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(*E*)-Ethyl *N*′-(3,4-dimethoxybenzylidene)-hydrazinecarboxylate monohydrate

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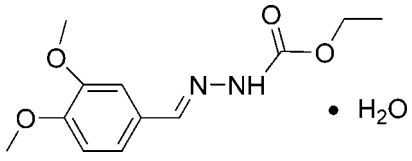
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 7.3.

In the title compound, $C_{12}H_{16}N_2O_4\cdot H_2O$, the molecular skeleton of the hydrazinecarboxylate is nearly planar [within 0.053 (3) Å]. In the crystal, chains propagating along the *c* axis arise, composed of alternating hydrazinecarboxylate molecules and crystalline water, which interact *via* N—H···O and O—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

$C_{12}H_{16}N_2O_4\cdot H_2O$
M_r = 270.28
Orthorhombic, $Pna2_1$
a = 7.211 (2) Å
b = 17.688 (5) Å
c = 11.026 (3) Å
V = 1406.3 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 193 (2) K
0.27 × 0.25 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.969, T_{max} = 0.976
7281 measured reflections
1312 independent reflections
1181 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.07
1312 reflections
180 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1F···O3	0.833 (19)	2.14 (2)	2.899 (3)	151 (3)
N2—H2···O1W^{i}	0.88	2.12	2.899 (3)	148

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

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: CV2464).

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supplementary materials

Acta Cryst. (2008). E**64**, o2196 [**doi:10.1107/S1600536808034417**]

(*E*)-Ethyl *N*'-(3,4-dimethoxybenzylidene)hydrazinecarboxylate monohydrate

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Comment

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

In the title compound, $C_{12}H_{16}N_2O_4$ (I) .H_2O, the molecular skeleton of (I) is nearly planar. The main molecule, (I), 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 bond lengths and angles of the main molecule agree with those observed in (E)-methyl N'-(4-hydroxybenzylidene)hydrazinecarboxylate (Shang *et al.*, 2007).

The crystal packing exhibits hydrogen-bonded chains extended along c axis and composed from alternating molecules of (I) and crystalline water, which interact via N-H···O [N···O 2.899 (3) Å] and O-H···O [O···O 2.899 (3) Å] hydrogen bonds (Table 1).

Experimental

3,4-Dimethoxybenzaldehyde (1.66g, 0.01mol) and ethyl hydrazinecarboxylate(1.04g, 0.01mol) were dissolved in stirred methanol (20ml) and left for 3h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 90% yield. Crystals suitable for X-ray analysis were obtained by slow evaporation of a ethanol solution at room temperature (m.p. 458-460 K).

Refinement

H atoms of the water molecule were located in a difference map and were refined with O-H distances restrained to 0.82 (2) Å, H atoms were included in the riding model approximation with N-H = 0.88 Å. C-bound H atoms were positioned geometrically (C-H = 0.95-0.99 Å) and refined using a riding model, with $U_{iso}(H) = 1.2\text{-}1.5U_{eq}(C)$. In the absence of significant anomalous scatterers, the 1193 Friedel pairs were merged before the final refinement.

Figures

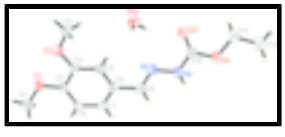

Fig. 1. Molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

(E)-Ethyl N'-(3,4-dimethoxybenzylidene)hydrazinecarboxylate monohydrate

Crystal data

$C_{12}H_{16}N_2O_4 \cdot H_2O$	$F_{000} = 576$
$M_r = 270.28$	$D_x = 1.277$ Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 1312 reflections
$a = 7.211$ (2) Å	$\theta = 2.2$–25.5º
$b = 17.688$ (5) Å	$\mu = 0.10$ mm^{-1}
$c = 11.026$ (3) Å	$T = 193$ (2) K
$V = 1406.3$ (7) Å^3	Block, colourless
$Z = 4$	0.27 × 0.25 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1312 independent reflections
Radiation source: fine-focus sealed tube	1181 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
$T = 193$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.2$º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$h = -8 \rightarrow 8$
$T_{min} = 0.969$, $T_{max} = 0.976$	$k = -20 \rightarrow 20$
7281 measured reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.0753P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1312 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
180 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
C1	1.0427 (4)	0.67958 (16)	0.2893 (3)	0.0656 (7)
H1A	1.1372	0.7154	0.3172	0.098*
H1B	0.9915	0.6969	0.2117	0.098*
H1C	1.0989	0.6296	0.2786	0.098*
C2	0.4765 (4)	0.69467 (15)	0.6084 (3)	0.0610 (6)
H2A	0.5117	0.7319	0.6701	0.091*
H2B	0.4388	0.6476	0.6482	0.091*
H2C	0.3730	0.7144	0.5603	0.091*
C3	0.6065 (3)	0.62962 (12)	0.4381 (2)	0.0458 (5)
C4	0.7529 (3)	0.62623 (13)	0.3535 (2)	0.0458 (6)
C5	0.4527 (3)	0.58339 (12)	0.4241 (2)	0.0462 (5)
H5	0.3536	0.5859	0.4806	0.055*
C6	0.7444 (3)	0.57723 (14)	0.2568 (2)	0.0514 (6)
H6	0.8428	0.5753	0.1995	0.062*
C7	0.5889 (3)	0.53002 (14)	0.2433 (2)	0.0519 (6)
H7	0.5833	0.4959	0.1769	0.062*
C8	0.4438 (3)	0.53255 (13)	0.3255 (2)	0.0469 (5)
C9	0.2842 (3)	0.48227 (14)	0.3094 (3)	0.0513 (6)
H9	0.2729	0.4534	0.2371	0.062*
C10	−0.1176 (4)	0.42102 (13)	0.4535 (2)	0.0511 (6)
C11	−0.4016 (4)	0.36234 (19)	0.5028 (3)	0.0719 (8)
H11A	−0.4699	0.4105	0.5130	0.086*
H11B	−0.3517	0.3471	0.5828	0.086*
C12	−0.5286 (5)	0.3030 (2)	0.4560 (4)	0.0882 (11)
H12A	−0.6307	0.2956	0.5134	0.132*
H12B	−0.4602	0.2555	0.4463	0.132*
H12C	−0.5786	0.3188	0.3773	0.132*
N1	0.1595 (3)	0.47690 (10)	0.3921 (2)	0.0520 (5)
N2	0.0153 (3)	0.42736 (11)	0.3681 (2)	0.0525 (5)
H2	0.0107	0.4012	0.3002	0.063*
O1	0.8976 (2)	0.67502 (9)	0.37704 (18)	0.0578 (5)
O2	0.6305 (2)	0.68036 (11)	0.53107 (17)	0.0620 (5)
O3	−0.1179 (3)	0.45274 (11)	0.55163 (18)	0.0689 (5)

O4	−0.2508 (2)	0.37243 (9)	0.41722 (18)	0.0582 (5)
O1W	0.0397 (3)	0.59890 (12)	0.6111 (2)	0.0667 (5)
H1E	0.003 (5)	0.6288 (18)	0.561 (3)	0.090 (12)*
H1F	0.030 (5)	0.5563 (13)	0.579 (3)	0.088 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0528 (15)	0.0662 (16)	0.0777 (18)	−0.0115 (12)	0.0121 (14)	−0.0002 (15)
C2	0.0693 (16)	0.0566 (14)	0.0570 (15)	−0.0026 (12)	0.0121 (15)	−0.0057 (13)
C3	0.0457 (12)	0.0442 (11)	0.0474 (12)	0.0005 (8)	−0.0055 (11)	−0.0020 (10)
C4	0.0389 (12)	0.0447 (10)	0.0537 (15)	0.0004 (9)	−0.0033 (10)	0.0030 (11)
C5	0.0414 (11)	0.0506 (12)	0.0466 (13)	−0.0001 (9)	−0.0005 (10)	0.0029 (10)
C6	0.0456 (12)	0.0538 (12)	0.0548 (15)	0.0026 (10)	0.0029 (11)	0.0000 (12)
C7	0.0536 (13)	0.0510 (12)	0.0513 (13)	0.0004 (10)	−0.0048 (12)	−0.0061 (11)
C8	0.0430 (12)	0.0462 (11)	0.0515 (13)	0.0010 (9)	−0.0065 (11)	0.0051 (10)
C9	0.0512 (12)	0.0483 (12)	0.0543 (14)	−0.0020 (10)	−0.0075 (12)	−0.0013 (11)
C10	0.0553 (14)	0.0453 (11)	0.0526 (14)	−0.0034 (10)	−0.0067 (12)	0.0022 (11)
C11	0.0618 (17)	0.082 (2)	0.0724 (19)	−0.0143 (14)	0.0081 (15)	0.0022 (15)
C12	0.0713 (19)	0.097 (2)	0.096 (3)	−0.0260 (18)	0.004 (2)	0.003 (2)
N1	0.0493 (11)	0.0485 (10)	0.0582 (13)	−0.0066 (8)	−0.0074 (11)	0.0029 (9)
N2	0.0500 (11)	0.0521 (11)	0.0554 (13)	−0.0116 (8)	−0.0024 (10)	−0.0037 (9)
O1	0.0451 (9)	0.0621 (10)	0.0661 (11)	−0.0105 (7)	0.0053 (9)	−0.0080 (9)
O2	0.0529 (10)	0.0723 (10)	0.0607 (12)	−0.0111 (8)	0.0041 (9)	−0.0181 (10)
O3	0.0744 (12)	0.0770 (11)	0.0553 (11)	−0.0204 (10)	0.0011 (10)	−0.0043 (10)
O4	0.0546 (10)	0.0570 (9)	0.0628 (12)	−0.0138 (7)	0.0022 (8)	−0.0010 (9)
O1W	0.0778 (13)	0.0662 (12)	0.0561 (12)	−0.0123 (10)	−0.0078 (11)	0.0050 (11)

Geometric parameters (Å, °)

C1—O1	1.427 (3)	C7—H7	0.9500
C1—H1A	0.9800	C8—C9	1.465 (3)
C1—H1B	0.9800	C9—N1	1.284 (3)
C1—H1C	0.9800	C9—H9	0.9500
C2—O2	1.423 (3)	C10—O3	1.219 (3)
C2—H2A	0.9800	C10—N2	1.348 (3)
C2—H2B	0.9800	C10—O4	1.350 (3)
C2—H2C	0.9800	C11—O4	1.451 (4)
C3—O2	1.374 (3)	C11—C12	1.485 (5)
C3—C5	1.387 (3)	C11—H11A	0.9900
C3—C4	1.410 (3)	C11—H11B	0.9900
C4—C6	1.376 (4)	C12—H12A	0.9800
C4—O1	1.379 (3)	C12—H12B	0.9800
C5—C8	1.412 (4)	C12—H12C	0.9800
C5—H5	0.9500	N1—N2	1.385 (3)
C6—C7	1.406 (4)	N2—H2	0.8800
C6—H6	0.9500	O1W—H1E	0.812 (19)
C7—C8	1.385 (3)	O1W—H1F	0.833 (19)

O1—C1—H1A	109.5	C7—C8—C9	119.6 (2)
O1—C1—H1B	109.5	C5—C8—C9	121.0 (2)
H1A—C1—H1B	109.5	N1—C9—C8	120.6 (2)
O1—C1—H1C	109.5	N1—C9—H9	119.7
H1A—C1—H1C	109.5	C8—C9—H9	119.7
H1B—C1—H1C	109.5	O3—C10—N2	125.7 (2)
O2—C2—H2A	109.5	O3—C10—O4	123.7 (2)
O2—C2—H2B	109.5	N2—C10—O4	110.6 (2)
H2A—C2—H2B	109.5	O4—C11—C12	108.8 (3)
O2—C2—H2C	109.5	O4—C11—H11A	109.9
H2A—C2—H2C	109.5	C12—C11—H11A	109.9
H2B—C2—H2C	109.5	O4—C11—H11B	109.9
O2—C3—C5	124.7 (2)	C12—C11—H11B	109.9
O2—C3—C4	115.30 (19)	H11A—C11—H11B	108.3
C5—C3—C4	120.0 (2)	C11—C12—H12A	109.5
C6—C4—O1	125.0 (2)	C11—C12—H12B	109.5
C6—C4—C3	120.4 (2)	H12A—C12—H12B	109.5
O1—C4—C3	114.5 (2)	C11—C12—H12C	109.5
C3—C5—C8	119.8 (2)	H12A—C12—H12C	109.5
C3—C5—H5	120.1	H12B—C12—H12C	109.5
C8—C5—H5	120.1	C9—N1—N2	115.9 (2)
C4—C6—C7	119.5 (2)	C10—N2—N1	116.9 (2)
C4—C6—H6	120.3	C10—N2—H2	121.5
C7—C6—H6	120.3	N1—N2—H2	121.5
C8—C7—C6	120.9 (2)	C4—O1—C1	117.5 (2)
C8—C7—H7	119.5	C3—O2—C2	117.7 (2)
C6—C7—H7	119.5	C10—O4—C11	114.8 (2)
C7—C8—C5	119.3 (2)	H1E—O1W—H1F	106 (4)
O2—C3—C4—C6	179.8 (2)	C7—C8—C9—N1	171.3 (2)
C5—C3—C4—C6	0.0 (3)	C5—C8—C9—N1	−8.3 (3)
O2—C3—C4—O1	−0.4 (3)	C8—C9—N1—N2	−179.5 (2)
C5—C3—C4—O1	179.8 (2)	O3—C10—N2—N1	−3.1 (4)
O2—C3—C5—C8	−179.2 (2)	O4—C10—N2—N1	178.61 (19)
C4—C3—C5—C8	0.5 (3)	C9—N1—N2—C10	−179.8 (2)
O1—C4—C6—C7	179.7 (2)	C6—C4—O1—C1	5.4 (3)
C3—C4—C6—C7	−0.5 (3)	C3—C4—O1—C1	−174.4 (2)
C4—C6—C7—C8	0.5 (4)	C5—C3—O2—C2	−9.6 (3)
C6—C7—C8—C5	0.0 (4)	C4—C3—O2—C2	170.6 (2)
C6—C7—C8—C9	−179.6 (2)	O3—C10—O4—C11	2.3 (3)
C3—C5—C8—C7	−0.5 (3)	N2—C10—O4—C11	−179.4 (2)
C3—C5—C8—C9	179.1 (2)	C12—C11—O4—C10	−175.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1F···O3	0.833 (19)	2.14 (2)	2.899 (3)	151 (3)
O1W—H1E···O1^{i}	0.812 (19)	2.31 (2)	3.086 (3)	160 (4)
N2—H2···O1W^{ii}	0.88	2.12	2.899 (3)	148

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

Fig. 1