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(*E*)-Methyl *N*′-[1-(4-methoxyphenyl)-ethylidene]hydrazinecarboxylate

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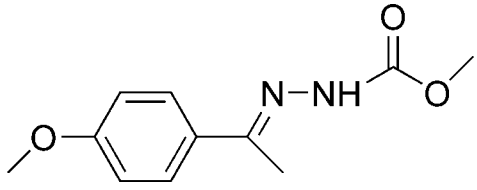
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.102; data-to-parameter ratio = 12.8.

The molecule of the title compound, $C_{11}H_{14}N_2O_3$, adopts a *trans* configuration with respect to the C=N bond. The dihedral angle between the benzene ring and the hydrazinecarboxylate plane is 12.06 (9)°. Molecules are linked into a one-dimensional network by N—H···O hydrogen bonds and C—H···π interactions. The benzene rings of inversion-related molecules are stacked with their centroids separated by 3.777 (1) Å, indicating π–π interactions.

Related literature

For general background, see: Parashar *et al.* (1988); Hadjoudis *et al.* (1987); Borg *et al.* (1999). For related structures, see: Shang *et al.* (2007).



Experimental

Crystal data

$C_{11}H_{14}N_2O_3$ M_r = 222.24
Monoclinic, $P2_1/c$
a = 12.416 (3) Å
b = 11.113 (3) Å
c = 8.073 (2) Å
β = 95.628 (3)°
V = 1108.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 (2) K
0.30 × 0.26 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.972, T_{max} = 0.978
7124 measured reflections
1952 independent reflections
1624 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.07
1952 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 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
N2—H2A···O2^{i}	0.89 (2)	2.01 (2)	2.8864 (17)	169.0
C4—H4···$Cg1^{ii}$	0.93	2.87	3.6488 (19)	142

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$. $Cg1$ is the centroid of the benzene 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: ZL2130).

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(*E*)-Methyl *N*′-[1-(4-methoxyphenyl)ethylidene]hydrazinecarboxylate

Lu-Ping Lv, Wei-Ping Yu, Wen-Bo Yu, Xue-Feng Zhou and Xian-Chao Hu

S1. Comment

Benzaldehydehydrazone derivatives have received considerable attention for a long time due to their pharmacological activity (Parashar *et al.*, 1988) and their photochromic properties (Hadjoudis *et al.*, 1987). Meanwhile, it's an important intermidiate of 1,3,4-oxadiazoles, which have been reported to be versatile compounds with many interesting properties (Borg *et al.*, 1999). As a further investigation of this type of derivatives, the crystal structure of the title compound, $C_{11}H_{14}N_2O_3$ (Fig.1), is described here.

The title molecule (Fig.1) adopts a *trans* configuration with respect to the C═N bond. The N1/N2/O2/O3/C10/C11 plane of the hydrazine carboxylic acid methyl ester group is slightly twisted away from the attached ring. The dihedral angle between the C2—C7 ring and the N1/N2/O2/O3/C10/C11 plane is 12.06 (9)°. The bond lengths and angles agree with those observed for methyl *N*′-[(*E*)-4-methoxybenzylidene] hydrazinecarboxylate (Shang *et al.*, 2007).

The molecules are linked into a one-dimensional network by N–H···O hydrogen bonds and C–H···π interactions (Table 1, Fig.2). The benzene rings of the inversion-related molecules are stacked with their centroids separated by a distance of 3.777 (1) Å, indicating π-π interactions.

S2. Experimental

4-Methoxy-acetophenone (1.50 g, 0.01 mol) and methyl hydrazinecarboxylate (0.90 g, 0.01 mol) were dissolved in stirred methanol (15 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 80% yield. Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 470–472 K).

S3. Refinement

The H atoms attached N2 were located in a difference map and its position and Uiso values were freely refined. C-bound H atoms were positioned geometrically (C—H = 0.93 or 0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

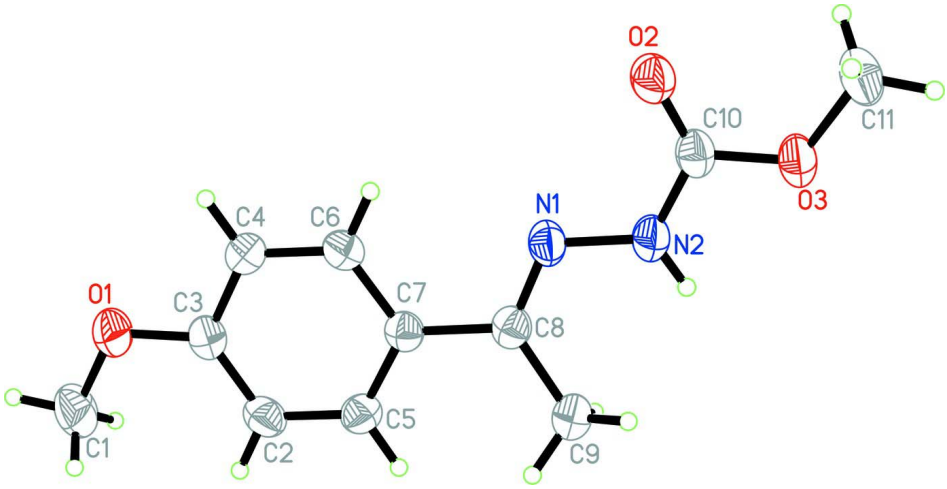

Figure 1

Molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

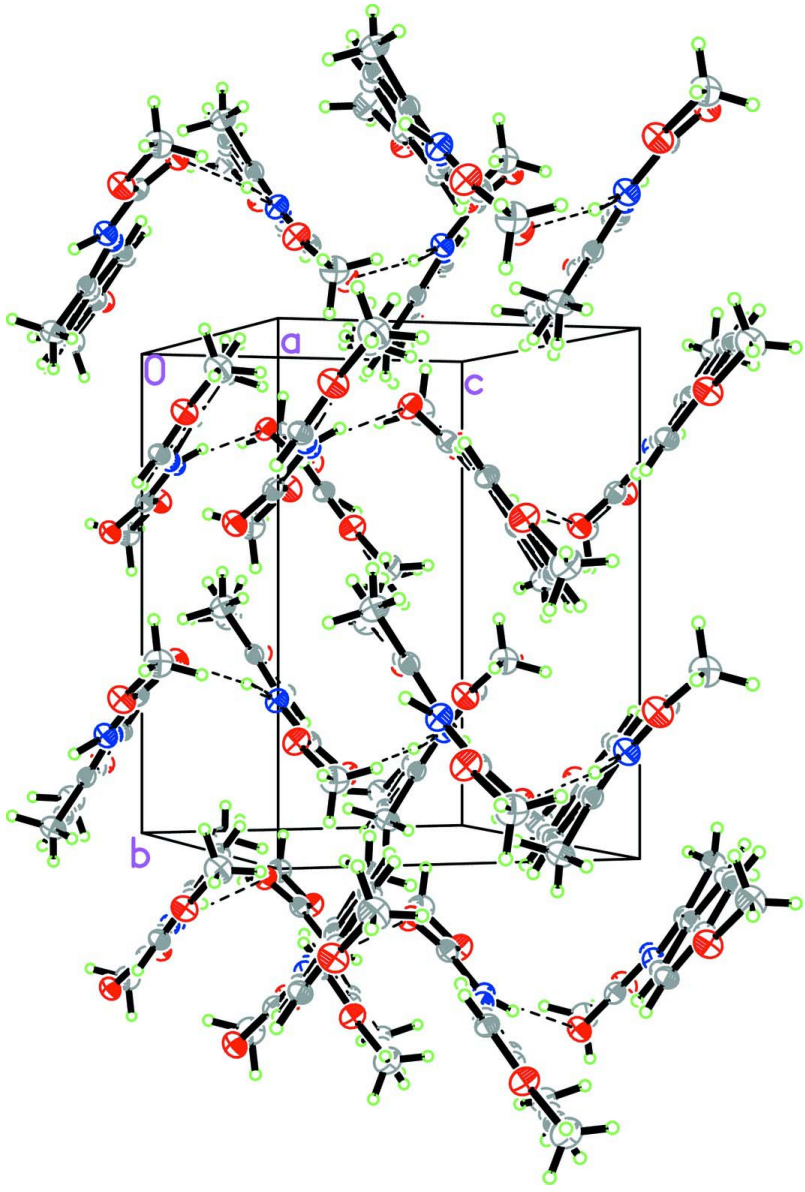

Figure 2

Crystal packing of the title compound, viewed approximately down the *a* axis. Dashed lines indicate intermolecular hydrogen bonds.

(*E*)-Methyl N′-[1-(4-methoxyphenyl)ethylidene]hydrazinecarboxylate

Crystal data

$C_{11}H_{14}N_2O_3$
$M_r = 222.24$
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Hall symbol: -P 2ybc
$a = 12.416$ (3) Å
$b = 11.113$ (3) Å
$c = 8.073$ (2) Å
$\beta = 95.628$ (3)°
$V = 1108.5$ (5) Å^3
$Z = 4$
$F(000) = 472$
$D_x = 1.332$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1952 reflections
$\theta = 1.6$–25.0°
$\mu = 0.10$ mm^{-1}

T = 273 K
Block, colourless

0.30 × 0.26 × 0.25 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} = 0.972, T_{max} = 0.978

7124 measured reflections
1952 independent reflections
1624 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°, θ_{min} = 1.7°
h = −14→14
k = −13→11
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 1.07
1952 reflections
153 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.2826P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.042 (4)

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}
H2A	0.6796 (14)	0.1822 (17)	0.308 (2)	0.059 (5)*
C7	0.96977 (11)	0.12453 (12)	0.16378 (16)	0.0297 (3)
C8	0.85779 (11)	0.12580 (12)	0.21369 (16)	0.0317 (3)
C10	0.62251 (11)	0.29834 (13)	0.14528 (18)	0.0348 (3)
C3	1.18244 (11)	0.12536 (13)	0.07457 (18)	0.0341 (3)
C5	1.04408 (11)	0.03686 (13)	0.22113 (17)	0.0351 (4)
H5	1.0223	−0.0236	0.2902	0.042*
C6	1.00522 (11)	0.21282 (12)	0.05809 (17)	0.0348 (4)
H6	0.9574	0.2727	0.0174	0.042*
C2	1.14990 (11)	0.03671 (13)	0.17850 (18)	0.0375 (4)
H2	1.1983	−0.0225	0.2196	0.045*
C4	1.10911 (11)	0.21306 (13)	0.01308 (18)	0.0364 (4)

H4	1.1305	0.2720	−0.0587	0.044*
C1	1.36336 (12)	0.05169 (17)	0.0921 (2)	0.0551 (5)
H1A	1.3691	0.0574	0.2113	0.083*
H1B	1.4320	0.0704	0.0531	0.083*
H1C	1.3425	−0.0286	0.0588	0.083*
C11	0.44286 (12)	0.36579 (16)	0.1528 (2)	0.0528 (5)
H11A	0.4628	0.4492	0.1589	0.079*
H11B	0.3810	0.3529	0.2132	0.079*
H11C	0.4254	0.3433	0.0385	0.079*
O1	1.28412 (8)	0.13431 (10)	0.02268 (14)	0.0460 (3)
O3	0.53202 (8)	0.29346 (10)	0.22464 (14)	0.0467 (3)
O2	0.63408 (8)	0.36576 (10)	0.03058 (13)	0.0432 (3)
N1	0.79718 (9)	0.21424 (11)	0.15959 (14)	0.0350 (3)
N2	0.69424 (10)	0.21707 (12)	0.21353 (17)	0.0391 (3)
C9	0.82244 (12)	0.02704 (14)	0.3229 (2)	0.0439 (4)
H9A	0.8329	0.0521	0.4372	0.066*
H9B	0.8646	−0.0439	0.3082	0.066*
H9C	0.7472	0.0098	0.2933	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C7	0.0318 (7)	0.0303 (7)	0.0271 (7)	−0.0005 (5)	0.0034 (5)	−0.0038 (5)
C8	0.0343 (7)	0.0334 (8)	0.0277 (7)	−0.0011 (6)	0.0052 (5)	−0.0037 (6)
C10	0.0317 (7)	0.0391 (8)	0.0346 (8)	0.0006 (6)	0.0082 (6)	−0.0059 (7)
C3	0.0301 (7)	0.0370 (8)	0.0357 (8)	0.0004 (6)	0.0048 (6)	−0.0050 (6)
C5	0.0388 (8)	0.0333 (8)	0.0336 (8)	0.0002 (6)	0.0058 (6)	0.0038 (6)
C6	0.0342 (7)	0.0328 (8)	0.0374 (8)	0.0051 (6)	0.0041 (6)	0.0021 (6)
C2	0.0362 (8)	0.0357 (8)	0.0403 (8)	0.0086 (6)	0.0021 (6)	0.0019 (6)
C4	0.0361 (8)	0.0354 (8)	0.0386 (8)	−0.0008 (6)	0.0079 (6)	0.0049 (6)
C1	0.0332 (8)	0.0649 (12)	0.0680 (12)	0.0125 (8)	0.0084 (8)	0.0035 (9)
C11	0.0314 (8)	0.0592 (11)	0.0684 (12)	0.0078 (7)	0.0075 (7)	0.0000 (9)
O1	0.0304 (5)	0.0522 (7)	0.0565 (7)	0.0053 (5)	0.0107 (5)	0.0060 (5)
O3	0.0316 (5)	0.0567 (7)	0.0539 (7)	0.0066 (5)	0.0153 (5)	0.0079 (5)
O2	0.0425 (6)	0.0493 (7)	0.0396 (6)	0.0093 (5)	0.0125 (5)	0.0054 (5)
N1	0.0308 (6)	0.0412 (7)	0.0345 (7)	0.0028 (5)	0.0097 (5)	0.0002 (5)
N2	0.0330 (6)	0.0467 (8)	0.0393 (7)	0.0047 (5)	0.0131 (5)	0.0059 (6)
C9	0.0379 (8)	0.0442 (9)	0.0516 (9)	0.0011 (7)	0.0144 (7)	0.0075 (7)

Geometric parameters (Å, º)

C7—C5	1.3896 (19)	C2—H2	0.9300
C7—C6	1.399 (2)	C4—H4	0.9300
C7—C8	1.4850 (19)	C1—O1	1.4201 (19)
C8—N1	1.2878 (18)	C1—H1A	0.9600
C8—C9	1.500 (2)	C1—H1B	0.9600
C10—O2	1.2105 (17)	C1—H1C	0.9600
C10—N2	1.3476 (19)	C11—O3	1.4429 (19)

C10—O3	1.3479 (17)	C11—H11A	0.9600
C3—O1	1.3722 (17)	C11—H11B	0.9600
C3—C2	1.380 (2)	C11—H11C	0.9600
C3—C4	1.392 (2)	N1—N2	1.3905 (16)
C5—C2	1.390 (2)	N2—H2A	0.891 (19)
C5—H5	0.9300	C9—H9A	0.9600
C6—C4	1.3740 (19)	C9—H9B	0.9600
C6—H6	0.9300	C9—H9C	0.9600
C5—C7—C6	117.16 (13)	O1—C1—H1A	109.5
C5—C7—C8	121.66 (12)	O1—C1—H1B	109.5
C6—C7—C8	121.18 (12)	H1A—C1—H1B	109.5
N1—C8—C7	116.53 (12)	O1—C1—H1C	109.5
N1—C8—C9	124.24 (13)	H1A—C1—H1C	109.5
C7—C8—C9	119.23 (12)	H1B—C1—H1C	109.5
O2—C10—N2	127.18 (13)	O3—C11—H11A	109.5
O2—C10—O3	123.74 (13)	O3—C11—H11B	109.5
N2—C10—O3	109.08 (13)	H11A—C11—H11B	109.5
O1—C3—C2	124.78 (13)	O3—C11—H11C	109.5
O1—C3—C4	115.44 (13)	H11A—C11—H11C	109.5
C2—C3—C4	119.78 (13)	H11B—C11—H11C	109.5
C7—C5—C2	122.07 (13)	C3—O1—C1	117.08 (12)
C7—C5—H5	119.0	C10—O3—C11	115.40 (12)
C2—C5—H5	119.0	C8—N1—N2	115.82 (12)
C4—C6—C7	121.48 (13)	C10—N2—N1	118.55 (13)
C4—C6—H6	119.3	C10—N2—H2A	117.4 (12)
C7—C6—H6	119.3	N1—N2—H2A	122.0 (11)
C3—C2—C5	119.33 (13)	C8—C9—H9A	109.5
C3—C2—H2	120.3	C8—C9—H9B	109.5
C5—C2—H2	120.3	H9A—C9—H9B	109.5
C6—C4—C3	120.17 (13)	C8—C9—H9C	109.5
C6—C4—H4	119.9	H9A—C9—H9C	109.5
C3—C4—H4	119.9	H9B—C9—H9C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{i}	0.89 (2)	2.01 (2)	2.8864 (17)	169.0
C4—H4···*Cg*1^{ii}	0.93	2.87	3.6488 (19)	142

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