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## Structure Reports

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## 5-Amino-4-bromo-2-benzofuran-1(3H)-one

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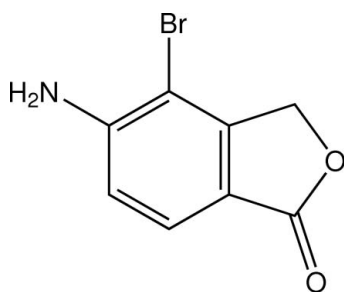
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Key indicators: single-crystal X-ray study;  $T = 153$  K; mean  $\sigma(\text{C}-\text{C}) = 0.008$  Å;  $R$  factor = 0.060;  $wR$  factor = 0.160; data-to-parameter ratio = 15.5.

Two independent but similar molecules, *A* and *B*, comprise the asymmetric unit of the title compound,  $\text{C}_8\text{H}_6\text{BrNO}_2$ . Supramolecular chains, involving one type of molecule only, are formed *via*  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds. These are connected into layers *via*  $\pi-\pi$  stacking [centroid-to-centroid separations = 3.763 (3)–3.783 (3) Å] and  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{Br}$  interactions, so that the global crystal packing comprises alternating layers of molecules *A* and *B*.

## Related literature

For related literature, see: Wang *et al.* (2007). The similarity of molecules *A* and *B* was established using *PLATON* (Spek, 2003).



## Experimental

## Crystal data

$\text{C}_8\text{H}_6\text{BrNO}_2$   
 $M_r = 228.05$   
 Monoclinic,  $P2_1/c$

$a = 14.353$  (5) Å  
 $b = 15.090$  (6) Å  
 $c = 7.5017$  (18) Å

$\beta = 93.287$  (10)°  
 $V = 1622.0$  (9) Å<sup>3</sup>  
 $Z = 8$   
 Mo  $K\alpha$  radiation

$\mu = 5.02$  mm<sup>-1</sup>  
 $T = 153$  (2) K  
 $0.35 \times 0.20 \times 0.02$  mm

## Data collection

Rigaku AFC12 $\kappa$ /SATURN724 diffractometer  
 Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)  
 $T_{\min} = 0.320$ ,  $T_{\max} = 1.000$   
 (expected range = 0.289–0.904)

19327 measured reflections  
 3356 independent reflections  
 3076 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.043$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$   
 $wR(F^2) = 0.160$   
 $S = 1.12$   
 3356 reflections

217 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 1.70$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.98$  e Å<sup>-3</sup>

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{i}}$	0.88	2.14	2.987 (7)	162
$\text{N2}-\text{H2B}\cdots\text{O4}^{\text{ii}}$	0.88	2.15	3.006 (7)	164
$\text{N1}-\text{H1B}\cdots\text{Br1}$	0.88	2.70	3.135 (6)	112
$\text{N2}-\text{H2A}\cdots\text{Br2}$	0.88	2.69	3.119 (5)	111
$\text{N2}-\text{H2A}\cdots\text{O1}^{\text{iii}}$	0.88	2.56	2.948 (7)	108
$\text{C8}-\text{H8A}\cdots\text{Br1}^{\text{iv}}$	0.99	2.92	3.895 (5)	168
$\text{C12}-\text{H12}\cdots\text{O3}^{\text{ii}}$	0.95	2.49	3.427 (7)	169

Symmetry codes: (i)  $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$ ; (ii)  $-x+2, y+\frac{1}{2}, -z-\frac{1}{2}$ ; (iii)  $x, -y+\frac{3}{2}, z-\frac{1}{2}$ ; (iv)  $x, -y+\frac{1}{2}, z+\frac{1}{2}$ .

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2505).

## References

- Altomare, A., Casciarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.  
 Brandenburg, K. (2006). *DIAMOND*. Release 3.1. Crystal Impact GbR, Bonn, Germany.  
 Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.  
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 Rigaku/MS (2005). *CrystalClear*. Rigaku/MS, The Woodlands, Texas, USA.  
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.  
 Wang, J., Johnson, D. M. & Tiekink, E. R. T. (2007). *Acta Cryst.* **E63**, o3634.

**supplementary materials**

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## 5-Amino-4-bromo-2-benzofuran-1(3H)-one

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### Comment

The title compound (I), was investigated as a part of our ongoing studies into the preparation of polyamides (Wang *et al.*, 2007) and was obtained from a two-step reduction reaction sequence to convert 4-aminophthalimide to 5-aminophthalide (see Experimental). The structure of (I) was confirmed by crystallography (Fig. 1).

Two independent molecules of (I), A and B, comprise the asymmetric unit, which are virtually identical as seen in the RMS-fit of 0.042 Å (Spek, 2003). The nine atoms comprising the framework are co-planar with a maximum deviation of 0.032 (6) Å for the C1 atom in molecule A and 0.015 (4) Å for the O1 atom in molecule B.

The crystal packing is dominated by N—H···O hydrogen bonding (Table 1) between one of the amine-H atoms and carbonyl groups leading to chains aligned along the b-direction. Each chain comprises only molecules A or only molecules B; the second amine-H atom forms an intramolecular contact with the Br atom in each case. Chains stack along the c-direction to form layers. Within layers comprising molecules of A, the connections are of the type  $\pi\cdots\pi$  with the distance between ring centroids of (C2–C7) being 3.783 (3) Å; symmetry operation:  $x, 1/2 - y, -1/2 + z$ . Further stabilization is afforded by C—H···Br contacts.

Within layers comprising molecules of B, there are also  $\pi\cdots\pi$  contacts, this time between (C10–C15) rings of 3.763 (3) Å for symmetry operation:  $x, 3/2 - y, 1/2 + z$ .

The amine-H2a atom is bifurcated in that it also forms a weak interaction to the ring-O1 atom as well as to the Br2 atom. Finally, C—H···O contacts are found in these layers further distinguishing the intermolecular connectivities operating within the layers defined by molecules of A or molecules of B. A view of the crystal packing is shown in Fig. 2.

### Experimental

In an ice bath and with stirring, aqueous NaOH (30%, 6.4 ml) and 0.2% CuSO<sub>4</sub> (4.2 ml) were added to Zn powder (6.36 g, 97.26 mmol). To this mixture was added 4-aminophthalimide (3.47 g, 21.37 mmol) over 30 min. The solid particles turned from yellow to orange, and the cloudy liquid was green. After 30 min stirring at room temperature, water (10 ml) was added and the reaction mixture was stirred at 353 K overnight. After cooling, a green-grey cloudy mixture was obtained. The precipitate was filtered off under reduced pressure. The filtrate was neutralized by 2 N HCl and further acidified with concentrated HCl (20 ml). Some solid precipitate formed when the pH was approximately 2. The mixture was heated to reflux for about 10 min, cooled to room temperature and then placed in a refrigerator. The yellow solid that precipitated was filtered off, washed with water (3 × 6 ml) and dried under vacuum at room temperature (yield 60%).

In a 100 ml round bottom flask, 5-aminophthalide (665.4 mg, 4.46 mmol) was dissolved in benzene (20 ml). After stirring for 5 min at room temperature, *N*-bromosuccinimide (886.1 mg, 49.8 mmol) and 2,2'-Azobis(2-methylpropionitrile) (AIBN) (108.6 mg, 0.66 mmol) were added to the reaction flask. The reaction mixture was heated overnight in an oil bath set at 348 K. The solvent was removed under reduced pressure. The residual was separated on a silica gel column (dry load), using

## supplementary materials

hexane and ethyl acetate (1:1 v/v;  $R_f = 0.44$ ). The product portion was collected and evaporated under reduced pressure (yield 48%). Orange needles of (I) were obtained by recrystallization from water/methanol (5:1 v/v).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  (p.p.m.): 7.62 (d, 1H, aromatic H,  $J = 8.29$  Hz), 6.81 (d, 1H, aromatic H,  $J = 8.29$  Hz), 5.09 (s, 2H,  $\alpha\text{-CH}_2$ ), 4.69 (s, 2H,  $-\text{NH}_2$ ).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  (p.p.m.): 165.18 ( $-\text{CO}-$ ), 149.34, 149.30, 125.91, 116.54, 116.12, 100.04, 69.44 ( $\alpha\text{-C}$ ). IR ( $\text{cm}^{-1}$ ) 3470 ( $-\text{NH}_2$ ), 3335 ( $-\text{NH}_2$ ), 3206, 1725 ( $-\text{CO}-$ ), 1618 ( $-\text{NH}_2$ ), 1596, 1492, 1356, 1269, 1257, 1124, 1041, 1012, 953. LCMS (APCI,  $m/z$ ) 230.1 (100), 228.5 (73), 231.1 (9).

### Refinement

All the H atoms were included in the riding-model approximation, with  $\text{C}-\text{H} = 0.95\text{--}0.99$  Å and  $\text{N}-\text{H} = 0.88$  Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ . The maximum and minimum difference peaks are located 1.30 and 0.68 Å, respectively, from Br2.

### Figures

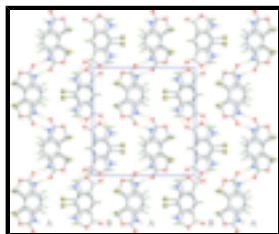
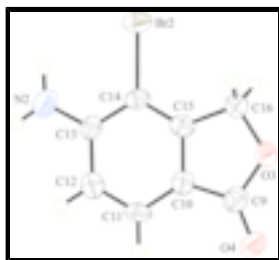
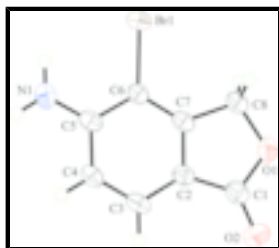


Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level (arbitrary spheres for the H atoms).

Fig. 2. View of the crystal packing in (I) highlighting the alternating layers molecules A and B. The  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds are shown as orange-dashed lines. Colour code: olive (bromine), red (oxygen), blue (nitrogen), grey (carbon) and green (hydrogen).

### 5-Amino-4-bromo-2-benzofuran-1(3H)-one

#### Crystal data

$\text{C}_8\text{H}_6\text{BrNO}_2$

$M_r = 228.05$

Monoclinic,  $P2_1/c$

$F_{000} = 896$

$D_x = 1.868$  Mg  $\text{m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71070$  Å

Hall symbol: -P 2ybc  
 $a = 14.353$  (5) Å  
 $b = 15.090$  (6) Å  
 $c = 7.5017$  (18) Å  
 $\beta = 93.287$  (10)°  
 $V = 1622.0$  (9) Å<sup>3</sup>  
 $Z = 8$

Cell parameters from 7543 reflections  
 $\theta = 2.7\text{--}30.3^\circ$   
 $\mu = 5.02$  mm<sup>-1</sup>  
 $T = 153$  (2) K  
 Plate, orange  
 $0.35 \times 0.20 \times 0.02$  mm

### Data collection

Rigaku AFC12κ/SATURN724  
 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 153$ (2) K

$\omega$  scans

Absorption correction: multi-scan  
 (ABSCOR; Higashi, 1995)

$T_{\min} = 0.320$ ,  $T_{\max} = 1.000$

19327 measured reflections

3356 independent reflections

3076 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\max} = 26.5^\circ$

$\theta_{\min} = 2.7^\circ$

$h = -13 \rightarrow 18$

$k = -18 \rightarrow 18$

$l = -8 \rightarrow 9$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.060$

$wR(F^2) = 0.160$

$S = 1.12$

3356 reflections

217 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0839P)^2 + 5.2755P]$$

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

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

$\Delta\rho_{\max} = 1.70$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.98$  e Å<sup>-3</sup>

Extinction correction: none

### 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  $F^2 > 2\sigma(F^2)$  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.

## supplementary materials

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### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.76354 (4)	0.14582 (4)	0.55865 (7)	0.0345 (2)
Br2	0.70712 (4)	0.71950 (4)	-0.02499 (8)	0.0423 (2)
O1	0.7102 (3)	0.4535 (3)	0.5012 (5)	0.0397 (9)
O2	0.5859 (3)	0.5183 (3)	0.3634 (6)	0.0498 (11)
O3	0.9376 (3)	0.5163 (2)	-0.1914 (6)	0.0428 (10)
O4	1.0792 (3)	0.5531 (3)	-0.2830 (7)	0.0494 (11)
N1	0.5726 (4)	0.0837 (3)	0.3766 (7)	0.0432 (12)
H1A	0.5214	0.0596	0.3282	0.052*
H1B	0.6183	0.0498	0.4202	0.052*
N2	0.8207 (4)	0.8909 (3)	-0.0956 (7)	0.0469 (12)
H2A	0.7636	0.8882	-0.0588	0.056*
H2B	0.8473	0.9427	-0.1108	0.056*
C1	0.6238 (4)	0.4504 (4)	0.4145 (7)	0.0372 (12)
C2	0.5946 (4)	0.3578 (3)	0.3977 (7)	0.0291 (10)
C3	0.5141 (3)	0.3199 (4)	0.3205 (7)	0.0332 (11)
H3	0.4644	0.3560	0.2730	0.040*
C4	0.5075 (4)	0.2281 (4)	0.3140 (7)	0.0331 (11)
H4	0.4525	0.2016	0.2614	0.040*
C5	0.5810 (4)	0.1726 (4)	0.3840 (7)	0.0317 (11)
C6	0.6623 (4)	0.2143 (3)	0.4641 (7)	0.0285 (10)
C7	0.6675 (3)	0.3056 (4)	0.4689 (6)	0.0300 (10)
C8	0.7448 (4)	0.3646 (4)	0.5389 (7)	0.0315 (11)
H8A	0.7575	0.3558	0.6688	0.038*
H8B	0.8027	0.3531	0.4771	0.038*
C9	1.0050 (4)	0.5772 (4)	-0.2323 (8)	0.0405 (13)
C10	0.9705 (4)	0.6654 (3)	-0.2033 (7)	0.0322 (11)
C11	1.0098 (4)	0.7477 (4)	-0.2266 (7)	0.0345 (11)
H11	1.0711	0.7528	-0.2669	0.041*
C12	0.9592 (4)	0.8218 (4)	-0.1906 (8)	0.0377 (12)
H12	0.9859	0.8785	-0.2073	0.045*
C13	0.8672 (4)	0.8159 (4)	-0.1287 (7)	0.0328 (11)
C14	0.8289 (4)	0.7314 (4)	-0.1073 (7)	0.0314 (11)
C15	0.8796 (4)	0.6578 (4)	-0.1420 (7)	0.0331 (11)
C16	0.8557 (4)	0.5614 (4)	-0.1312 (8)	0.0383 (12)
H16A	0.8438	0.5443	-0.0071	0.046*
H16B	0.7999	0.5473	-0.2096	0.046*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.0283 (3)	0.0415 (3)	0.0335 (3)	0.0073 (2)	-0.0005 (2)	0.0012 (2)
Br2	0.0261 (3)	0.0616 (4)	0.0394 (4)	0.0013 (2)	0.0024 (2)	0.0019 (2)
O1	0.038 (2)	0.034 (2)	0.046 (2)	-0.0087 (16)	0.0001 (17)	0.0015 (17)
O2	0.050 (3)	0.038 (2)	0.062 (3)	0.0083 (19)	0.000 (2)	0.008 (2)

O3	0.038 (2)	0.0272 (18)	0.063 (3)	-0.0030 (16)	0.0041 (19)	-0.0018 (18)
O4	0.036 (2)	0.042 (2)	0.070 (3)	0.0068 (18)	0.009 (2)	-0.007 (2)
N1	0.041 (3)	0.038 (3)	0.050 (3)	-0.009 (2)	-0.005 (2)	-0.004 (2)
N2	0.043 (3)	0.040 (3)	0.057 (3)	0.008 (2)	-0.002 (2)	-0.003 (2)
C1	0.031 (3)	0.044 (3)	0.037 (3)	-0.002 (2)	0.003 (2)	0.002 (2)
C2	0.025 (2)	0.034 (3)	0.029 (2)	0.0017 (19)	0.0050 (19)	0.0019 (19)
C3	0.022 (2)	0.049 (3)	0.029 (3)	0.002 (2)	0.0000 (19)	0.003 (2)
C4	0.022 (2)	0.043 (3)	0.034 (3)	-0.001 (2)	0.003 (2)	-0.004 (2)
C5	0.025 (2)	0.044 (3)	0.026 (2)	0.002 (2)	0.0027 (19)	-0.002 (2)
C6	0.024 (2)	0.034 (3)	0.027 (2)	-0.0004 (19)	0.0029 (19)	0.0002 (19)
C7	0.023 (2)	0.041 (3)	0.026 (2)	0.000 (2)	0.0027 (18)	-0.001 (2)
C8	0.026 (3)	0.040 (3)	0.028 (3)	-0.002 (2)	0.0022 (19)	0.000 (2)
C9	0.036 (3)	0.041 (3)	0.044 (3)	0.004 (2)	-0.001 (2)	0.000 (2)
C10	0.032 (3)	0.031 (2)	0.032 (3)	0.000 (2)	-0.004 (2)	-0.002 (2)
C11	0.021 (2)	0.050 (3)	0.033 (3)	-0.007 (2)	0.001 (2)	0.004 (2)
C12	0.041 (3)	0.035 (3)	0.036 (3)	-0.002 (2)	-0.003 (2)	0.001 (2)
C13	0.029 (3)	0.035 (3)	0.033 (3)	0.004 (2)	-0.006 (2)	0.002 (2)
C14	0.025 (3)	0.042 (3)	0.027 (3)	0.003 (2)	-0.0002 (19)	0.000 (2)
C15	0.029 (3)	0.039 (3)	0.031 (3)	-0.005 (2)	-0.003 (2)	0.000 (2)
C16	0.034 (3)	0.034 (3)	0.047 (3)	-0.003 (2)	0.003 (2)	0.002 (2)

*Geometric parameters (Å, °)*

Br1—C6	1.888 (5)	C4—C5	1.422 (7)
Br2—C14	1.895 (5)	C4—H4	0.9500
O1—C1	1.367 (7)	C5—C6	1.427 (7)
O1—C8	1.452 (7)	C6—C7	1.379 (7)
O2—C1	1.212 (7)	C7—C8	1.495 (7)
O3—C9	1.381 (7)	C8—H8A	0.9900
O3—C16	1.453 (7)	C8—H8B	0.9900
O4—C9	1.208 (7)	C9—C10	1.441 (8)
N1—C5	1.348 (7)	C10—C11	1.379 (7)
N1—H1A	0.8800	C10—C15	1.412 (8)
N1—H1B	0.8800	C11—C12	1.369 (8)
N2—C13	1.344 (7)	C11—H11	0.9500
N2—H2A	0.8800	C12—C13	1.427 (8)
N2—H2B	0.8800	C12—H12	0.9500
C1—C2	1.463 (7)	C13—C14	1.401 (8)
C2—C3	1.385 (7)	C14—C15	1.361 (8)
C2—C7	1.391 (7)	C15—C16	1.497 (7)
C3—C4	1.390 (8)	C16—H16A	0.9900
C3—H3	0.9500	C16—H16B	0.9900
C1—O1—C8	110.6 (4)	C7—C8—H8A	110.9
C9—O3—C16	110.2 (4)	O1—C8—H8B	110.9
C5—N1—H1A	120.0	C7—C8—H8B	110.9
C5—N1—H1B	120.0	H8A—C8—H8B	109.0
H1A—N1—H1B	120.0	O4—C9—O3	120.7 (5)
C13—N2—H2A	120.0	O4—C9—C10	130.0 (6)
C13—N2—H2B	120.0	O3—C9—C10	109.3 (5)

## supplementary materials

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H2A—N2—H2B	120.0	C11—C10—C15	120.4 (5)
O2—C1—O1	120.0 (5)	C11—C10—C9	131.7 (5)
O2—C1—C2	131.2 (5)	C15—C10—C9	107.8 (5)
O1—C1—C2	108.8 (5)	C12—C11—C10	119.0 (5)
C3—C2—C7	121.2 (5)	C12—C11—H11	120.5
C3—C2—C1	131.2 (5)	C10—C11—H11	120.5
C7—C2—C1	107.6 (5)	C11—C12—C13	121.7 (5)
C2—C3—C4	118.6 (5)	C11—C12—H12	119.2
C2—C3—H3	120.7	C13—C12—H12	119.2
C4—C3—H3	120.7	N2—C13—C14	122.9 (5)
C3—C4—C5	121.7 (5)	N2—C13—C12	119.1 (5)
C3—C4—H4	119.1	C14—C13—C12	118.0 (5)
C5—C4—H4	119.1	C15—C14—C13	120.2 (5)
N1—C5—C4	120.5 (5)	C15—C14—Br2	119.8 (4)
N1—C5—C6	121.7 (5)	C13—C14—Br2	119.9 (4)
C4—C5—C6	117.8 (5)	C14—C15—C10	120.6 (5)
C7—C6—C5	119.5 (5)	C14—C15—C16	131.0 (5)
C7—C6—Br1	119.8 (4)	C10—C15—C16	108.3 (5)
C5—C6—Br1	120.6 (4)	O3—C16—C15	104.3 (4)
C6—C7—C2	121.1 (5)	O3—C16—H16A	110.9
C6—C7—C8	130.0 (5)	C15—C16—H16A	110.9
C2—C7—C8	108.9 (5)	O3—C16—H16B	110.9
O1—C8—C7	104.1 (4)	C15—C16—H16B	110.9
O1—C8—H8A	110.9	H16A—C16—H16B	108.9
C8—O1—C1—O2	-176.0 (5)	C16—O3—C9—O4	179.1 (6)
C8—O1—C1—C2	2.7 (6)	C16—O3—C9—C10	-0.7 (6)
O2—C1—C2—C3	-1.1 (11)	O4—C9—C10—C11	1.2 (11)
O1—C1—C2—C3	-179.6 (5)	O3—C9—C10—C11	-179.0 (6)
O2—C1—C2—C7	175.7 (6)	O4—C9—C10—C15	-179.5 (6)
O1—C1—C2—C7	-2.8 (6)	O3—C9—C10—C15	0.3 (6)
C7—C2—C3—C4	-0.5 (8)	C15—C10—C11—C12	-0.5 (8)
C1—C2—C3—C4	175.9 (5)	C9—C10—C11—C12	178.8 (6)
C2—C3—C4—C5	-0.1 (8)	C10—C11—C12—C13	0.4 (8)
C3—C4—C5—N1	179.7 (5)	C11—C12—C13—N2	-179.9 (5)
C3—C4—C5—C6	0.7 (7)	C11—C12—C13—C14	-0.8 (8)
N1—C5—C6—C7	-179.8 (5)	N2—C13—C14—C15	-179.7 (5)
C4—C5—C6—C7	-0.8 (7)	C12—C13—C14—C15	1.1 (8)
N1—C5—C6—Br1	1.6 (7)	N2—C13—C14—Br2	-1.0 (7)
C4—C5—C6—Br1	-179.5 (4)	C12—C13—C14—Br2	179.9 (4)
C5—C6—C7—C2	0.3 (7)	C13—C14—C15—C10	-1.2 (8)
Br1—C6—C7—C2	179.0 (4)	Br2—C14—C15—C10	-180.0 (4)
C5—C6—C7—C8	-177.9 (5)	C13—C14—C15—C16	-179.7 (5)
Br1—C6—C7—C8	0.7 (7)	Br2—C14—C15—C16	1.6 (8)
C3—C2—C7—C6	0.4 (8)	C11—C10—C15—C14	0.9 (8)
C1—C2—C7—C6	-176.8 (5)	C9—C10—C15—C14	-178.6 (5)
C3—C2—C7—C8	179.0 (5)	C11—C10—C15—C16	179.6 (5)
C1—C2—C7—C8	1.8 (6)	C9—C10—C15—C16	0.2 (6)
C1—O1—C8—C7	-1.5 (5)	C9—O3—C16—C15	0.8 (6)
C6—C7—C8—O1	178.2 (5)	C14—C15—C16—O3	178.0 (5)



C2—C7—C8—O1

−0.2 (5)

C10—C15—C16—O3

−0.6 (6)

*Hydrogen-bond geometry* (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O2 <sup>i</sup>	0.88	2.14	2.987 (7)	162
N2—H2B···O4 <sup>ii</sup>	0.88	2.15	3.006 (7)	164
N1—H1B···Br1	0.88	2.70	3.135 (6)	112
N2—H2A···Br2	0.88	2.69	3.119 (5)	111
N2—H2A···O1 <sup>iii</sup>	0.88	2.56	2.948 (7)	108
C8—H8A···Br1 <sup>iv</sup>	0.99	2.92	3.895 (5)	168
C12—H12···O3 <sup>ii</sup>	0.95	2.49	3.427 (7)	169

Symmetry codes: (i)  $-x+1, y-1/2, -z+1/2$ ; (ii)  $-x+2, y+1/2, -z-1/2$ ; (iii)  $x, -y+3/2, z-1/2$ ; (iv)  $x, -y+1/2, z+1/2$ .

Fig. 1

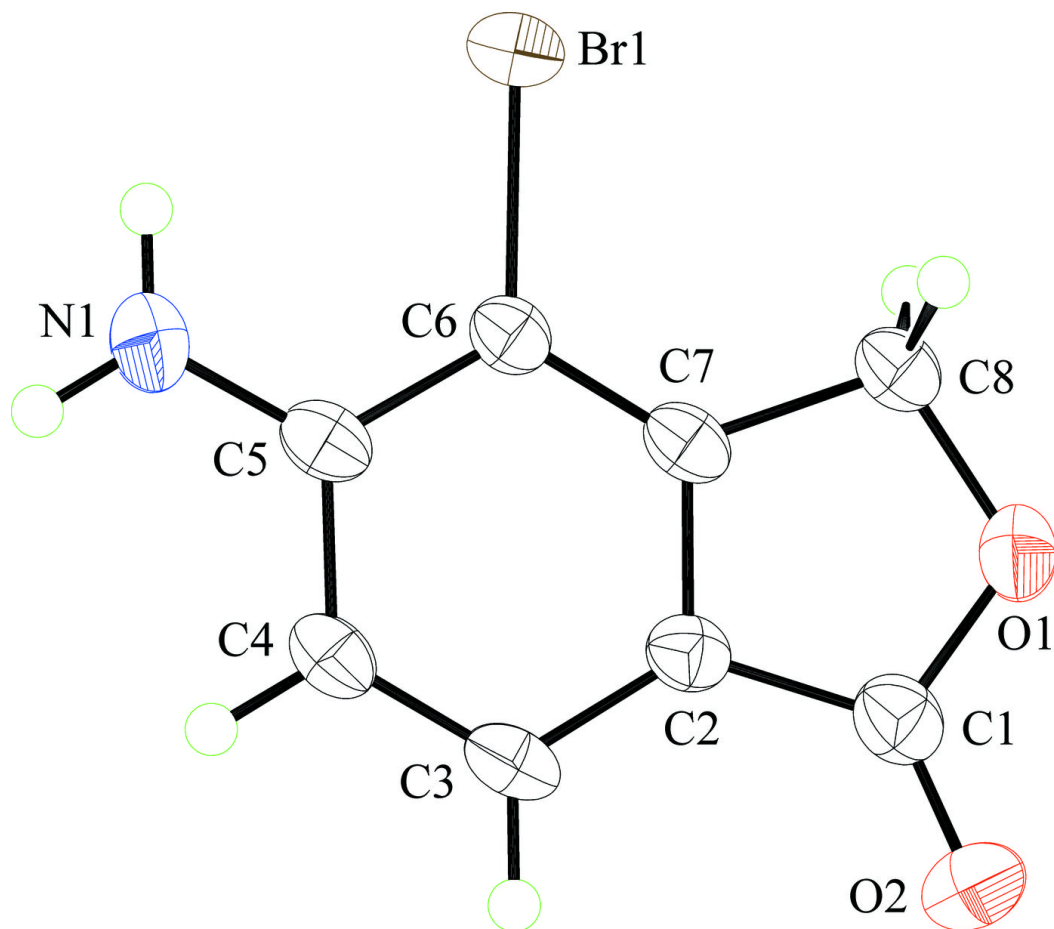


Fig. 2

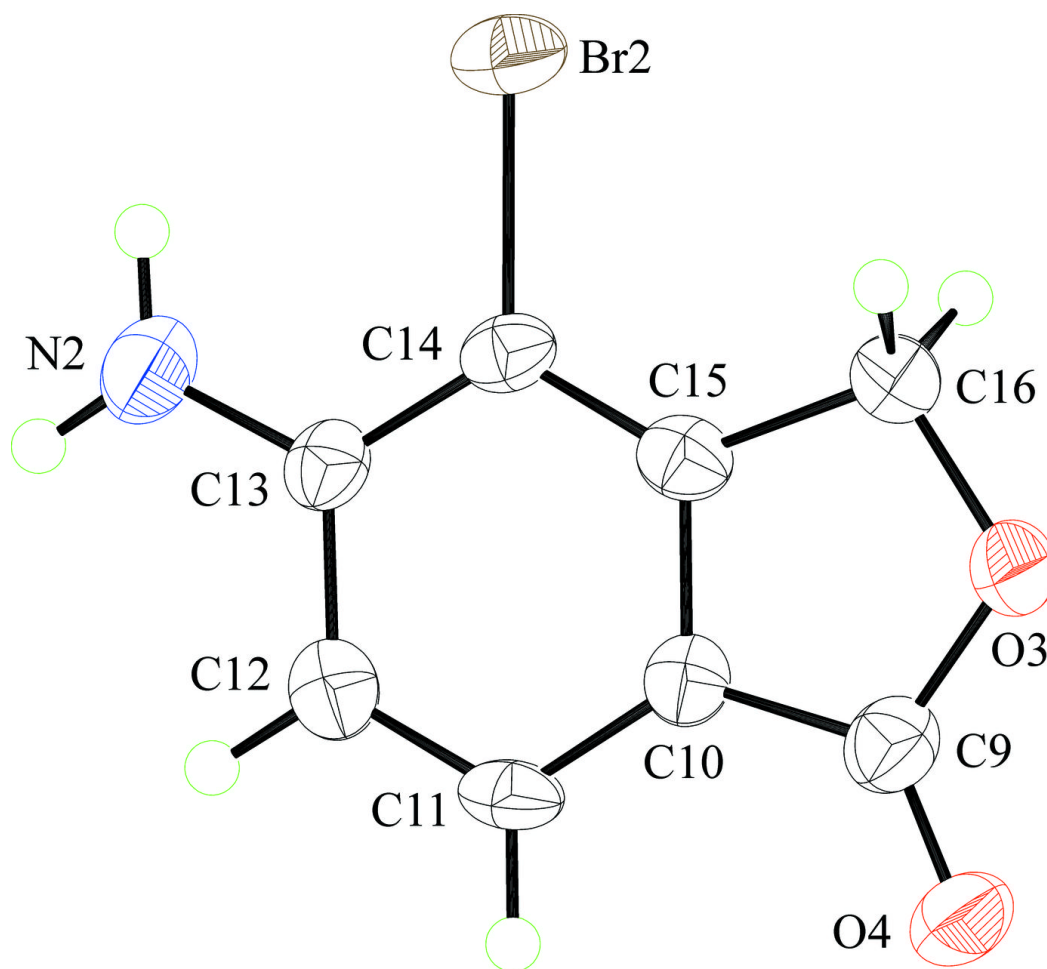


Fig. 3

