Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 5-bromo-1-benzofuran-2-carboxylate

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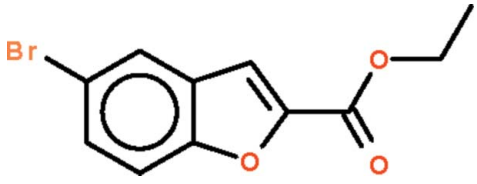
Received 14 February 2011; accepted 16 February 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.055; wR factor = 0.109; data-to-parameter ratio = 16.5.

In the title compound, $C_{11}H_9BrO_3$, the benzofuran fused-ring system is almost planar, with a maximum atomic deviation of 0.024 (5) Å; the carboxyl –CO_2 fragment is aligned at 4.8 (7)° with respect to the fused-ring plane. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure. π–π stacking is also observed between parallel molecules, the centroid–centroid distance between benzene and furan rings of adjacent molecules being 3.662 (3) Å.

Related literature

For our previous reports of the pharmacological properties of benzofurans, see: Abdel-Aziz & Mekawey (2009); Abdel-Aziz *et al.* (2009). For a related structure, see: Kossakowski *et al.* (2005).



Experimental

Crystal data

$C_{11}H_9BrO_3$
M_r = 269.09
Monoclinic, $P2_1/n$
a = 3.8869 (3) Å
b = 23.780 (2) Å
c = 11.0820 (7) Å
β = 96.905 (8)°
V = 1016.89 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.02 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.378, T_{max} = 0.689
6060 measured reflections
2250 independent reflections
1843 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.109
S = 1.18
2250 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.97 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2^{i}	0.95	2.57	3.400 (6)	146
C11—H11A···O2^{ii}	0.98	2.53	3.472 (6)	160

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank King Saud University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5164).

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supplementary materials

Acta Cryst. (2011). E**67**, o696 [**doi:10.1107/S1600536811005897**]

Ethyl 5-bromo-1-benzofuran-2-carboxylate

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Comment

Ethyl 5-bromobenzofuran-2-carboxylate (Scheme I) is a commercially available chemical that has been evaluated for its pharmacological properties. We have reported the pharmacological properties of related compounds (Abdel-Aziz & Mekawey, 2009; Abdel-Aziz *et al.*, 2009). The title compound is an approximately planar molecule; the carboxyl $–CO_2$ fragment is aligned at 4.8 (7)° with respect to the benzofuran fused-ring (Fig. 1). Bond dimensions are similar to those found in methyl 7-methoxybenzofuran-2-carboxylate (Kossakowski *et al.*, 2005).

Experimental

5-Bromosalicyladehyde (2.01 g, 10 mm l), diethyl bromomalonate (2.63 g 11 mmol) and potassium carbonate (2.28 g, 20 mmol) were heated in 2-butanone (20 ml) for 14 h. The solvent was evaporated and water was added to the residue. The organic compound was extracted by ether. The ether phase was washed with 5% sodium hydroxide. The ether was then evaporated and the product recrystallized from ethanol to give the title ester, m.p. 333–335 K.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

Figures

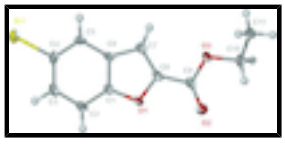

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{11}H_9BrO_3$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Ethyl 5-bromo-1-benzofuran-2-carboxylate

Crystal data

$C_{11}H_9BrO_3$	$F(000) = 536$
$M_r = 269.09$	$D_x = 1.758$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2599 reflections
$a = 3.8869$ (3) Å	$\theta = 2.5–29.3°$
$b = 23.780$ (2) Å	$\mu = 4.02$ mm^{-1}
$c = 11.0820$ (7) Å	$T = 100$ K

β = 96.905 (8)°	Prism, colorless
V = 1016.89 (13) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	2250 independent reflections
Radiation source: SuperNova (Mo) X-ray Source	1843 reflections with $I > 2\sigma(I)$
Mirror	R_{int} = 0.045
Detector resolution: 10.4041 pixels mm^{-1}	θ_{max} = 27.5°, θ_{min} = 2.5°
ω scans	h = −3→5
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	k = −30→30
T_{min} = 0.378, T_{max} = 0.689	l = −13→14
6060 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.055	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.109	H-atom parameters constrained
S = 1.18	$w = 1/[\sigma^2(F_o^2) + (0.0086P)^2 + 5.1797P]$ where $P = (F_o^2 + 2F_c^2)/3$
2250 reflections	$(\Delta/\sigma)_{max}$ = 0.001
136 parameters	$\Delta\rho_{max}$ = 0.97 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.84947 (14)	0.52531 (2)	0.18162 (5)	0.02119 (16)
O1	0.2581 (9)	0.32380 (14)	0.4040 (3)	0.0157 (7)
O2	0.0007 (10)	0.25870 (14)	0.5783 (3)	0.0228 (9)
O3	0.1752 (9)	0.32488 (14)	0.7194 (3)	0.0165 (8)
C1	0.3994 (13)	0.3662 (2)	0.3427 (4)	0.0146 (10)
C2	0.4194 (14)	0.3670 (2)	0.2183 (4)	0.0189 (11)
H2	0.3416	0.3363	0.1672	0.023*
C3	0.5588 (15)	0.4150 (2)	0.1733 (4)	0.0216 (12)
H3	0.5746	0.4181	0.0886	0.026*
C4	0.6767 (13)	0.4590 (2)	0.2510 (4)	0.0163 (11)
C5	0.6666 (13)	0.4576 (2)	0.3740 (4)	0.0151 (10)
H5	0.7541	0.4878	0.4248	0.018*
C6	0.5205 (12)	0.4095 (2)	0.4220 (4)	0.0135 (10)
C7	0.4517 (13)	0.3916 (2)	0.5397 (4)	0.0153 (10)

H7	0.5060	0.4113	0.6141	0.018*
C8	0.2932 (14)	0.3408 (2)	0.5243 (4)	0.0161 (11)
C9	0.1403 (13)	0.3029 (2)	0.6073 (4)	0.0157 (10)
C10	0.0273 (14)	0.2924 (2)	0.8131 (4)	0.0202 (11)
H10A	0.1957	0.2639	0.8488	0.024*
H10B	−0.1857	0.2728	0.7775	0.024*
C11	−0.0555 (14)	0.3333 (2)	0.9091 (4)	0.0202 (11)
H11A	−0.1540	0.3130	0.9738	0.030*
H11B	−0.2234	0.3611	0.8728	0.030*
H11C	0.1571	0.3526	0.9433	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0225 (3)	0.0210 (3)	0.0205 (3)	−0.0006 (2)	0.0046 (2)	0.0051 (2)
O1	0.020 (2)	0.0151 (17)	0.0107 (15)	−0.0015 (15)	−0.0019 (14)	−0.0010 (14)
O2	0.032 (2)	0.0169 (19)	0.0191 (18)	−0.0038 (17)	0.0022 (17)	−0.0014 (15)
O3	0.019 (2)	0.0196 (18)	0.0108 (15)	−0.0032 (15)	0.0007 (14)	0.0015 (14)
C1	0.015 (3)	0.016 (2)	0.013 (2)	0.003 (2)	−0.0009 (19)	0.0018 (19)
C2	0.021 (3)	0.022 (3)	0.013 (2)	0.002 (2)	−0.001 (2)	−0.002 (2)
C3	0.032 (3)	0.021 (3)	0.013 (2)	0.004 (2)	0.006 (2)	0.002 (2)
C4	0.013 (3)	0.018 (2)	0.019 (2)	0.002 (2)	0.005 (2)	0.007 (2)
C5	0.013 (3)	0.014 (2)	0.018 (2)	0.000 (2)	0.001 (2)	−0.003 (2)
C6	0.008 (2)	0.018 (2)	0.012 (2)	0.001 (2)	−0.0063 (19)	−0.0007 (19)
C7	0.016 (3)	0.016 (2)	0.013 (2)	0.004 (2)	−0.001 (2)	−0.0006 (19)
C8	0.021 (3)	0.016 (2)	0.010 (2)	0.007 (2)	0.000 (2)	0.0000 (19)
C9	0.014 (3)	0.019 (3)	0.013 (2)	0.005 (2)	−0.003 (2)	0.001 (2)
C10	0.023 (3)	0.022 (3)	0.016 (2)	−0.003 (2)	0.004 (2)	0.006 (2)
C11	0.020 (3)	0.027 (3)	0.014 (2)	−0.007 (2)	0.001 (2)	0.003 (2)

Geometric parameters (Å, °)

Br1—C4	1.912 (5)	C5—C6	1.410 (7)
O1—C1	1.367 (6)	C5—H5	0.9500
O1—C8	1.384 (5)	C6—C7	1.428 (6)
O2—C9	1.208 (6)	C7—C8	1.357 (7)
O3—C9	1.340 (5)	C7—H7	0.9500
O3—C10	1.465 (6)	C8—C9	1.464 (7)
C1—C2	1.390 (6)	C10—C11	1.505 (7)
C1—C6	1.398 (7)	C10—H10A	0.9900
C2—C3	1.382 (7)	C10—H10B	0.9900
C2—H2	0.9500	C11—H11A	0.9800
C3—C4	1.397 (7)	C11—H11B	0.9800
C3—H3	0.9500	C11—H11C	0.9800
C4—C5	1.369 (6)		
C1—O1—C8	105.3 (4)	C8—C7—H7	126.8
C9—O3—C10	116.5 (4)	C6—C7—H7	126.8
O1—C1—C2	125.2 (4)	C7—C8—O1	111.9 (4)

O1—C1—C6	110.8 (4)	C7—C8—C9	133.0 (4)
C2—C1—C6	124.0 (5)	O1—C8—C9	115.1 (4)
C3—C2—C1	116.1 (5)	O2—C9—O3	125.3 (5)
C3—C2—H2	121.9	O2—C9—C8	124.9 (4)
C1—C2—H2	121.9	O3—C9—C8	109.8 (4)
C2—C3—C4	120.6 (4)	O3—C10—C11	107.2 (4)
C2—C3—H3	119.7	O3—C10—H10A	110.3
C4—C3—H3	119.7	C11—C10—H10A	110.3
C5—C4—C3	123.4 (5)	O3—C10—H10B	110.3
C5—C4—Br1	118.2 (4)	C11—C10—H10B	110.3
C3—C4—Br1	118.4 (4)	H10A—C10—H10B	108.5
C4—C5—C6	117.1 (4)	C10—C11—H11A	109.5
C4—C5—H5	121.5	C10—C11—H11B	109.5
C6—C5—H5	121.5	H11A—C11—H11B	109.5
C1—C6—C5	118.8 (4)	C10—C11—H11C	109.5
C1—C6—C7	105.6 (4)	H11A—C11—H11C	109.5
C5—C6—C7	135.6 (5)	H11B—C11—H11C	109.5
C8—C7—C6	106.4 (4)		
C8—O1—C1—C2	−179.8 (5)	C4—C5—C6—C7	−178.1 (5)
C8—O1—C1—C6	−0.3 (5)	C1—C6—C7—C8	−1.0 (6)
O1—C1—C2—C3	177.3 (5)	C5—C6—C7—C8	177.8 (6)
C6—C1—C2—C3	−2.1 (8)	C6—C7—C8—O1	0.9 (6)
C1—C2—C3—C4	1.2 (8)	C6—C7—C8—C9	−175.5 (5)
C2—C3—C4—C5	0.7 (8)	C1—O1—C8—C7	−0.4 (6)
C2—C3—C4—Br1	−177.6 (4)	C1—O1—C8—C9	176.7 (4)
C3—C4—C5—C6	−1.6 (7)	C10—O3—C9—O2	−0.7 (7)
Br1—C4—C5—C6	176.6 (4)	C10—O3—C9—C8	178.6 (4)
O1—C1—C6—C5	−178.3 (4)	C7—C8—C9—O2	178.6 (6)
C2—C1—C6—C5	1.2 (8)	O1—C8—C9—O2	2.3 (7)
O1—C1—C6—C7	0.8 (5)	C7—C8—C9—O3	−0.7 (8)
C2—C1—C6—C7	−179.7 (5)	O1—C8—C9—O3	−177.1 (4)
C4—C5—C6—C1	0.7 (7)	C9—O3—C10—C11	−154.2 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2^{i}	0.95	2.57	3.400 (6)	146
C11—H11A···O2^{ii}	0.98	2.53	3.472 (6)	160

Symmetry codes: (i) *x*+1/2, −*y*+1/2, *z*−1/2; (ii) *x*−1/2, −*y*+1/2, *z*+1/2.

Fig. 1