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

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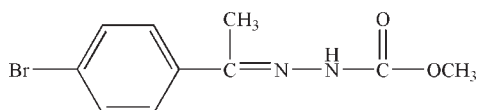
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.052; wR factor = 0.152; data-to-parameter ratio = 18.5.

In the crystal of the title compound, $\text{C}_{10}\text{H}_{11}\text{BrN}_2\text{O}_2$, the molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming $S(4)$ chains propagating in $[100]$. A $\text{C}-\text{H}\cdots\text{O}$ interaction also occurs.

Related literature

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



Experimental

Crystal data

$\text{C}_{10}\text{H}_{11}\text{BrN}_2\text{O}_2$

$M_r = 271.11$

Orthorhombic, $Pca2_1$

$a = 7.6810$ (15) Å

$b = 5.9520$ (12) Å

$c = 24.750$ (5) Å

$V = 1131.5$ (4) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 3.62$ mm⁻¹

$T = 293$ K

$0.25 \times 0.20 \times 0.19$ mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
10040 measured reflections

2583 independent reflections
1675 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.116$

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

$\Delta\rho_{\text{max}} = 0.45$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.54$ e Å⁻³

Absolute structure: Flack (1983), 1254 Friedel pairs

Flack parameter: -0.008 (16)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1D}\cdots\text{O1}^{\text{i}}$	0.76 (10)	2.26 (10)	2.929 (6)	148 (10)
$\text{C3}-\text{H3A}\cdots\text{O1}^{\text{i}}$	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*.

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

References

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supplementary materials

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

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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.

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 $U_{\text{iso}}=1.2\text{--}1.5U_{\text{eq}}$.

Figures

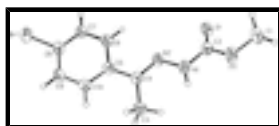


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids.

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

Crystal data

$\text{C}_{10}\text{H}_{11}\text{BrN}_2\text{O}_2$

$M_r = 271.11$

Orthorhombic, $Pca2_1$

$a = 7.6810(15) \text{ \AA}$

$b = 5.9520(12) \text{ \AA}$

$c = 24.750(5) \text{ \AA}$

$V = 1131.5(4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 544$

$D_x = 1.591 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1982 reflections

$\theta = 3.6\text{--}27.5^\circ$

$\mu = 3.62 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colourless

$0.25 \times 0.20 \times 0.19 \text{ mm}$

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293 \text{ K}$

ω scans

1675 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.116$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 3.3^\circ$

$h = -9 \rightarrow 8$

supplementary materials

Absorption correction: none
10040 measured reflections
2583 independent reflections

$k = -7 \rightarrow 7$
 $l = -31 \rightarrow 32$

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

$R[F^2 > 2\sigma(F^2)] = 0.052$

$$w = 1/[\sigma^2(F_o^2) + (0.068P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$wR(F^2) = 0.152$

$$(\Delta/\sigma)_{\max} = 0.054$$

$S = 1.02$

$$\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$$

2583 reflections

$$\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$$

140 parameters

Extinction correction: none

1 restraint

Absolute structure: Flack (1983), 1254 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: $-0.008 (16)$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^2)

	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 (\AA , $^\circ$)

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

supplementary materials

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 (\AA , $^\circ$)

<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>
N1—H1D \cdots O1 ⁱ	0.76 (10)	2.26 (10)	2.929 (6)	148 (10)
C3—H3A \cdots O1 ⁱ	0.96	2.43	3.242 (8)	142

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

Fig. 1

