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Ethyl 3-(3-ethoxy-2-hydroxybenzylidene)carbazate

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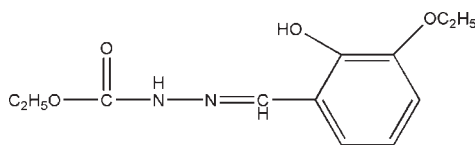
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.090; data-to-parameter ratio = 17.9.

In the title compound, $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_4$, an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond occurs. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains propagating in the [010] direction.

Related literature

For background to Schiff bases, see: Cimerman *et al.* (1997).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_4$ $M_r = 252.27$ Orthorhombic, $P2_12_12_1$ $a = 7.1140$ (14) Å $b = 9.6010$ (19) Å $c = 18.570$ (4) Å $V = 1268.4$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.10$ mm⁻¹ $T = 293$ K $0.22 \times 0.20 \times 0.19$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
11976 measured reflections

2917 independent reflections
2680 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$ $wR(F^2) = 0.090$ $S = 1.07$

2917 reflections

163 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4A}\cdots\text{N2}$	0.82	1.91	2.6290 (15)	145
$\text{N1}-\text{H1A}\cdots\text{O2}^i$	0.86	2.31	2.9633 (15)	132

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; 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: HB5155).

References

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Cimerman, Z., Galic, N. & Bosner, B. (1997). *Anal. Chim. Acta*, **343**, 145–153.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

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Ethyl 3-(3-ethoxy-2-hydroxybenzylidene)carbazate

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S1. Experimental

A mixture of 3-ethoxy-2-hydroxybenzaldehyde (0.1 mol), and ethyl carbazate (0.1 mol) was stirred in refluxing ethanol (20 ml) for 4 h to afford the title compound (0.082 mol, yield 82%). Colourless blocks of (I) were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

Refinement of the Flack absolute structure parameter was indeterminate.

H atoms were fixed geometrically (C—H = 0.93–0.97 Å, O—H = 0.82 Å, N—H = 0.86 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{carrier})$.

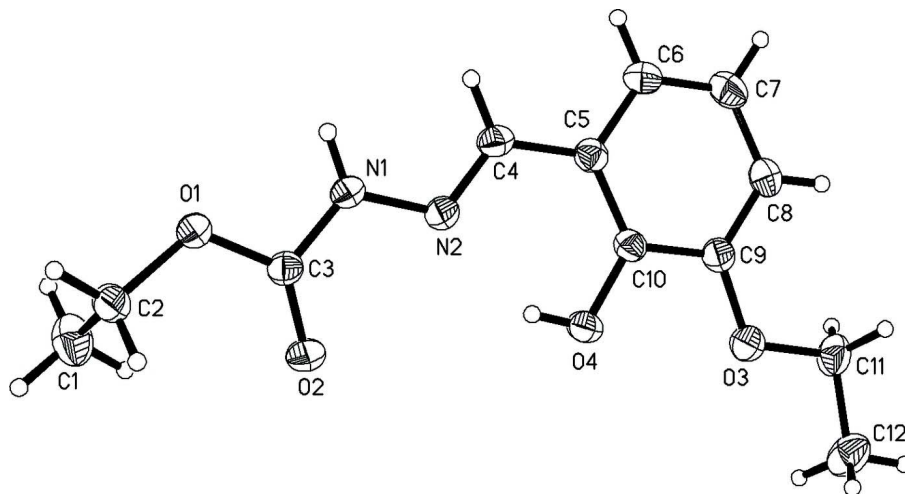


Figure 1

The molecular structure of (I) showing 30% probability displacement ellipsoids.

Ethyl 3-(3-ethoxy-2-hydroxybenzylidene)carbazate

Crystal data

$\text{C}_{12}\text{H}_{16}\text{N}_2\text{O}_4$

$M_r = 252.27$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.1140$ (14) Å

$b = 9.6010$ (19) Å

$c = 18.570$ (4) Å

$V = 1268.4$ (4) Å³

$Z = 4$

$F(000) = 536$

$D_x = 1.321$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1982 reflections

$\theta = 3.5\text{--}27.3^\circ$

$\mu = 0.10$ mm⁻¹

$T = 293$ K

Block, colourless

$0.22 \times 0.20 \times 0.19$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

11976 measured reflections

2917 independent reflections

2680 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.1^\circ$

$h = -8 \rightarrow 9$

$k = -12 \rightarrow 12$

$l = -23 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.090$

$S = 1.07$

2917 reflections

163 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

$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2 + 0.0849P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.32636 (13)	0.00340 (9)	0.17092 (5)	0.0507 (3)
N2	0.73581 (14)	-0.03948 (10)	0.26990 (6)	0.0400 (2)
O4	0.90478 (14)	-0.23476 (9)	0.34513 (6)	0.0514 (3)
H4A	0.8193	-0.2008	0.3212	0.077*
N1	0.59340 (15)	0.01343 (11)	0.22830 (6)	0.0447 (3)
H1A	0.5964	0.0986	0.2140	0.054*
O2	0.43343 (15)	-0.19175 (9)	0.22560 (6)	0.0530 (3)
C3	0.44812 (18)	-0.07057 (13)	0.21020 (7)	0.0398 (3)
O3	1.19430 (15)	-0.31435 (11)	0.41917 (6)	0.0563 (3)
C10	1.04046 (17)	-0.13798 (13)	0.35560 (7)	0.0379 (3)
C4	0.87138 (19)	0.04316 (13)	0.28510 (7)	0.0418 (3)
H4B	0.8679	0.1344	0.2684	0.050*
C5	1.03034 (18)	-0.00269 (13)	0.32791 (7)	0.0396 (3)
C8	1.3410 (2)	-0.08581 (17)	0.40856 (8)	0.0522 (3)
H8A	1.4449	-0.1125	0.4356	0.063*
C9	1.1983 (2)	-0.17984 (14)	0.39607 (7)	0.0433 (3)
C11	1.3491 (2)	-0.36144 (18)	0.46182 (8)	0.0569 (4)

H11A	1.3584	-0.3070	0.5057	0.068*
H11B	1.4657	-0.3519	0.4352	0.068*
C6	1.1784 (2)	0.08977 (15)	0.34131 (8)	0.0500 (3)
H6A	1.1732	0.1799	0.3231	0.060*
C2	0.1594 (2)	-0.06859 (15)	0.14570 (9)	0.0503 (3)
H2B	0.0623	-0.0007	0.1351	0.060*
H2C	0.1131	-0.1291	0.1836	0.060*
C7	1.3308 (2)	0.04877 (18)	0.38097 (9)	0.0583 (4)
H7A	1.4282	0.1112	0.3895	0.070*
C12	1.3135 (3)	-0.51120 (19)	0.47940 (11)	0.0702 (5)
H12A	1.4159	-0.5468	0.5076	0.105*
H12B	1.3032	-0.5638	0.4356	0.105*
H12C	1.1987	-0.5191	0.5063	0.105*
C1	0.1972 (3)	-0.1532 (2)	0.08008 (10)	0.0710 (5)
H1B	0.0839	-0.1994	0.0654	0.106*
H1C	0.2922	-0.2212	0.0905	0.106*
H1D	0.2399	-0.0933	0.0420	0.106*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0523 (5)	0.0346 (5)	0.0654 (6)	0.0008 (4)	-0.0193 (5)	0.0033 (4)
N2	0.0422 (5)	0.0344 (5)	0.0433 (5)	0.0040 (4)	-0.0032 (4)	0.0007 (4)
O4	0.0488 (5)	0.0345 (5)	0.0708 (6)	-0.0054 (4)	-0.0190 (5)	0.0043 (4)
N1	0.0486 (6)	0.0311 (5)	0.0545 (6)	0.0019 (4)	-0.0116 (5)	0.0051 (4)
O2	0.0618 (6)	0.0288 (4)	0.0683 (6)	0.0011 (4)	-0.0139 (5)	0.0029 (4)
C3	0.0454 (6)	0.0310 (5)	0.0429 (6)	0.0050 (5)	-0.0035 (6)	-0.0037 (5)
O3	0.0554 (6)	0.0443 (5)	0.0693 (6)	0.0021 (4)	-0.0214 (5)	0.0068 (4)
C10	0.0393 (6)	0.0355 (6)	0.0387 (6)	-0.0011 (5)	-0.0014 (5)	-0.0041 (5)
C4	0.0484 (6)	0.0325 (6)	0.0445 (6)	0.0012 (5)	0.0015 (6)	0.0015 (5)
C5	0.0413 (6)	0.0398 (6)	0.0376 (6)	-0.0017 (5)	0.0014 (5)	-0.0025 (5)
C8	0.0446 (7)	0.0583 (9)	0.0535 (8)	-0.0041 (6)	-0.0122 (7)	-0.0008 (6)
C9	0.0457 (7)	0.0416 (7)	0.0426 (6)	0.0011 (5)	-0.0049 (6)	-0.0016 (5)
C11	0.0536 (8)	0.0620 (9)	0.0550 (8)	0.0109 (7)	-0.0132 (7)	0.0057 (7)
C6	0.0533 (8)	0.0414 (7)	0.0554 (8)	-0.0104 (6)	-0.0007 (7)	0.0036 (6)
C2	0.0455 (7)	0.0405 (7)	0.0647 (8)	0.0012 (6)	-0.0117 (7)	-0.0035 (6)
C7	0.0506 (8)	0.0592 (9)	0.0650 (9)	-0.0189 (7)	-0.0082 (7)	-0.0003 (7)
C12	0.0665 (10)	0.0650 (10)	0.0791 (11)	0.0153 (9)	-0.0034 (9)	0.0201 (9)
C1	0.0691 (10)	0.0819 (12)	0.0618 (9)	0.0041 (10)	-0.0130 (9)	-0.0153 (9)

Geometric parameters (Å, °)

O1—C3	1.3367 (15)	C8—C7	1.392 (2)
O1—C2	1.4516 (17)	C8—H8A	0.9300
N2—C4	1.2803 (17)	C11—C12	1.496 (3)
N2—N1	1.3715 (14)	C11—H11A	0.9700
O4—C10	1.3539 (15)	C11—H11B	0.9700
O4—H4A	0.8200	C6—C7	1.368 (2)

N1—C3	1.3533 (16)	C6—H6A	0.9300
N1—H1A	0.8600	C2—C1	1.489 (2)
O2—C3	1.2026 (15)	C2—H2B	0.9700
O3—C9	1.3611 (17)	C2—H2C	0.9700
O3—C11	1.4299 (17)	C7—H7A	0.9300
C10—C5	1.3988 (18)	C12—H12A	0.9600
C10—C9	1.4095 (18)	C12—H12B	0.9600
C4—C5	1.4508 (18)	C12—H12C	0.9600
C4—H4B	0.9300	C1—H1B	0.9600
C5—C6	1.3998 (18)	C1—H1C	0.9600
C8—C9	1.3783 (19)	C1—H1D	0.9600
C3—O1—C2	116.95 (10)	O3—C11—H11B	110.3
C4—N2—N1	116.81 (10)	C12—C11—H11B	110.3
C10—O4—H4A	109.5	H11A—C11—H11B	108.5
C3—N1—N2	118.90 (10)	C7—C6—C5	120.62 (13)
C3—N1—H1A	120.5	C7—C6—H6A	119.7
N2—N1—H1A	120.5	C5—C6—H6A	119.7
O2—C3—O1	125.97 (12)	O1—C2—C1	112.09 (13)
O2—C3—N1	125.72 (12)	O1—C2—H2B	109.2
O1—C3—N1	108.29 (10)	C1—C2—H2B	109.2
C9—O3—C11	117.30 (12)	O1—C2—H2C	109.2
O4—C10—C5	123.22 (11)	C1—C2—H2C	109.2
O4—C10—C9	116.66 (11)	H2B—C2—H2C	107.9
C5—C10—C9	120.12 (11)	C6—C7—C8	120.42 (13)
N2—C4—C5	121.33 (11)	C6—C7—H7A	119.8
N2—C4—H4B	119.3	C8—C7—H7A	119.8
C5—C4—H4B	119.3	C11—C12—H12A	109.5
C10—C5—C6	119.00 (12)	C11—C12—H12B	109.5
C10—C5—C4	121.56 (11)	H12A—C12—H12B	109.5
C6—C5—C4	119.44 (12)	C11—C12—H12C	109.5
C9—C8—C7	120.52 (13)	H12A—C12—H12C	109.5
C9—C8—H8A	119.7	H12B—C12—H12C	109.5
C7—C8—H8A	119.7	C2—C1—H1B	109.5
O3—C9—C8	125.71 (12)	C2—C1—H1C	109.5
O3—C9—C10	114.97 (12)	H1B—C1—H1C	109.5
C8—C9—C10	119.32 (12)	C2—C1—H1D	109.5
O3—C11—C12	107.13 (14)	H1B—C1—H1D	109.5
O3—C11—H11A	110.3	H1C—C1—H1D	109.5
C12—C11—H11A	110.3		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O4—H4A \cdots N2	0.82	1.91	2.6290 (15)	145
N1—H1A \cdots O2 ⁱ	0.86	2.31	2.9633 (15)	132

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