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Ethyl 3-[1-(2-hydroxyphenyl)ethylidene]-carbazate

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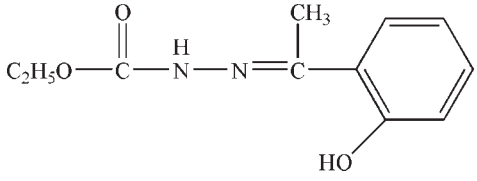
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.180; data-to-parameter ratio = 15.6.

The title compound, $C_{11}H_{14}N_2O_3$, was prepared by the reaction of ethyl carbazate and 1-(2-hydroxyphenyl)ethanone. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds, forming centrosymmetric dimers. An intramolecular O—H···N interaction also occurs.

Related literature

For the applications of Schiff base compounds, see: Cimerman *et al.* (1997); Forthe C=N double-bond length in a related structure, see: Girgis (2006).



Experimental

Crystal data

$C_{11}H_{14}N_2O_3$
M_r = 222.24
Triclinic, $P\overline{1}$
a = 5.4830 (11) Å
b = 10.191 (2) Å
c = 11.410 (2) Å
α = 112.53 (3)°
β = 95.79 (3)°
γ = 99.68 (3)°
V = 570.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.491, T_{max} = 0.728
5650 measured reflections
2597 independent reflections
1839 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.180
S = 1.08
2597 reflections
166 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.92 (2)	2.05 (2)	2.9649 (19)	170.4 (16)
O3—H3A···N1	0.98 (3)	1.68 (3)	2.5728 (19)	149 (2)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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: LH2935).

References

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supplementary materials

Acta Cryst. (2009). E**65**, o2935 [**doi:10.1107/S1600536809044602**]

Ethyl 3-[1-(2-hydroxyphenyl)ethylidene]carbazate

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Comment

Schiff bases have received considerable attention in the literature and have potential analytical applications (Cimerman *et al.*, 1997). As part of our search for new schiff base compounds we synthesized the title compound (I), and its crystal structure is determined herein.

The molecular structure of (I) is shown in Fig. 1. The C7—N1 bond length of 1.2836 (18)Å is comparable with C—N double bond [1.281 (2) Å] reported (Girgis, 2006). In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds to form centrosymmetric dimers.

Experimental

A mixture of the 1-(2-hydroxyphenyl)ethanone (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%). Single crystals suitable for X-ray measurements were obtained by recrystallization of (I) from ethanol at room temperature.

Refinement

H atoms bonded to the O atom, the N atom and those bonded to C8 were refined independently with isotropic displacement parameters. All other H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93–0.97 Å U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

Figures

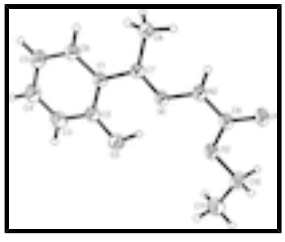

Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Ethyl 3-[1-(2-hydroxyphenyl)ethylidene]carbazate

Crystal data

$C_{11}H_{14}N_2O_3$	$Z = 2$
$M_r = 222.24$	$F_{000} = 236$
Triclinic, $P\overline{1}$	$D_x = 1.293$ Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 5.4830 (11) Å	Cell parameters from 1974 reflections
b = 10.191 (2) Å	θ = 3.5–27.5º
c = 11.410 (2) Å	μ = 0.10 mm^{-1}
α = 112.53 (3)º	T = 293 K
β = 95.79 (3)º	Block, colorless
γ = 99.68 (3)º	0.22 × 0.20 × 0.18 mm
V = 570.9 (2) Å^3	

Data collection

Bruker SMART CCD diffractometer	2597 independent reflections
Radiation source: fine-focus sealed tube	1839 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.016
T = 293 K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 6$
T_{min} = 0.491, T_{max} = 0.728	$k = -13 \rightarrow 13$
5650 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.1217P)^2 + 0.0113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.180	$(\Delta/\sigma)_{max}$ = 0.003
S = 1.08	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
2597 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
166 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.043 (14)
Secondary atom site location: difference Fourier map	

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.4674 (2)	0.34070 (13)	0.74631 (11)	0.0681 (4)
O1	0.2984 (2)	0.09910 (13)	1.08787 (10)	0.0676 (4)
O2	0.52301 (18)	0.24189 (11)	1.00894 (9)	0.0549 (3)
N1	0.1757 (2)	0.16596 (12)	0.81259 (10)	0.0476 (3)
N2	0.1444 (2)	0.10360 (14)	0.89965 (11)	0.0536 (3)
C1	0.0453 (2)	0.20676 (14)	0.63011 (12)	0.0453 (3)
C9	0.3238 (3)	0.14591 (16)	1.00535 (13)	0.0505 (4)
C7	−0.0055 (2)	0.13722 (14)	0.71973 (12)	0.0440 (3)
C3	0.3121 (3)	0.36489 (19)	0.55821 (16)	0.0682 (5)
H3B	0.4653	0.4273	0.5690	0.082*
C2	0.2752 (3)	0.30312 (15)	0.64688 (14)	0.0512 (4)
C10	0.7203 (3)	0.29608 (19)	1.12246 (15)	0.0635 (4)
H10A	0.7912	0.2169	1.1274	0.076*
H10B	0.6526	0.3396	1.2002	0.076*
C8	−0.2561 (3)	0.0382 (2)	0.69869 (19)	0.0638 (5)
C5	−0.0996 (4)	0.2415 (2)	0.43765 (17)	0.0728 (5)
H5A	−0.2254	0.2202	0.3677	0.087*
C6	−0.1370 (3)	0.17999 (19)	0.52442 (16)	0.0626 (4)
H6A	−0.2912	0.1176	0.5120	0.075*
C4	0.1279 (4)	0.3354 (2)	0.45604 (17)	0.0737 (5)
H4A	0.1555	0.3787	0.3987	0.088*
C11	0.9167 (3)	0.40714 (19)	1.10931 (17)	0.0706 (5)
H11A	1.0503	0.4458	1.1829	0.106*
H11B	0.8441	0.4846	1.1043	0.106*
H11C	0.9825	0.3626	1.0323	0.106*
H3A	0.407 (5)	0.283 (3)	0.794 (2)	0.107 (8)*
H2A	−0.001 (4)	0.039 (2)	0.8940 (17)	0.074 (5)*
H8A	−0.392 (5)	0.072 (3)	0.670 (2)	0.127 (9)*
H8B	−0.298 (5)	0.034 (3)	0.767 (3)	0.133 (10)*
H8C	−0.259 (6)	−0.042 (4)	0.637 (3)	0.139 (11)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0548 (6)	0.0760 (7)	0.0715 (7)	−0.0132 (5)	−0.0027 (5)	0.0428 (6)
O1	0.0639 (7)	0.0847 (8)	0.0621 (6)	−0.0099 (5)	−0.0004 (5)	0.0522 (6)
O2	0.0530 (6)	0.0615 (6)	0.0509 (5)	−0.0086 (4)	−0.0006 (4)	0.0349 (5)
N1	0.0485 (6)	0.0524 (6)	0.0464 (6)	0.0009 (4)	0.0051 (5)	0.0302 (5)
N2	0.0497 (7)	0.0614 (7)	0.0534 (6)	−0.0050 (5)	0.0018 (5)	0.0365 (5)
C1	0.0476 (7)	0.0474 (7)	0.0444 (7)	0.0075 (5)	0.0079 (5)	0.0239 (5)
C9	0.0533 (7)	0.0528 (7)	0.0499 (7)	0.0019 (6)	0.0069 (6)	0.0306 (6)
C7	0.0426 (6)	0.0461 (7)	0.0435 (6)	0.0042 (5)	0.0072 (5)	0.0209 (5)

C3	0.0708 (10)	0.0677 (10)	0.0746 (10)	−0.0033 (7)	0.0139 (8)	0.0458 (8)
C2	0.0524 (8)	0.0489 (7)	0.0534 (7)	0.0032 (5)	0.0080 (6)	0.0257 (6)
C10	0.0605 (9)	0.0695 (10)	0.0554 (8)	−0.0072 (7)	−0.0040 (7)	0.0326 (7)
C8	0.0498 (8)	0.0802 (11)	0.0650 (9)	−0.0084 (7)	0.0004 (7)	0.0454 (9)
C5	0.0723 (11)	0.0932 (13)	0.0665 (9)	0.0105 (9)	0.0020 (8)	0.0531 (9)
C6	0.0535 (8)	0.0794 (10)	0.0601 (8)	0.0011 (7)	0.0020 (7)	0.0417 (8)
C4	0.0878 (12)	0.0808 (11)	0.0724 (10)	0.0118 (9)	0.0155 (9)	0.0552 (9)
C11	0.0631 (9)	0.0640 (10)	0.0740 (10)	−0.0072 (7)	0.0042 (8)	0.0274 (8)

Geometric parameters (Å, °)

O3—C2	1.3543 (19)	C3—H3B	0.9300
O3—H3A	0.98 (2)	C10—C11	1.489 (2)
O1—C9	1.2173 (16)	C10—H10A	0.9700
O2—C9	1.3237 (17)	C10—H10B	0.9700
O2—C10	1.4563 (18)	C8—H8A	0.95 (3)
N1—C7	1.2836 (18)	C8—H8B	0.85 (3)
N1—N2	1.3790 (15)	C8—H8C	0.84 (3)
N2—C9	1.3521 (19)	C5—C6	1.374 (2)
N2—H2A	0.92 (2)	C5—C4	1.381 (3)
C1—C6	1.393 (2)	C5—H5A	0.9300
C1—C2	1.408 (2)	C6—H6A	0.9300
C1—C7	1.4737 (18)	C4—H4A	0.9300
C7—C8	1.4988 (19)	C11—H11A	0.9600
C3—C4	1.365 (2)	C11—H11B	0.9600
C3—C2	1.395 (2)	C11—H11C	0.9600
C2—O3—H3A	104.2 (15)	O2—C10—H10B	110.3
C9—O2—C10	116.29 (10)	C11—C10—H10B	110.3
C7—N1—N2	119.65 (11)	H10A—C10—H10B	108.6
C9—N2—N1	119.71 (11)	C7—C8—H8A	112.7 (16)
C9—N2—H2A	116.8 (11)	C7—C8—H8B	114 (2)
N1—N2—H2A	123.3 (11)	H8A—C8—H8B	101 (2)
C6—C1—C2	116.97 (12)	C7—C8—H8C	107 (2)
C6—C1—C7	120.60 (12)	H8A—C8—H8C	105 (3)
C2—C1—C7	122.43 (12)	H8B—C8—H8C	117 (3)
O1—C9—O2	124.75 (13)	C6—C5—C4	118.87 (16)
O1—C9—N2	121.90 (13)	C6—C5—H5A	120.6
O2—C9—N2	113.34 (11)	C4—C5—H5A	120.6
N1—C7—C1	116.09 (11)	C5—C6—C1	123.05 (15)
N1—C7—C8	123.84 (12)	C5—C6—H6A	118.5
C1—C7—C8	120.06 (12)	C1—C6—H6A	118.5
C4—C3—C2	121.27 (14)	C3—C4—C5	120.15 (14)
C4—C3—H3B	119.4	C3—C4—H4A	119.9
C2—C3—H3B	119.4	C5—C4—H4A	119.9
O3—C2—C3	117.02 (13)	C10—C11—H11A	109.5
O3—C2—C1	123.31 (12)	C10—C11—H11B	109.5
C3—C2—C1	119.67 (13)	H11A—C11—H11B	109.5
O2—C10—C11	107.04 (13)	C10—C11—H11C	109.5
O2—C10—H10A	110.3	H11A—C11—H11C	109.5

C11—C10—H10A	110.3	H11B—C11—H11C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{i}	0.92 (2)	2.05 (2)	2.9649 (19)	170.4 (16)
O3—H3A···N1	0.98 (3)	1.68 (3)	2.5728 (19)	149 (2)

Symmetry codes: (i) −*x*, −*y*, −*z*+2.

Fig. 1