
H2B	0.6418	0.1831	0.1377	0.056*
N1	0.48811 (16)	0.2697 (2)	0.01548 (15)	0.0448 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0468 (9)	0.0614 (10)	0.0448 (9)	0.0087 (7)	0.0047 (7)	0.0031 (7)
O2	0.0550 (9)	0.0643 (11)	0.0377 (8)	0.0175 (8)	0.0030 (7)	−0.0073 (7)
O1	0.0555 (10)	0.0627 (11)	0.0593 (10)	0.0148 (8)	0.0072 (8)	−0.0074 (8)
O3	0.0557 (9)	0.0700 (11)	0.0409 (9)	−0.0038 (8)	−0.0012 (7)	−0.0013 (8)
C6	0.0421 (11)	0.0408 (12)	0.0367 (11)	−0.0074 (9)	0.0102 (9)	0.0013 (9)
C7	0.0427 (11)	0.0445 (12)	0.0380 (11)	−0.0073 (9)	0.0084 (9)	0.0079 (9)
C2	0.0478 (12)	0.0482 (13)	0.0362 (11)	0.0001 (10)	0.0041 (9)	−0.0042 (9)
C9	0.0403 (11)	0.0470 (13)	0.0401 (12)	−0.0083 (10)	0.0044 (9)	0.0045 (10)
C1	0.0432 (11)	0.0401 (12)	0.0361 (11)	−0.0026 (9)	0.0096 (9)	−0.0024 (9)
C3	0.0423 (11)	0.0439 (12)	0.0489 (12)	−0.0008 (9)	0.0107 (10)	−0.0023 (10)
C5	0.0568 (13)	0.0568 (14)	0.0357 (11)	−0.0069 (11)	0.0127 (10)	−0.0018 (10)
C4	0.0574 (13)	0.0538 (14)	0.0482 (13)	0.0002 (11)	0.0202 (11)	−0.0081 (11)
C10	0.0548 (14)	0.0669 (16)	0.0635 (16)	0.0034 (12)	0.0191 (12)	−0.0028 (13)
C8	0.0664 (15)	0.0796 (18)	0.0404 (13)	0.0092 (13)	0.0053 (11)	0.0068 (12)
N2	0.0413 (10)	0.0562 (11)	0.0370 (9)	0.0021 (8)	0.0031 (8)	0.0065 (8)
N1	0.0377 (9)	0.0485 (11)	0.0435 (10)	−0.0013 (8)	0.0055 (7)	0.0078 (8)

Geometric parameters (Å, º)

O4—C9	1.335 (2)	C2—H2A	0.93
O4—C10	1.445 (3)	C9—N2	1.352 (3)
O2—C1	1.362 (2)	C3—C4	1.381 (3)
O2—H2	0.82	C5—C4	1.375 (3)
O1—C3	1.369 (2)	C5—H5	0.93
O1—H1	0.82	C4—H4	0.93
O3—C9	1.210 (2)	C10—H10A	0.96
C6—C5	1.404 (3)	C10—H10B	0.96
C6—C1	1.414 (3)	C10—H10C	0.96
C6—C7	1.472 (3)	C8—H8A	0.96
C7—N1	1.291 (3)	C8—H8B	0.96
C7—C8	1.502 (3)	C8—H8C	0.96
C2—C3	1.378 (3)	N2—N1	1.378 (2)

C2—C1	1.381 (3)	N2—H2B	0.86
C9—O4—C10	116.06 (17)	C4—C5—H5	118.4
C1—O2—H2	109.5	C6—C5—H5	118.4
C3—O1—H1	109.5	C5—C4—C3	119.5 (2)
C5—C6—C1	115.61 (19)	C5—C4—H4	120.2
C5—C6—C7	121.90 (19)	C3—C4—H4	120.2
C1—C6—C7	122.48 (19)	O4—C10—H10A	109.5
N1—C7—C6	115.85 (18)	O4—C10—H10B	109.5
N1—C7—C8	123.37 (19)	H10A—C10—H10B	109.5
C6—C7—C8	120.8 (2)	O4—C10—H10C	109.5
C3—C2—C1	121.02 (19)	H10A—C10—H10C	109.5
C3—C2—H2A	119.5	H10B—C10—H10C	109.5
C1—C2—H2A	119.5	C7—C8—H8A	109.5
O3—C9—O4	124.6 (2)	C7—C8—H8B	109.5
O3—C9—N2	125.6 (2)	H8A—C8—H8B	109.5
O4—C9—N2	109.78 (17)	C7—C8—H8C	109.5
O2—C1—C2	116.30 (17)	H8A—C8—H8C	109.5
O2—C1—C6	122.56 (18)	H8B—C8—H8C	109.5
C2—C1—C6	121.14 (19)	C9—N2—N1	117.11 (17)
O1—C3—C2	121.94 (19)	C9—N2—H2B	121.4
O1—C3—C4	118.56 (19)	N1—N2—H2B	121.4
C2—C3—C4	119.5 (2)	C7—N1—N2	120.52 (17)
C4—C5—C6	123.2 (2)		
C5—C6—C7—N1	−178.96 (18)	C1—C2—C3—O1	179.24 (19)
C1—C6—C7—N1	−0.3 (3)	C1—C2—C3—C4	0.1 (3)
C5—C6—C7—C8	0.9 (3)	C1—C6—C5—C4	−0.6 (3)
C1—C6—C7—C8	179.6 (2)	C7—C6—C5—C4	178.17 (19)
C10—O4—C9—O3	3.7 (3)	C6—C5—C4—C3	−1.2 (3)
C10—O4—C9—N2	−177.44 (17)	O1—C3—C4—C5	−177.7 (2)
C3—C2—C1—O2	177.60 (19)	C2—C3—C4—C5	1.5 (3)
C3—C2—C1—C6	−2.0 (3)	O3—C9—N2—N1	−0.1 (3)
C5—C6—C1—O2	−177.40 (18)	O4—C9—N2—N1	−178.99 (16)
C7—C6—C1—O2	3.8 (3)	C6—C7—N1—N2	179.30 (16)
C5—C6—C1—C2	2.2 (3)	C8—C7—N1—N2	−0.5 (3)
C7—C6—C1—C2	−176.56 (19)	C9—N2—N1—C7	−178.00 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82	1.84	2.557 (2)	145
O1—H1···O3^{i}	0.82	1.98	2.791 (2)	170
N2—H2*B*···O2^{ii}	0.86	2.35	3.185 (2)	164
C2—H2*A*···O3^{i}	0.93	2.42	3.141 (3)	135

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*−1/2; (ii) *x*+1/2, −*y*+1/2, *z*+1/2.