



Crystal structure of 1-(5-bromo-1-benzofuran-2-yl)ethanone oxime

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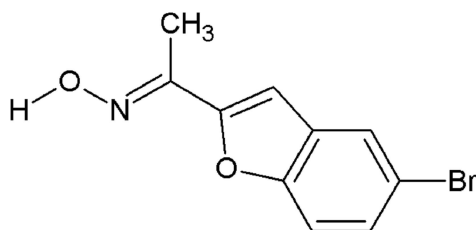
The title compound, $C_{10}H_8BrNO_2$, is almost planar (r.m.s. deviation for the non-H atoms = 0.031 Å) and the conformation across the C=N bond is *trans*. Further, the O atom of the benzofuran ring is *syn* to the N atom of the oxime group. In the crystal, inversion dimers linked by pairs of O—H...N hydrogen bonds generate $R_2^2(6)$ loops. Very weak aromatic π – π stacking interactions [centroid–centroid separations = 3.9100 (12) and 3.9447 (12) Å] are also observed.

Keywords: crystal structure; 1-(5-bromobenzofuran-2-yl) ethanone oxime; hydrogen bonding; π – π stacking interactions.

CCDC reference: 1425831

1. Related literature

For the various biological activities of the benzofuran moiety, see: Rida *et al.* (2006); Manna *et al.* (2010); Patil *et al.* (2010); Patel *et al.* (2006). For the antifungal activity of (benzofuran-2-yl) keoximes, see: Demirayak *et al.* (2002). For related structures, see: Aruna Kumar *et al.* (2014).



‡ These authors contributed equally.

2. Experimental

2.1. Crystal data

$C_{10}H_8BrNO_2$	$V = 969.91 (18) \text{ \AA}^3$
$M_r = 254.08$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 5.9548 (6) \text{ \AA}$	$\mu = 4.21 \text{ mm}^{-1}$
$b = 9.4897 (10) \text{ \AA}$	$T = 296 \text{ K}$
$c = 17.2906 (19) \text{ \AA}$	$0.32 \times 0.25 \times 0.21 \text{ mm}$
$\beta = 96.943 (6)^\circ$	

2.2. Data collection

Bruker APEXII diffractometer	10152 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2009)	2766 independent reflections
$T_{\min} = 0.294$, $T_{\max} = 0.413$	1937 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.023$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	129 parameters
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
2766 reflections	$\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O2-H2A\cdots N1^i$	0.82	2.13	2.808 (2)	140

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT-Plus (Bruker, 2009); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7505).

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S1. Chemical context

The literature indicates that compounds having benzofuran nucleus possesses versatile pharmacological activities like antifungal, antiarrhythmic, uricisuric, vasodilator and antimigraine agent (Rida *et al.*, 2006; Manna *et al.*, 2010; Patil *et al.*, 2010; Patel *et al.*, 2006). Further, (Benzofuran-2-yl) keoxime derivatives are known to show good antifungal activities (Demirayak *et al.*, 2002). In view of the above and in continuation of our efforts to study the crystal structures of benzofuran moieties (Aruna Kumar *et al.*, 2014), the title compound was synthesized and its crystal structure was determined.

S2. Structural commentary

The title compound (I), C₁₀H₈BrNO₂, is almost planar (r.m.s. deviation for the non-H atoms = 0.031 Å) and the conformation across the C=N bond is trans in (I) (Figure 1). In contrast to this, the conformation across the C=N bond is syn in (1Z)-1-(1-Benzofuran-2-yl)ethanone oxime (II) (Aruna Kumar *et al.*, 2014). Further, the O atom of the benzofuran ring is trans to the CH₃ group in the side chain of (I), where as, in (II) (Aruna Kumar *et al.*, 2014), it is syn. The torsions in the side chain of (I) have values: O1—C8—C9—N1 = 3.3 (3)°, C8—C9—N1—O2 = 179.41 (17)° and C7—C8—C9—C10 = 3.9 (4)°. The corresponding torsions in (II) have values 177.02 (16)°, 0.6 (3)° and 178.2 (2)° respectively (Aruna Kumar *et al.*, 2014).

S3. Supramolecular features

The crystal structure features strong O2—H2A···N1 hydrogen bonds leading into R₂²(6) dimers, and these dimers are further interconnected via two π ··· π interactions, namely cg1···cg1 and cg1···cg2 (where cg1 is the centroid of the furan ring C4/C5/C7/C8/O1 and cg2 is the centroid of the aryl ring C1—C6) (Figure 2, Table 2), the centroid-centroid separations being 3.9447 (12) Å and 3.9100 (12) Å respectively.

S4. Synthesis and crystallization

5-bromo-2-acetylbenzofuran (1 g, 0.0062 mmol), hydroxylamine hydrochloride (0.65 g, 0.0093 mmol) and anhydrous K₂CO₃ (1.29 g, 0.0093 mmol) were taken in EtOH: H₂O (3:1, 10 mL) and refluxed for 3 h. After the completion of the reaction, the reaction mixture was poured into ice cold water. The separated white solid was filtered, washed with water and dried. It was recrystallized from EtOH.

Colourless prisms were obtained from the solvent system: ethyl acetate: methanol (4:1) by slow evaporation technique.

S5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å and O—H = 0.82 Å. The isotropic displacement parameters for all H atoms were set to 1.2 times U_{eq}(Caromatic) and 1.5 times U_{eq}(Cmethyl, O).

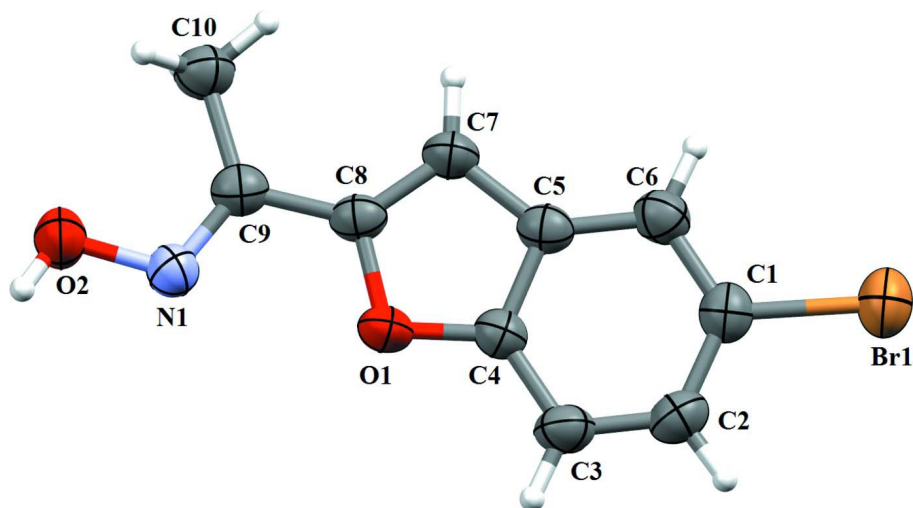


Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

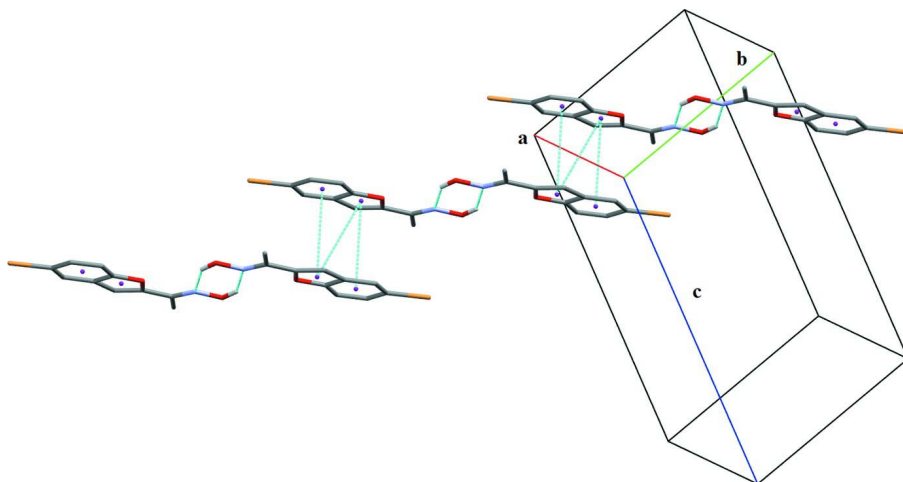


Figure 2

Crystal packing of the title compound displaying O—H...N and π – π interactions.

1-(5-Bromo-1-benzofuran-2-yl)ethanone oxime

Crystal data

$C_{10}H_8BrNO_2$

$M_r = 254.08$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 5.9548$ (6) Å

$b = 9.4897$ (10) Å

$c = 17.2906$ (19) Å

$\beta = 96.943$ (6)°

$V = 969.91$ (18) Å³

$Z = 4$

$F(000) = 504$

Prism

$D_x = 1.740$ Mg m⁻³

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

Cell parameters from 125 reflections

$\theta = 3.5$ – 29.9 °

$\mu = 4.21$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.32 \times 0.25 \times 0.21$ mm

Data collection

Bruker APEXII
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and φ scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.294$, $T_{\max} = 0.413$
10152 measured reflections

2766 independent reflections
1937 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\max} = 29.9^\circ$, $\theta_{\min} = 3.5^\circ$
 $h = -8 \rightarrow 7$
 $k = -13 \rightarrow 10$
 $l = -18 \rightarrow 24$
1 standard reflections every 2 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.096$
 $S = 1.01$
2766 reflections
129 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.0157P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C8	0.5278 (3)	-0.2686 (2)	-0.00305 (11)	0.0391 (4)
C1	0.3056 (3)	0.0480 (2)	0.16815 (12)	0.0447 (5)
C2	0.5215 (3)	0.0179 (2)	0.20434 (11)	0.0492 (5)
H2	0.5745	0.0626	0.2509	0.059*
C3	0.6569 (3)	-0.0767 (2)	0.17233 (12)	0.0492 (5)
H3	0.8014	-0.0979	0.1961	0.059*
C4	0.5691 (3)	-0.1394 (2)	0.10298 (11)	0.0390 (4)
C5	0.3543 (3)	-0.1120 (2)	0.06601 (11)	0.0403 (4)
C6	0.2169 (3)	-0.0159 (2)	0.09927 (12)	0.0470 (5)
H6	0.0714	0.0043	0.0761	0.056*
C7	0.3324 (3)	-0.1982 (2)	-0.00278 (12)	0.0437 (5)
H7	0.2065	-0.2041	-0.0401	0.052*
C9	0.6021 (3)	-0.3724 (2)	-0.05598 (11)	0.0412 (4)
C10	0.4500 (4)	-0.4102 (3)	-0.12802 (12)	0.0513 (5)
H10A	0.4955	-0.3594	-0.1716	0.077*

H10B	0.2969	-0.3859	-0.1215	0.077*
H10C	0.4598	-0.5096	-0.1373	0.077*
N1	0.7969 (3)	-0.42793 (19)	-0.03532 (10)	0.0445 (4)
O1	0.6789 (2)	-0.23533 (16)	0.06169 (8)	0.0440 (3)
O2	0.8582 (2)	-0.52765 (17)	-0.08799 (9)	0.0548 (4)
H2A	0.9720	-0.5695	-0.0688	0.082*
Br1	0.13019 (4)	0.18428 (3)	0.214769 (13)	0.05936 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C8	0.0409 (10)	0.0392 (10)	0.0356 (10)	-0.0021 (8)	-0.0023 (7)	0.0055 (8)
C1	0.0505 (11)	0.0403 (11)	0.0447 (11)	0.0004 (8)	0.0111 (8)	0.0024 (9)
C2	0.0551 (12)	0.0525 (13)	0.0388 (11)	-0.0044 (10)	0.0009 (8)	-0.0040 (9)
C3	0.0452 (10)	0.0586 (14)	0.0412 (11)	-0.0014 (9)	-0.0058 (8)	-0.0012 (10)
C4	0.0362 (9)	0.0410 (11)	0.0394 (10)	-0.0003 (8)	0.0032 (7)	0.0031 (8)
C5	0.0401 (9)	0.0391 (11)	0.0405 (10)	-0.0005 (8)	0.0002 (7)	0.0062 (8)
C6	0.0442 (10)	0.0492 (12)	0.0464 (12)	0.0043 (9)	0.0010 (8)	0.0035 (9)
C7	0.0391 (10)	0.0498 (13)	0.0403 (11)	0.0035 (8)	-0.0037 (8)	-0.0002 (9)
C9	0.0429 (10)	0.0416 (11)	0.0384 (10)	-0.0034 (8)	0.0028 (8)	0.0051 (9)
C10	0.0520 (11)	0.0552 (14)	0.0444 (12)	0.0022 (10)	-0.0030 (9)	-0.0011 (10)
N1	0.0447 (9)	0.0462 (10)	0.0420 (9)	0.0057 (7)	0.0028 (7)	-0.0024 (7)
O1	0.0379 (7)	0.0496 (8)	0.0420 (7)	0.0041 (6)	-0.0052 (5)	-0.0014 (6)
O2	0.0562 (9)	0.0591 (10)	0.0493 (8)	0.0142 (7)	0.0066 (6)	-0.0069 (7)
Br1	0.0661 (2)	0.05433 (19)	0.06028 (19)	0.00532 (9)	0.01851 (12)	-0.00638 (10)

Geometric parameters (Å, °)

C8—C7	1.343 (3)	C5—C6	1.394 (3)
C8—O1	1.385 (2)	C5—C7	1.437 (3)
C8—C9	1.449 (3)	C6—H6	0.9300
C1—C6	1.383 (3)	C7—H7	0.9300
C1—C2	1.390 (3)	C9—N1	1.285 (3)
C1—Br1	1.901 (2)	C9—C10	1.493 (3)
C2—C3	1.367 (3)	C10—H10A	0.9600
C2—H2	0.9300	C10—H10B	0.9600
C3—C4	1.383 (3)	C10—H10C	0.9600
C3—H3	0.9300	N1—O2	1.392 (2)
C4—O1	1.371 (2)	O2—H2A	0.8200
C4—C5	1.384 (2)		
C7—C8—O1	111.24 (18)	C1—C6—C5	117.40 (17)
C7—C8—C9	132.16 (16)	C1—C6—H6	121.3
O1—C8—C9	116.56 (17)	C5—C6—H6	121.3
C6—C1—C2	122.23 (19)	C8—C7—C5	107.11 (16)
C6—C1—Br1	119.44 (15)	C8—C7—H7	126.4
C2—C1—Br1	118.33 (15)	C5—C7—H7	126.4
C3—C2—C1	120.75 (18)	N1—C9—C8	115.94 (16)

C3—C2—H2	119.6	N1—C9—C10	124.7 (2)
C1—C2—H2	119.6	C8—C9—C10	119.31 (17)
C2—C3—C4	117.00 (18)	C9—C10—H10A	109.5
C2—C3—H3	121.5	C9—C10—H10B	109.5
C4—C3—H3	121.5	H10A—C10—H10B	109.5
O1—C4—C3	125.70 (16)	C9—C10—H10C	109.5
O1—C4—C5	110.89 (16)	H10A—C10—H10C	109.5
C3—C4—C5	123.4 (2)	H10B—C10—H10C	109.5
C4—C5—C6	119.22 (18)	C9—N1—O2	113.35 (16)
C4—C5—C7	105.18 (17)	C4—O1—C8	105.58 (15)
C6—C5—C7	135.61 (16)	N1—O2—H2A	109.5
C6—C1—C2—C3	0.5 (3)	C9—C8—C7—C5	178.2 (2)
Br1—C1—C2—C3	-178.43 (16)	C4—C5—C7—C8	-0.3 (2)
C1—C2—C3—C4	0.2 (3)	C6—C5—C7—C8	179.5 (2)
C2—C3—C4—O1	179.3 (2)	C7—C8—C9—N1	-174.5 (2)
C2—C3—C4—C5	-0.6 (3)	O1—C8—C9—N1	3.3 (3)
O1—C4—C5—C6	-179.65 (18)	C7—C8—C9—C10	3.9 (4)
C3—C4—C5—C6	0.3 (3)	O1—C8—C9—C10	-178.38 (19)
O1—C4—C5—C7	0.2 (2)	C8—C9—N1—O2	179.41 (17)
C3—C4—C5—C7	-179.9 (2)	C10—C9—N1—O2	1.1 (3)
C2—C1—C6—C5	-0.9 (3)	C3—C4—O1—C8	-179.9 (2)
Br1—C1—C6—C5	178.09 (15)	C5—C4—O1—C8	0.0 (2)
C4—C5—C6—C1	0.4 (3)	C7—C8—O1—C4	-0.3 (2)
C7—C5—C6—C1	-179.3 (2)	C9—C8—O1—C4	-178.48 (16)
O1—C8—C7—C5	0.4 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2A \cdots N1 ⁱ	0.82	2.13	2.808 (2)	140

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