Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 3-[1-(4-bromophenyl)ethylidene]-carbazate

Yu-Feng Li,[a] Hai-Xing Liu[a] and Fang-Fang Jian[b]*

[a]Microscale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China, and [b]Microscale Science Institute, Weifang University, Weifang 261061, People's Republic of China Correspondence e-mail: liyufeng8111@163.com

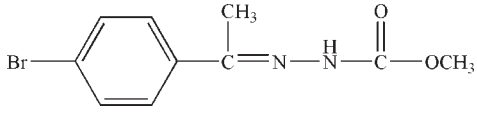
Received 26 October 2009; accepted 27 October 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.052; wR factor = 0.152; data-to-parameter ratio = 18.5.

In the crystal of the title compound, $C_{10}H_{11}BrN_2O_2$, the molecules are linked by N—H···O hydrogen bonds, forming S(4) chains propagating in [100]. A C—H···O interaction also occurs.

Related literature

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



Experimental

Crystal data

$C_{10}H_{11}BrN_2O_2$
M_r = 271.11
Orthorhombic, $Pca2_1$
a = 7.6810 (15) Å
b = 5.9520 (12) Å
c = 24.750 (5) Å
V = 1131.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.62 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.19 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
10040 measured reflections
2583 independent reflections
1675 reflections with $I > 2\sigma(I)$
R_{int} = 0.116

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.152
S = 1.02
2583 reflections
140 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Absolute structure: Flack (1983), 1254 Friedel pairs
Flack parameter: −0.008 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1D···O1^{i}	0.76 (10)	2.26 (10)	2.929 (6)	148 (10)
C3—H3A···O1^{i}	0.96	2.43	3.242 (8)	142

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

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*.

The authors would like to thank the Science Foundation of Weifang University (No. 2009Z24).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5186).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cimerman, Z., Galic, N. & Bosner, B. (1997). *Anal. Chim. Acta*, **343**, 145–153.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2938 [https://doi.org/10.1107/S1600536809044614]

Ethyl 3-[1-(4-bromophenyl)ethylidene]carbazate

Yu-Feng Li, Hai-Xing Liu and Fang-Fang Jian

S1. Experimental

A mixture of 1-(4-bromophenyl)ethanone (0.1 mol), and methyl carbazate (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.085 mol, yield 85%). Colourless blocks of (I) were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

The N-bound H atom was located in a difference map and freely refined. The other H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.97 Å, and with U_{iso}=1.2–1.5U_{eq}.

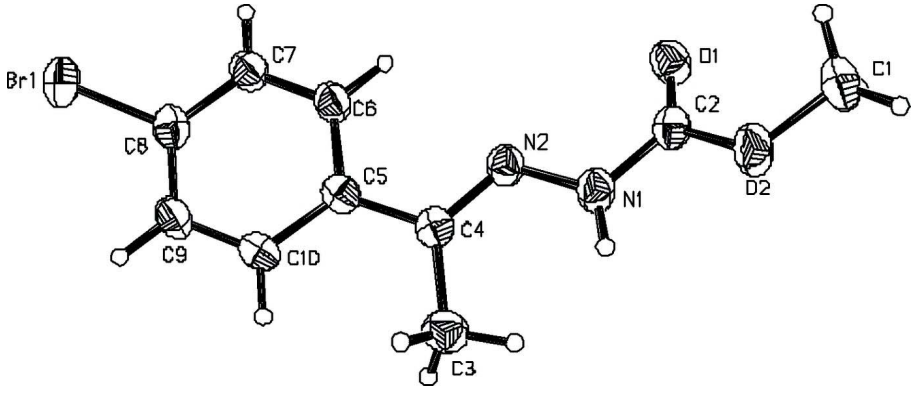


Figure 1

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

Ethyl 3-[1-(4-bromophenyl)ethylidene]carbazate

Crystal data

$C_{10}H_{11}BrN_2O_2$
M_r = 271.11
Orthorhombic, $Pca2_1$
a = 7.6810 (15) Å
b = 5.9520 (12) Å
c = 24.750 (5) Å
V = 1131.5 (4) Å^3
Z = 4
F(000) = 544
D_x = 1.591 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1982 reflections
θ = 3.6–27.5°
μ = 3.62 mm^{-1}
T = 293 K
Block, colourless
0.25 × 0.20 × 0.19 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
10040 measured reflections
2583 independent reflections
1675 reflections with $I > 2\sigma(I)$
$R_{int} = 0.116$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -9 \rightarrow 8$
$k = -7 \rightarrow 7$
$l = -31 \rightarrow 32$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.152$
$S = 1.02$
2583 reflections
140 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.054$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}
Absolute structure: Flack (1983), 1254 Friedel pairs
Absolute structure parameter: −0.008 (16)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.44214 (8)	0.19577 (12)	0.58046 (2)	0.0815 (3)
C8	0.3535 (7)	0.2162 (9)	0.5091 (2)	0.0584 (13)
C1	0.0415 (10)	−0.3211 (12)	0.1630 (3)	0.080 (2)
H1A	−0.0495	−0.3171	0.1364	0.120*
H1B	0.0344	−0.4590	0.1830	0.120*
H1C	0.1526	−0.3121	0.1454	0.120*
N1	0.1035 (6)	0.0551 (8)	0.27043 (18)	0.0548 (10)
C7	0.3861 (7)	0.0435 (10)	0.4736 (2)	0.0600 (12)
H7A	0.4517	−0.0798	0.4845	0.072*
C2	0.1408 (6)	−0.1185 (9)	0.23832 (19)	0.0515 (11)
O2	0.0223 (6)	−0.1363 (9)	0.19887 (18)	0.0724 (12)
C9	0.2600 (7)	0.4010 (9)	0.4932 (2)	0.0610 (12)
H9A	0.2385	0.5177	0.5172	0.073*
C4	0.1498 (6)	0.2404 (8)	0.3496 (2)	0.0463 (10)
C5	0.2249 (6)	0.2378 (8)	0.4043 (2)	0.0470 (10)

N2	0.1849 (5)	0.0686 (7)	0.31978 (17)	0.0509 (9)
C10	0.1984 (6)	0.4113 (9)	0.4408 (2)	0.0542 (11)
H10A	0.1374	0.5381	0.4297	0.065*
C6	0.3215 (7)	0.0544 (9)	0.4220 (2)	0.0552 (12)
H6A	0.3428	−0.0637	0.3983	0.066*
O1	0.2632 (5)	−0.2456 (6)	0.24299 (17)	0.0611 (9)
C3	0.0382 (7)	0.4343 (10)	0.3314 (3)	0.0633 (14)
H3A	0.0001	0.4090	0.2950	0.095*
H3B	0.1047	0.5707	0.3329	0.095*
H3C	−0.0613	0.4469	0.3547	0.095*
H1D	0.017 (13)	0.111 (17)	0.276 (4)	0.10 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1019 (5)	0.0934 (5)	0.0491 (3)	−0.0009 (3)	−0.0070 (4)	−0.0089 (4)
C8	0.057 (3)	0.067 (3)	0.051 (3)	−0.003 (2)	0.007 (2)	−0.008 (2)
C1	0.099 (5)	0.077 (5)	0.063 (4)	−0.003 (3)	−0.014 (3)	−0.023 (3)
N1	0.049 (2)	0.066 (3)	0.050 (2)	0.004 (2)	−0.0056 (19)	−0.007 (2)
C7	0.063 (3)	0.060 (3)	0.057 (3)	0.010 (2)	−0.006 (2)	−0.008 (2)
C2	0.050 (3)	0.059 (3)	0.045 (2)	−0.006 (2)	−0.003 (2)	−0.003 (2)
O2	0.070 (2)	0.085 (3)	0.062 (3)	0.011 (2)	−0.024 (2)	−0.020 (2)
C9	0.073 (3)	0.055 (3)	0.056 (3)	0.004 (3)	0.008 (2)	−0.015 (3)
C4	0.040 (2)	0.051 (2)	0.049 (3)	−0.0062 (19)	0.0064 (19)	−0.001 (2)
C5	0.039 (2)	0.047 (2)	0.055 (3)	−0.0015 (18)	0.0064 (19)	−0.004 (2)
N2	0.048 (2)	0.057 (2)	0.047 (2)	0.0031 (17)	0.0013 (16)	−0.002 (2)
C10	0.055 (3)	0.045 (3)	0.063 (3)	0.0062 (19)	0.003 (2)	−0.007 (2)
C6	0.058 (3)	0.057 (3)	0.051 (3)	0.009 (2)	0.003 (2)	−0.009 (2)
O1	0.055 (2)	0.064 (2)	0.065 (2)	0.0031 (18)	−0.0094 (16)	−0.0121 (18)
C3	0.061 (3)	0.058 (3)	0.070 (4)	0.008 (2)	−0.007 (2)	−0.002 (3)

Geometric parameters (Å, º)

Br1—C8	1.896 (6)	C2—O2	1.339 (6)
C8—C9	1.372 (8)	C9—C10	1.380 (7)
C8—C7	1.376 (7)	C9—H9A	0.9300
C1—O2	1.421 (8)	C4—N2	1.289 (6)
C1—H1A	0.9600	C4—C5	1.473 (7)
C1—H1B	0.9600	C4—C3	1.507 (7)
C1—H1C	0.9600	C5—C10	1.387 (7)
N1—C2	1.335 (7)	C5—C6	1.391 (7)
N1—N2	1.374 (6)	C10—H10A	0.9300
N1—H1D	0.75 (10)	C6—H6A	0.9300
C7—C6	1.371 (7)	C3—H3A	0.9600
C7—H7A	0.9300	C3—H3B	0.9600
C2—O1	1.212 (6)	C3—H3C	0.9600
C9—C8—C7	120.7 (5)	C10—C9—H9A	120.5

C9—C8—Br1	120.5 (4)	N2—C4—C5	115.8 (4)
C7—C8—Br1	118.8 (4)	N2—C4—C3	123.8 (5)
O2—C1—H1A	109.5	C5—C4—C3	120.4 (4)
O2—C1—H1B	109.5	C10—C5—C6	117.2 (5)
H1A—C1—H1B	109.5	C10—C5—C4	122.3 (4)
O2—C1—H1C	109.5	C6—C5—C4	120.5 (4)
H1A—C1—H1C	109.5	C4—N2—N1	117.4 (4)
H1B—C1—H1C	109.5	C9—C10—C5	121.9 (5)
C2—N1—N2	118.5 (4)	C9—C10—H10A	119.1
C2—N1—H1D	129 (7)	C5—C10—H10A	119.1
N2—N1—H1D	103 (7)	C7—C6—C5	121.6 (5)
C8—C7—C6	119.6 (5)	C7—C6—H6A	119.2
C8—C7—H7A	120.2	C5—C6—H6A	119.2
C6—C7—H7A	120.2	C4—C3—H3A	109.5
O1—C2—N1	126.4 (5)	C4—C3—H3B	109.5
O1—C2—O2	123.2 (5)	H3A—C3—H3B	109.5
N1—C2—O2	110.4 (5)	C4—C3—H3C	109.5
C2—O2—C1	116.5 (5)	H3A—C3—H3C	109.5
C8—C9—C10	119.0 (5)	H3B—C3—H3C	109.5
C8—C9—H9A	120.5		

C9—C8—C7—C6	−1.3 (8)	C3—C4—C5—C6	177.1 (5)
Br1—C8—C7—C6	179.3 (4)	C5—C4—N2—N1	173.9 (4)
N2—N1—C2—O1	−15.0 (8)	C3—C4—N2—N1	−5.5 (7)
N2—N1—C2—O2	164.9 (5)	C2—N1—N2—C4	178.4 (5)
O1—C2—O2—C1	2.8 (9)	C8—C9—C10—C5	1.5 (8)
N1—C2—O2—C1	−177.1 (6)	C6—C5—C10—C9	−2.0 (8)
C7—C8—C9—C10	0.2 (8)	C4—C5—C10—C9	175.8 (4)
Br1—C8—C9—C10	179.5 (4)	C8—C7—C6—C5	0.8 (8)
N2—C4—C5—C10	180.0 (4)	C10—C5—C6—C7	0.8 (7)
C3—C4—C5—C10	−0.6 (7)	C4—C5—C6—C7	−177.0 (5)
N2—C4—C5—C6	−2.3 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*D*···O1^{i}	0.76 (10)	2.26 (10)	2.929 (6)	148 (10)
C3—H3*A*···O1^{i}	0.96	2.43	3.242 (8)	142

Symmetry code: (i) *x*−1/2, −*y*, *z*.