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Ethyl 3-benzylidenecarbazate

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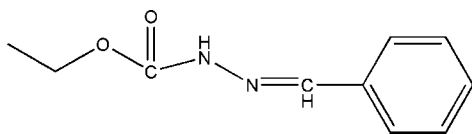
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
 R factor = 0.067; wR factor = 0.206; data-to-parameter ratio = 19.3.

In the title compound, $\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_2$, the dihedral angle between the mean planes of the aromatic ring and the side chain (r.m.s. deviation = 0.035 Å) is 18.23 (13)°. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, generating $C(4)$ amide chains propagating in [010].

Related literature

For related structures, see: Li & Jian (2010); Li & Meng (2010).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_2$
 $M_r = 192.22$

Orthorhombic, $Pbca$
 $a = 11.309$ (2) Å

$b = 7.6693$ (15) Å
 $c = 24.684$ (5) Å
 $V = 2140.8$ (7) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.22 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD
diffractometer
19439 measured reflections

2449 independent reflections
1147 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.100$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.206$
 $S = 1.00$
2449 reflections

127 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O2}^i$	0.86	2.04	2.885 (3)	168

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5754).

References

- Bruker (1997). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* **E66**, o1399.
Li, Y.-F. & Meng, F.-Y. (2010). *Acta Cryst.* **E66**, o2685.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2010). E66, o3337 [doi:10.1107/S1600536810048865]

Ethyl 3-benzylidenecarbazate

J.-H. Jiang

Experimental

A mixture of benzaldehyde (0.01 mol) and ethyl carbazate (0.01 mol) was stirred in refluxing ethanol (20 mL) for 2 h to afford the title compound (0.095 mol, yield 95%). Colourless blocks were obtained by recrystallization from ethanol at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.97 Å, and with $U_{\text{iso}}=1.2-1.5U_{\text{eq}}$.

Figures

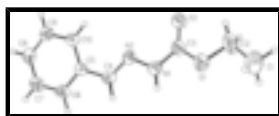


Fig. 1. The structure of the title compound showing 30% probability displacement ellipsoids.

Ethyl 3-benzylidenecarbazate

Crystal data

$\text{C}_{10}\text{H}_{12}\text{N}_2\text{O}_2$

$M_r = 192.22$

Orthorhombic, *Pbca*

$a = 11.309$ (2) Å

$b = 7.6693$ (15) Å

$c = 24.684$ (5) Å

$V = 2140.8$ (7) Å³

$Z = 8$

$F(000) = 816$

$D_x = 1.193$ Mg m⁻³

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

Cell parameters from 2449 reflections

$\theta = 3.3-27.5^\circ$

$\mu = 0.09$ mm⁻¹

$T = 293$ K

Block, colorless

$0.22 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

graphite

φ and ω scans

19439 measured reflections

1147 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.100$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.3^\circ$

$h = -14 \rightarrow 14$

$k = -9 \rightarrow 8$

supplementary materials

2449 independent reflections

$l = -32 \rightarrow 31$

Refinement

Refinement on F^2

Primary atom site location: structure-invariant direct methods

Least-squares matrix: full

Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.067$

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.206$

H-atom parameters constrained

$S = 1.00$

$w = 1/[\sigma^2(F_o^2) + (0.1041P)^2]$

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

2449 reflections

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

127 parameters

$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$

0 restraints

$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.26886 (18)	0.1974 (3)	0.64212 (7)	0.0581 (6)
C5	0.3635 (2)	0.2155 (3)	0.72854 (9)	0.0531 (6)
N1	0.2584 (2)	0.2687 (3)	0.59115 (7)	0.0683 (6)
H1A	0.3045	0.3519	0.5811	0.082*
C3	0.1754 (3)	0.2069 (4)	0.55722 (10)	0.0662 (7)
O2	0.11238 (17)	0.0819 (2)	0.56498 (7)	0.0752 (6)
C4	0.3463 (2)	0.2693 (3)	0.67226 (9)	0.0591 (7)
H4A	0.3930	0.3582	0.6581	0.071*
C6	0.4598 (2)	0.2798 (4)	0.75763 (10)	0.0663 (7)
H6A	0.5139	0.3528	0.7406	0.080*
O1	0.1713 (2)	0.3068 (3)	0.51302 (7)	0.0940 (7)
C10	0.2827 (2)	0.1092 (3)	0.75513 (9)	0.0591 (7)
H10A	0.2171	0.0668	0.7366	0.071*
C8	0.3955 (3)	0.1299 (4)	0.83678 (10)	0.0730 (8)
H8A	0.4062	0.1007	0.8730	0.088*
C9	0.2993 (2)	0.0663 (3)	0.80883 (10)	0.0689 (8)
H9A	0.2453	-0.0059	0.8263	0.083*

C7	0.4757 (2)	0.2363 (4)	0.81131 (11)	0.0751 (8)
H7A	0.5407	0.2790	0.8302	0.090*
C2	0.0890 (4)	0.2517 (5)	0.47098 (14)	0.1325 (16)
H2B	0.1114	0.1378	0.4573	0.159*
H2C	0.0097	0.2435	0.4858	0.159*
C1	0.0915 (5)	0.3756 (8)	0.42843 (15)	0.167 (2)
H1B	0.0372	0.3408	0.4005	0.250*
H1C	0.1699	0.3820	0.4137	0.250*
H1D	0.0689	0.4878	0.4422	0.250*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0598 (13)	0.0575 (13)	0.0569 (11)	0.0006 (10)	-0.0028 (9)	0.0056 (9)
C5	0.0501 (14)	0.0482 (14)	0.0610 (13)	0.0036 (11)	-0.0004 (10)	0.0012 (10)
N1	0.0776 (15)	0.0659 (14)	0.0613 (12)	-0.0123 (11)	-0.0069 (11)	0.0136 (10)
C3	0.0709 (18)	0.0651 (18)	0.0625 (15)	0.0030 (15)	-0.0064 (13)	0.0077 (13)
O2	0.0767 (13)	0.0716 (13)	0.0773 (11)	-0.0072 (11)	-0.0111 (9)	0.0063 (9)
C4	0.0573 (15)	0.0547 (15)	0.0653 (14)	-0.0023 (12)	0.0026 (11)	0.0063 (11)
C6	0.0498 (15)	0.0712 (18)	0.0778 (17)	-0.0037 (13)	-0.0045 (12)	0.0056 (12)
O1	0.1229 (17)	0.0917 (15)	0.0675 (11)	-0.0224 (13)	-0.0252 (11)	0.0214 (10)
C10	0.0559 (15)	0.0573 (15)	0.0643 (15)	-0.0027 (12)	-0.0024 (11)	0.0006 (11)
C8	0.0762 (19)	0.077 (2)	0.0653 (15)	0.0128 (16)	-0.0065 (14)	0.0001 (12)
C9	0.0742 (19)	0.0658 (17)	0.0666 (15)	-0.0025 (14)	0.0069 (13)	0.0058 (12)
C7	0.0577 (17)	0.089 (2)	0.0783 (17)	-0.0010 (15)	-0.0175 (14)	-0.0027 (15)
C2	0.174 (4)	0.142 (3)	0.082 (2)	-0.046 (3)	-0.063 (2)	0.026 (2)
C1	0.172 (5)	0.231 (6)	0.096 (3)	-0.051 (4)	-0.045 (3)	0.054 (3)

Geometric parameters (\AA , $^\circ$)

N2—C4	1.275 (3)	C10—C9	1.379 (3)
N2—N1	1.377 (2)	C10—H10A	0.9300
C5—C10	1.389 (3)	C8—C7	1.372 (4)
C5—C6	1.394 (3)	C8—C9	1.378 (4)
C5—C4	1.462 (3)	C8—H8A	0.9300
N1—C3	1.345 (3)	C9—H9A	0.9300
N1—H1A	0.8600	C7—H7A	0.9300
C3—O2	1.210 (3)	C2—C1	1.417 (5)
C3—O1	1.334 (3)	C2—H2B	0.9700
C4—H4A	0.9300	C2—H2C	0.9700
C6—C7	1.378 (3)	C1—H1B	0.9600
C6—H6A	0.9300	C1—H1C	0.9600
O1—C2	1.457 (4)	C1—H1D	0.9600
C4—N2—N1	114.8 (2)	C7—C8—H8A	119.9
C10—C5—C6	118.5 (2)	C9—C8—H8A	119.9
C10—C5—C4	121.8 (2)	C8—C9—C10	120.3 (3)
C6—C5—C4	119.5 (2)	C8—C9—H9A	119.9
C3—N1—N2	119.3 (2)	C10—C9—H9A	119.9

supplementary materials

C3—N1—H1A	120.4	C8—C7—C6	119.8 (3)
N2—N1—H1A	120.4	C8—C7—H7A	120.1
O2—C3—O1	124.3 (2)	C6—C7—H7A	120.1
O2—C3—N1	126.3 (2)	C1—C2—O1	108.7 (3)
O1—C3—N1	109.3 (3)	C1—C2—H2B	109.9
N2—C4—C5	121.6 (2)	O1—C2—H2B	109.9
N2—C4—H4A	119.2	C1—C2—H2C	109.9
C5—C4—H4A	119.2	O1—C2—H2C	109.9
C7—C6—C5	120.8 (3)	H2B—C2—H2C	108.3
C7—C6—H6A	119.6	C2—C1—H1B	109.5
C5—C6—H6A	119.6	C2—C1—H1C	109.5
C3—O1—C2	116.0 (2)	H1B—C1—H1C	109.5
C9—C10—C5	120.3 (2)	C2—C1—H1D	109.5
C9—C10—H10A	119.8	H1B—C1—H1D	109.5
C5—C10—H10A	119.8	H1C—C1—H1D	109.5
C7—C8—C9	120.2 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O2^i$	0.86	2.04	2.885 (3)	168

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

Fig. 1

