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(*E*)-Methyl *N*′-(3,4,5-trimethoxybenzylidene)hydrazinecarboxylate

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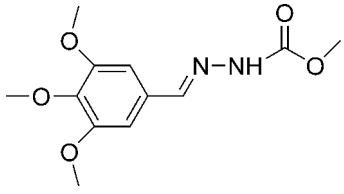
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.133; data-to-parameter ratio = 13.5.

The molecule of the title compound, $C_{12}H_{16}N_2O_5$, adopts a *trans* configuration with respect to the C=N double bond. The dihedral angle between the benzene and hydrazinecarboxylic acid methyl ester planes is 12.55 (7)°. The molecules are linked into a chain along [001] by intermolecular N—H···O hydrogen bonds, and the chains are cross-linked into a two-dimensional zigzag structure by C—H···O hydrogen bonds.

Related literature

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



Experimental

Crystal data

$C_{12}H_{16}N_2O_5$
M_r = 268.27
Monoclinic, $P2_1/c$
a = 8.554 (3) Å
b = 22.705 (7) Å
c = 7.813 (2) Å
β = 116.15 (1)°
V = 1362.1 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 (2) K
0.27 × 0.25 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.965, T_{max} = 0.968
7173 measured reflections
2394 independent reflections
1671 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.133
S = 1.03
2394 reflections
177 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 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
N2—H2···O4^{i}	0.86	2.16	3.000 (2)	166
C2—H2B···O2^{ii}	0.96	2.57	3.498 (3)	161

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

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: CI2687).

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supporting information

Acta Cryst. (2008). E**64**, o2063 [doi:10.1107/S1600536808031590]

(*E*)-Methyl *N*′-(3,4,5-trimethoxybenzylidene)hydrazinecarboxylate

Lu-Ping Lv, Jian-Wu Xie, Wen-Bo Yu, Wei-Wei Li 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). They are important intermidiates of 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 bond. The hydrazine carboxylic acid methyl ester group is slightly twisted away from the attached ring. The dihedral angle between the benzene ring and the C10/C11//N1/N2/O4/O5 plane [r.m.s. deviation 0.051 Å] is 12.55 (7)°. The O1-C1 and O3-C3 methoxy groups are coplanar with the benzene ring [C8—C4—O1—C1 = -1.7 (3)° and C7—C6—O3—C3 = -1.9 (3)°] while the O2-C2 group is twisted almost perpendicular to the attached ring [C6—C5—O2—C2 = 91.6 (2)°]. The bond lengths and angles agree with those observed for N′-(4-methoxybenzylidene)methoxyformohydrazide (Shang *et al.*, 2007).

The molecules are linked into a chain along the [001] by intermolecular N–H···O hydrogen bonds (Fig.2 and Table 1). The chains are cross-linked into a two-dimensional zigzag structure by C—H···O hydrogen bonds.

S2. Experimental

3,4,5-Trimethoxybenzaldehyde (1.96g, 0.01mol) and methyl hydrazinecarboxylate (0.9 g, 0.01 mol) were dissolved in stirred methanol (15 ml) and left for 3.2 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 94% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 472-474 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}). A rotating group model was used for the methy groups.

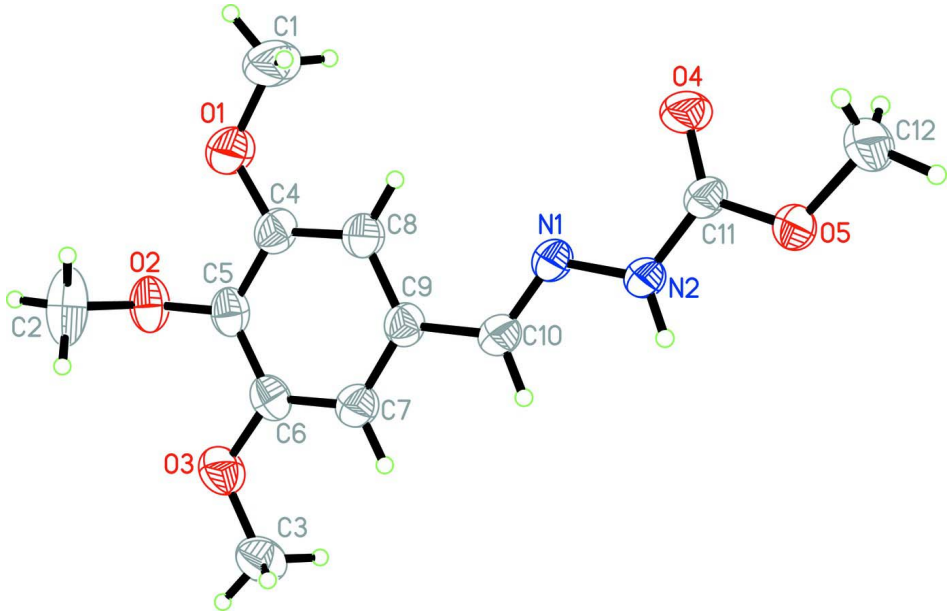

Figure 1

The molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atomic numbering.

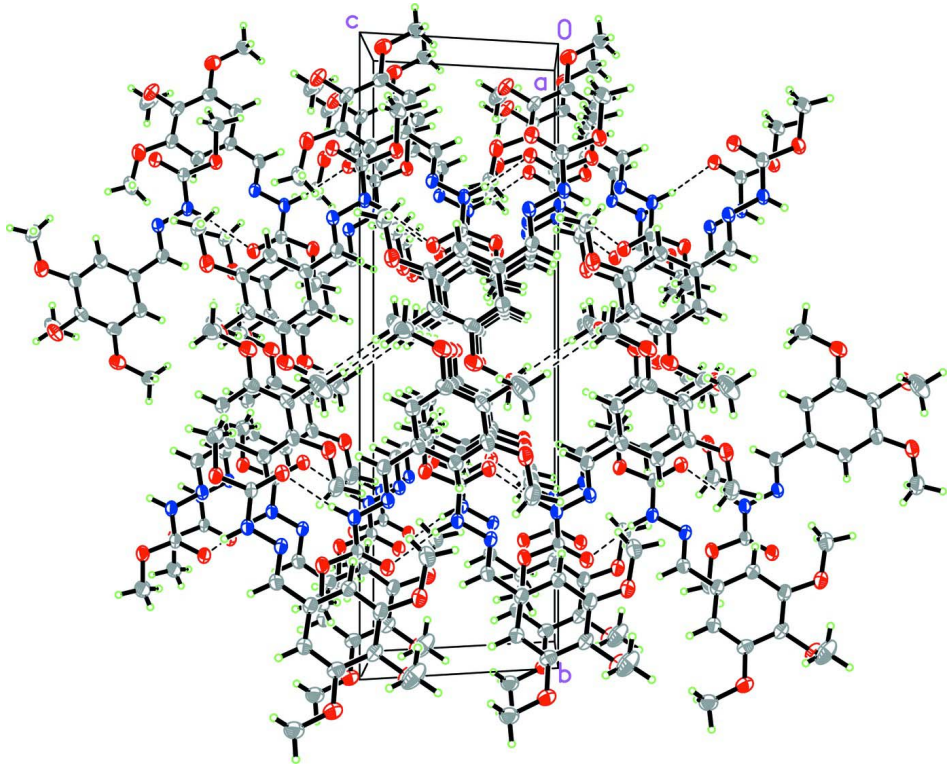

Figure 2

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

(*E*)-Methyl *N*′-(3,4,5-trimethoxybenzylidene)hydrazinecarboxylate

Crystal data

$C_{12}H_{16}N_2O_5$
$M_r = 268.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.554$ (3) Å
$b = 22.705$ (7) Å
$c = 7.813$ (2) Å
$\beta = 116.15$ (1)°
$V = 1362.1$ (7) Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.308$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2394 reflections
$\theta = 1.8$–25.0°
$\mu = 0.10$ mm^{-1}
$T = 273$ K
Block, colourless
0.27 × 0.25 × 0.24 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)
$T_{min} = 0.965$, $T_{max} = 0.968$
7173 measured reflections
2394 independent reflections
1671 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.8°$
$h = -10 \rightarrow 10$
$k = -27 \rightarrow 26$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.133$
$S = 1.04$
2394 reflections
177 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
$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (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}
C11	0.1250 (2)	0.29913 (8)	0.4985 (2)	0.0442 (5)
C7	0.5205 (3)	0.07771 (8)	0.7843 (3)	0.0522 (5)
H7	0.4723	0.0607	0.6636	0.063*

C10	0.3534 (3)	0.16740 (8)	0.6404 (3)	0.0493 (5)
H10	0.3171	0.1508	0.5201	0.059*
C9	0.4743 (2)	0.13484 (8)	0.8080 (2)	0.0467 (5)
C6	0.6382 (3)	0.04596 (8)	0.9394 (3)	0.0508 (5)
C8	0.5451 (3)	0.16011 (9)	0.9895 (2)	0.0529 (5)
H8	0.5130	0.1980	1.0067	0.063*
C4	0.6637 (3)	0.12845 (9)	1.1436 (3)	0.0526 (5)
C5	0.7116 (2)	0.07116 (8)	1.1194 (3)	0.0499 (5)
C12	−0.0302 (4)	0.37985 (10)	0.3123 (3)	0.0850 (8)
H12A	−0.0984	0.3825	0.3820	0.127*
H12B	−0.1010	0.3900	0.1813	0.127*
H12C	0.0666	0.4065	0.3662	0.127*
C3	0.6171 (3)	−0.03833 (9)	0.7485 (3)	0.0677 (6)
H3A	0.4927	−0.0390	0.7001	0.102*
H3B	0.6600	−0.0779	0.7609	0.102*
H3C	0.6482	−0.0169	0.6620	0.102*
C1	0.7014 (4)	0.20690 (11)	1.3617 (3)	0.0986 (10)
H1A	0.7253	0.2340	1.2818	0.148*
H1B	0.7713	0.2169	1.4931	0.148*
H1C	0.5804	0.2092	1.3335	0.148*
C2	1.0022 (3)	0.04729 (13)	1.3133 (4)	0.0950 (9)
H2A	1.0190	0.0332	1.2067	0.143*
H2B	1.0752	0.0254	1.4253	0.143*
H2C	1.0323	0.0883	1.3335	0.143*
O5	0.03345 (18)	0.32059 (5)	0.32345 (16)	0.0574 (4)
O4	0.1466 (2)	0.32391 (5)	0.64414 (17)	0.0632 (5)
O2	0.82545 (17)	0.03987 (6)	1.27554 (19)	0.0608 (4)
O3	0.6917 (2)	−0.01046 (6)	0.9293 (2)	0.0670 (5)
O1	0.7416 (2)	0.14863 (6)	1.32715 (18)	0.0745 (5)
N1	0.29649 (19)	0.21810 (6)	0.65536 (19)	0.0442 (4)
N2	0.1871 (2)	0.24529 (6)	0.4883 (2)	0.0492 (4)
H2	0.1588	0.2285	0.3801	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0513 (12)	0.0478 (10)	0.0312 (9)	0.0013 (9)	0.0160 (8)	−0.0003 (8)
C7	0.0572 (13)	0.0525 (11)	0.0440 (11)	0.0038 (9)	0.0198 (10)	0.0014 (8)
C10	0.0541 (13)	0.0511 (11)	0.0382 (10)	0.0028 (9)	0.0162 (9)	−0.0023 (8)
C9	0.0474 (11)	0.0509 (11)	0.0403 (10)	0.0040 (8)	0.0180 (9)	0.0054 (8)
C6	0.0510 (12)	0.0468 (11)	0.0576 (12)	0.0075 (9)	0.0268 (10)	0.0085 (9)
C8	0.0588 (13)	0.0498 (10)	0.0445 (11)	0.0071 (10)	0.0178 (10)	0.0048 (8)
C4	0.0564 (13)	0.0591 (12)	0.0382 (10)	0.0032 (10)	0.0171 (9)	0.0068 (8)
C5	0.0466 (11)	0.0554 (11)	0.0478 (11)	0.0073 (9)	0.0211 (9)	0.0165 (9)
C12	0.118 (2)	0.0590 (14)	0.0569 (14)	0.0348 (14)	0.0188 (14)	0.0078 (10)
C3	0.0801 (17)	0.0546 (12)	0.0745 (16)	0.0101 (11)	0.0395 (13)	0.0015 (10)
C1	0.134 (3)	0.0820 (17)	0.0484 (13)	0.0263 (17)	0.0111 (15)	−0.0086 (11)
C2	0.0506 (16)	0.118 (2)	0.101 (2)	0.0070 (14)	0.0191 (14)	0.0537 (17)

O5	0.0733 (10)	0.0556 (8)	0.0359 (7)	0.0213 (7)	0.0174 (7)	0.0050 (5)
O4	0.0951 (12)	0.0523 (8)	0.0379 (8)	0.0124 (7)	0.0253 (7)	−0.0012 (6)
O2	0.0523 (9)	0.0694 (9)	0.0573 (9)	0.0100 (7)	0.0211 (7)	0.0266 (7)
O3	0.0761 (10)	0.0544 (9)	0.0661 (10)	0.0191 (7)	0.0275 (8)	0.0098 (7)
O1	0.0913 (13)	0.0709 (10)	0.0415 (8)	0.0192 (8)	0.0111 (8)	0.0042 (7)
N1	0.0497 (10)	0.0482 (9)	0.0317 (8)	0.0036 (7)	0.0152 (7)	0.0034 (6)
N2	0.0616 (11)	0.0501 (9)	0.0296 (7)	0.0133 (8)	0.0145 (7)	0.0004 (6)

Geometric parameters (Å, º)

C11—O4	1.209 (2)	C12—H12A	0.96
C11—O5	1.333 (2)	C12—H12B	0.96
C11—N2	1.349 (2)	C12—H12C	0.96
C7—C6	1.388 (2)	C3—O3	1.417 (2)
C7—C9	1.392 (3)	C3—H3A	0.96
C7—H7	0.93	C3—H3B	0.96
C10—N1	1.275 (2)	C3—H3C	0.96
C10—C9	1.462 (2)	C1—O1	1.422 (3)
C10—H10	0.93	C1—H1A	0.96
C9—C8	1.396 (2)	C1—H1B	0.96
C6—O3	1.374 (2)	C1—H1C	0.96
C6—C5	1.386 (3)	C2—O2	1.417 (3)
C8—C4	1.386 (2)	C2—H2A	0.96
C8—H8	0.93	C2—H2B	0.96
C4—O1	1.367 (2)	C2—H2C	0.96
C4—C5	1.401 (3)	N1—N2	1.3723 (19)
C5—O2	1.376 (2)	N2—H2	0.86
C12—O5	1.439 (2)		
O4—C11—O5	124.93 (17)	H12A—C12—H12C	109.5
O4—C11—N2	125.21 (16)	H12B—C12—H12C	109.5
O5—C11—N2	109.85 (14)	O3—C3—H3A	109.5
C6—C7—C9	120.45 (17)	O3—C3—H3B	109.5
C6—C7—H7	119.8	H3A—C3—H3B	109.5
C9—C7—H7	119.8	O3—C3—H3C	109.5
N1—C10—C9	121.47 (17)	H3A—C3—H3C	109.5
N1—C10—H10	119.3	H3B—C3—H3C	109.5
C9—C10—H10	119.3	O1—C1—H1A	109.5
C7—C9—C8	119.77 (17)	O1—C1—H1B	109.5
C7—C9—C10	118.81 (16)	H1A—C1—H1B	109.5
C8—C9—C10	121.41 (17)	O1—C1—H1C	109.5
O3—C6—C5	115.43 (16)	H1A—C1—H1C	109.5
O3—C6—C7	124.47 (17)	H1B—C1—H1C	109.5
C5—C6—C7	120.11 (17)	O2—C2—H2A	109.5
C4—C8—C9	119.57 (18)	O2—C2—H2B	109.5
C4—C8—H8	120.2	H2A—C2—H2B	109.5
C9—C8—H8	120.2	O2—C2—H2C	109.5
O1—C4—C8	124.72 (18)	H2A—C2—H2C	109.5

O1—C4—C5	114.63 (16)	H2B—C2—H2C	109.5
C8—C4—C5	120.65 (17)	C11—O5—C12	116.06 (14)
O2—C5—C6	120.91 (17)	C5—O2—C2	113.36 (15)
O2—C5—C4	119.61 (17)	C6—O3—C3	117.27 (15)
C6—C5—C4	119.44 (16)	C4—O1—C1	117.67 (16)
O5—C12—H12A	109.5	C10—N1—N2	116.53 (14)
O5—C12—H12B	109.5	C11—N2—N1	118.23 (14)
H12A—C12—H12B	109.5	C11—N2—H2	120.9
O5—C12—H12C	109.5	N1—N2—H2	120.9

C6—C7—C9—C8	−0.7 (3)	C8—C4—C5—O2	−177.98 (18)
C6—C7—C9—C10	178.45 (18)	O1—C4—C5—C6	178.96 (17)
N1—C10—C9—C7	174.74 (18)	C8—C4—C5—C6	−0.5 (3)
N1—C10—C9—C8	−6.1 (3)	O4—C11—O5—C12	5.6 (3)
C9—C7—C6—O3	−179.79 (18)	N2—C11—O5—C12	−175.51 (18)
C9—C7—C6—C5	−0.5 (3)	C6—C5—O2—C2	91.6 (2)
C7—C9—C8—C4	1.3 (3)	C4—C5—O2—C2	−91.0 (2)
C10—C9—C8—C4	−177.86 (18)	C5—C6—O3—C3	178.74 (18)
C9—C8—C4—O1	179.89 (18)	C7—C6—O3—C3	−1.9 (3)
C9—C8—C4—C5	−0.7 (3)	C8—C4—O1—C1	−1.7 (3)
O3—C6—C5—O2	−2.1 (3)	C5—C4—O1—C1	178.9 (2)
C7—C6—C5—O2	178.52 (17)	C9—C10—N1—N2	178.42 (17)
O3—C6—C5—C4	−179.53 (17)	O4—C11—N2—N1	−6.2 (3)
C7—C6—C5—C4	1.1 (3)	O5—C11—N2—N1	174.90 (15)
O1—C4—C5—O2	1.5 (3)	C10—N1—N2—C11	−179.66 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4^{i}	0.86	2.16	3.000 (2)	166
C2—H2*B*···O2^{ii}	0.96	2.57	3.498 (3)	161

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+2, −*y*, −*z*+3.