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Methyl *N*′-[(*E*)-1-phenylethylidene]-hydrazinecarboxylate

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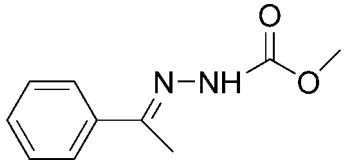
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.028; wR factor = 0.077; data-to-parameter ratio = 6.8.

The molecule of the title compound, $C_{10}H_{12}N_2O_2$, adopts a *trans* configuration with respect to the C=N bond. The dihedral angle between the phenyl ring and the hydrazine carboxylic acid mean plane is 25.23 (9)°. In the crystal structure, molecules are linked into chains by N—H···O hydrogen bonds and C—H···π interactions.

Related literature

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



Experimental

Crystal data

$C_{10}H_{12}N_2O_2$
M_r = 192.22
Orthorhombic, $Pca2_1$
a = 6.6733 (5) Å
b = 19.8940 (14) Å
c = 7.7254 (5) Å
V = 1025.61 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 123 (2) K
0.26 × 0.25 × 0.23 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.965, T_{max} = 0.968
10169 measured reflections
971 independent reflections
935 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.076
S = 1.14
971 reflections
143 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.09 e Å^{-3}

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

*Cg*1 is the centroid of the C1–C6 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H10···O1^{i}	0.84 (4)	2.16 (4)	2.977 (2)	167
C2—H2A···*Cg*1^{ii}	0.95	2.96	3.827 (2)	156
C5—H5···*Cg*1^{iii}	0.95	2.88	3.753 (2)	156

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

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: HB2751).

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**, o1384 [doi:10.1107/S1600536808019259]

Methyl *N*′-[(*E*)-1-phenylethylidene]hydrazinecarboxylate

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S1. Comment

As part of our ongoing 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 C9/C10/N2/O1/O2 plane of the hydrazine carboxylic acid methyl ester group is slightly twisted away from the attached ring. The dihedral angle between the C1—C6 ring and the C9/C10/N2/O1/O2 plane is 25.23 (9)°. Otherwise, the bond lengths and angles ij (I) agree with those observed for (*E*)-methyl *N*′-(4-hydroxybenzylidene) hydrazinecarboxylate (Cheng, 2008).

In the crystal structure, N–H···O hydrogen bonds and C–H···π interactions (Table 1) link the molecules into chains (Fig. 2).

S2. Experimental

Acetophenone (1.2 g, 0.01 mol) and methyl hydrazinecarboxylate (0.9 g, 0.01 mol) were dissolved in stirred methanol (20 ml) and left for 2 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. 450–452 K).

S3. Refinement

Anomalous dispersion was negligible and Friedel pairs were merged before refinement. The H atoms attached to C7 and N2 were located in a difference map and their positions and U_{iso} values were freely refined. The other H atoms were geometrically placed (C—H = 0.95-0.98Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C).

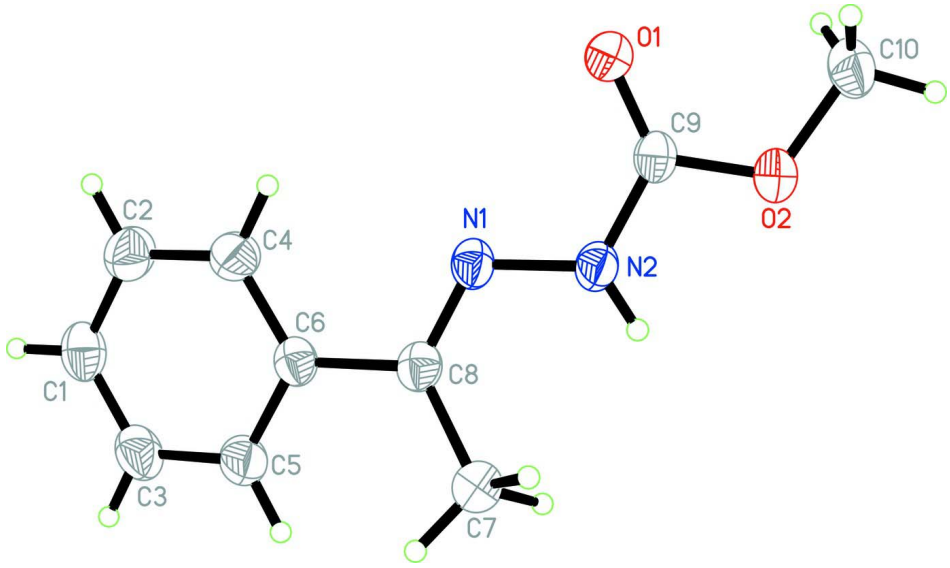

Figure 1

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

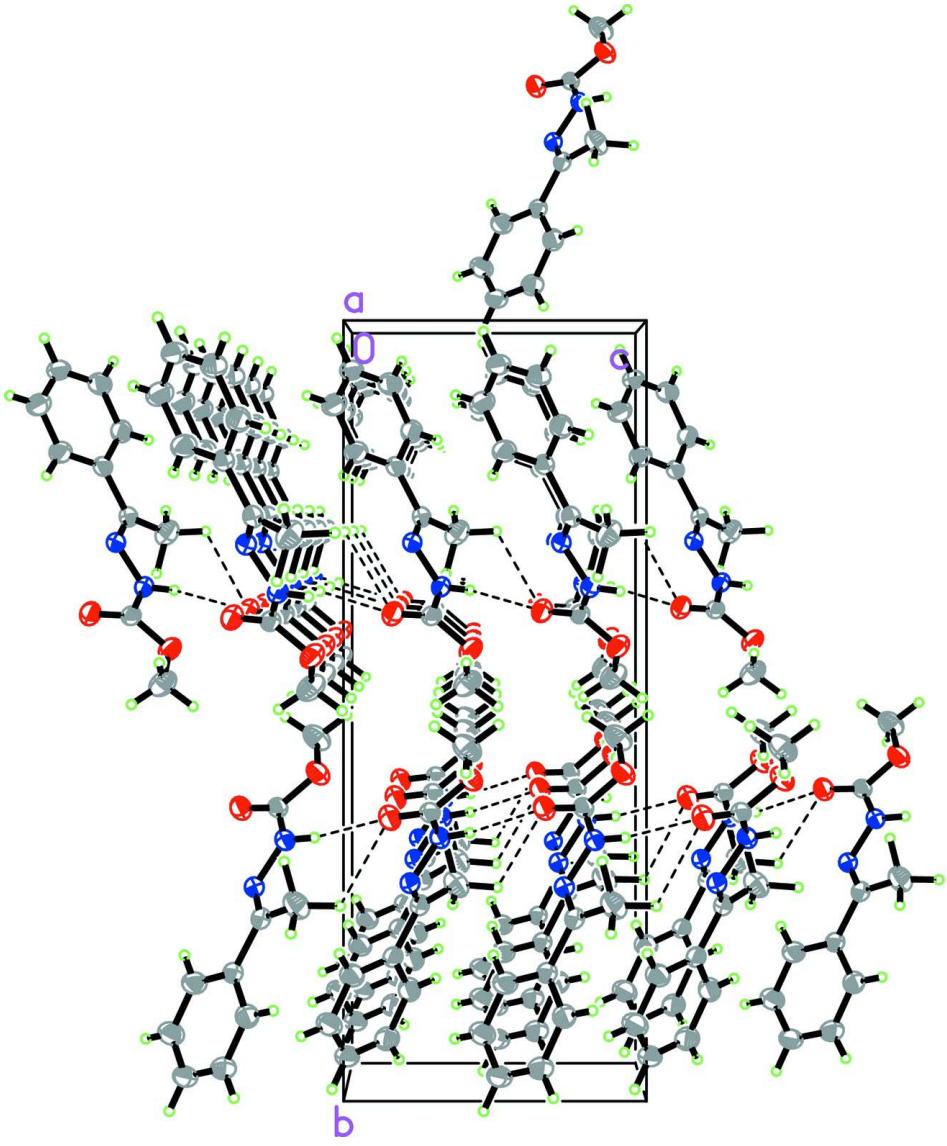

Figure 2

Crystal packing of (I), viewed approximately down the *a* axis. Dashed lines indicate hydrogen bonds.

Methyl *N*′-[(*E*)-1-phenylethylidene]hydrazinecarboxylate

Crystal data

$C_{10}H_{12}N_2O_2$
$M_r = 192.22$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 6.6733\ (5)$ Å
$b = 19.8940\ (14)$ Å
$c = 7.7254\ (5)$ Å
$V = 1025.61\ (12)$ Å^3
$Z = 4$

$F(000) = 408$
$D_x = 1.245$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 971 reflections
$\theta = 2.0$–$25.0°$
$\mu = 0.09$ mm^{-1}
$T = 123$ K
Block, colourless
0.26 × 0.25 × 0.23 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
10169 measured reflections
971 independent reflections
935 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°, θ_{min} = 2.1°
$h = -7 \rightarrow 7$
$k = -23 \rightarrow 21$
$l = -8 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.076
S = 1.14
971 reflections
143 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difmap and geom
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.0621P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.048
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.09 e Å^{-3}

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}
O2	0.8998 (2)	0.42251 (7)	0.4199 (2)	0.0606 (4)
O1	0.9765 (2)	0.37588 (7)	0.1622 (2)	0.0563 (4)
N2	0.7026 (3)	0.34290 (8)	0.3197 (2)	0.0491 (4)
N1	0.6763 (2)	0.28503 (7)	0.2224 (2)	0.0472 (4)
C6	0.5017 (3)	0.18464 (9)	0.1633 (3)	0.0467 (4)
C8	0.5153 (3)	0.25135 (9)	0.2518 (2)	0.0471 (4)
C9	0.8707 (3)	0.37937 (9)	0.2891 (3)	0.0448 (4)
C4	0.6331 (3)	0.16677 (11)	0.0313 (4)	0.0632 (6)
H4	0.7287	0.1986	−0.0087	0.076*
C5	0.3635 (3)	0.13717 (10)	0.2157 (3)	0.0610 (5)
H5	0.2700	0.1480	0.3041	0.073*
C7	0.3524 (4)	0.27326 (16)	0.3717 (4)	0.0703 (7)
C2	0.6270 (4)	0.10375 (11)	−0.0424 (4)	0.0688 (7)
H2A	0.7188	0.0926	−0.1320	0.083*
C1	0.4902 (4)	0.05692 (10)	0.0119 (4)	0.0634 (6)
H1	0.4865	0.0135	−0.0393	0.076*

C10	1.0751 (3)	0.46413 (12)	0.4084 (4)	0.0729 (7)
H10A	1.0816	0.4937	0.5096	0.109*
H10B	1.0684	0.4914	0.3029	0.109*
H10C	1.1949	0.4357	0.4048	0.109*
C3	0.3594 (4)	0.07359 (11)	0.1404 (4)	0.0665 (6)
H3	0.2642	0.0414	0.1792	0.080*
H10	0.642 (4)	0.3456 (12)	0.414 (5)	0.074 (8)*
H11	0.239 (7)	0.2525 (14)	0.336 (5)	0.100 (9)*
H12	0.347 (4)	0.3212 (16)	0.373 (5)	0.092 (9)*
H13	0.400 (8)	0.261 (2)	0.480 (9)	0.16 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0663 (8)	0.0588 (8)	0.0566 (9)	−0.0113 (7)	0.0026 (8)	−0.0173 (7)
O1	0.0578 (7)	0.0621 (9)	0.0488 (8)	−0.0081 (6)	0.0045 (7)	−0.0092 (7)
N2	0.0621 (10)	0.0459 (8)	0.0394 (9)	−0.0059 (7)	0.0030 (8)	−0.0046 (7)
N1	0.0612 (8)	0.0410 (7)	0.0393 (8)	−0.0045 (6)	−0.0020 (7)	−0.0006 (7)
C6	0.0555 (10)	0.0438 (9)	0.0408 (10)	−0.0039 (8)	−0.0054 (8)	0.0032 (8)
C8	0.0562 (10)	0.0469 (9)	0.0381 (10)	−0.0007 (8)	−0.0028 (8)	0.0023 (8)
C9	0.0507 (9)	0.0410 (9)	0.0428 (10)	0.0029 (7)	−0.0068 (8)	−0.0024 (8)
C4	0.0729 (13)	0.0512 (11)	0.0656 (14)	−0.0112 (10)	0.0152 (12)	−0.0035 (11)
C5	0.0684 (12)	0.0596 (11)	0.0549 (12)	−0.0151 (10)	0.0044 (10)	−0.0012 (11)
C7	0.0636 (13)	0.0744 (17)	0.0728 (18)	−0.0115 (12)	0.0119 (12)	−0.0210 (14)
C2	0.0804 (14)	0.0563 (12)	0.0698 (16)	−0.0010 (11)	0.0110 (13)	−0.0104 (12)
C1	0.0815 (14)	0.0429 (11)	0.0660 (14)	−0.0022 (10)	−0.0125 (12)	−0.0032 (10)
C10	0.0673 (13)	0.0730 (15)	0.0786 (16)	−0.0169 (11)	−0.0033 (13)	−0.0243 (14)
C3	0.0798 (13)	0.0547 (12)	0.0650 (15)	−0.0209 (10)	−0.0032 (12)	0.0034 (11)

Geometric parameters (Å, º)

O2—C9	1.340 (2)	C5—C3	1.392 (3)
O2—C10	1.436 (2)	C5—H5	0.9500
O1—C9	1.211 (3)	C7—H11	0.91 (4)
N2—C9	1.357 (2)	C7—H12	0.95 (3)
N2—N1	1.386 (2)	C7—H13	0.93 (6)
N2—H10	0.84 (3)	C2—C1	1.370 (3)
N1—C8	1.286 (2)	C2—H2A	0.9500
C6—C5	1.381 (3)	C1—C3	1.363 (4)
C6—C4	1.391 (3)	C1—H1	0.9500
C6—C8	1.496 (3)	C10—H10A	0.9800
C8—C7	1.494 (3)	C10—H10B	0.9800
C4—C2	1.378 (3)	C10—H10C	0.9800
C4—H4	0.9500	C3—H3	0.9500
C9—O2—C10	116.16 (17)	C8—C7—H12	109 (2)
C9—N2—N1	117.04 (17)	H11—C7—H12	115 (3)
C9—N2—H10	121.1 (18)	C8—C7—H13	103 (3)

N1—N2—H10	117.8 (18)	H11—C7—H13	116 (4)
C8—N1—N2	116.30 (16)	H12—C7—H13	106 (4)
C5—C6—C4	117.5 (2)	C1—C2—C4	120.8 (2)
C5—C6—C8	120.90 (19)	C1—C2—H2A	119.6
C4—C6—C8	121.57 (17)	C4—C2—H2A	119.6
N1—C8—C7	124.42 (19)	C3—C1—C2	119.0 (2)
N1—C8—C6	115.63 (16)	C3—C1—H1	120.5
C7—C8—C6	119.88 (18)	C2—C1—H1	120.5
O1—C9—O2	124.28 (17)	O2—C10—H10A	109.5
O1—C9—N2	126.33 (18)	O2—C10—H10B	109.5
O2—C9—N2	109.35 (17)	H10A—C10—H10B	109.5
C2—C4—C6	121.1 (2)	O2—C10—H10C	109.5
C2—C4—H4	119.4	H10A—C10—H10C	109.5
C6—C4—H4	119.4	H10B—C10—H10C	109.5
C6—C5—C3	120.8 (2)	C1—C3—C5	120.8 (2)
C6—C5—H5	119.6	C1—C3—H3	119.6
C3—C5—H5	119.6	C5—C3—H3	119.6
C8—C7—H11	107 (2)		

C9—N2—N1—C8	−179.38 (17)	N1—N2—C9—O2	−164.20 (15)
N2—N1—C8—C7	4.7 (3)	C5—C6—C4—C2	−0.9 (3)
N2—N1—C8—C6	−172.29 (15)	C8—C6—C4—C2	176.1 (2)
C5—C6—C8—N1	164.0 (2)	C4—C6—C5—C3	1.1 (3)
C4—C6—C8—N1	−12.9 (3)	C8—C6—C5—C3	−175.9 (2)
C5—C6—C8—C7	−13.1 (3)	C6—C4—C2—C1	0.3 (4)
C4—C6—C8—C7	170.0 (3)	C4—C2—C1—C3	0.0 (4)
C10—O2—C9—O1	−3.3 (3)	C2—C1—C3—C5	0.2 (4)
C10—O2—C9—N2	178.85 (18)	C6—C5—C3—C1	−0.8 (4)
N1—N2—C9—O1	18.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H10···O1^{i}	0.84 (4)	2.16 (4)	2.977 (2)	167
C2—H2*A*···*Cg*1^{ii}	0.95	2.96	3.827 (2)	156
C5—H5···*Cg*1^{iii}	0.95	2.88	3.753 (2)	156

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