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5-Bromo-3*H*-isobenzofuran-1-one (5-bromophthalide)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.008 Å
R factor = 0.049
wR factor = 0.122
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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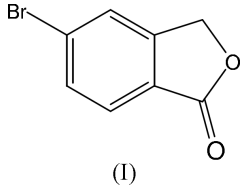
The title compound, $C_8H_5BrO_2$, serves as a starting material for the synthesis of citalopram. It crystallizes with two almost identical molecules in the asymmetric unit.

Comment

In a separate paper, we have reported the synthesis and crystal structure of 5-amino-3*H*-isobenzofuran-1-one or 5-aminophthalide (Yathirajan *et al.*, 2005). In the present paper, we report the structure of 5-bromophthalide, which crystallizes with two almost identical molecules in the asymmetric unit. A perspective view of the title compound, (I), is shown in Fig. 1. The packing diagram (Fig. 2) might imply that the two molecules in the asymmetric unit are related by a translation operator (0.2488, −0.0054, −0.0052), but none could be found fulfilling the space-group symmetry. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002). They agree with the values determined for *o*-phthalaldehyde (Majeed *et al.*, 1998; Mendenhall *et al.*, 2003), 6-nitrophthalide (Bradley *et al.*, 1997), 3-hydroxyphthalide (Khoo & Hazell, 1999) and 5-aminophthalide (Yathirajan *et al.*, 2005). In each molecule, all non-H atoms are coplanar (r.m.s. deviations = 0.025 and 0.011 Å for the two molecules in the asymmetric unit).



(I)

Experimental

5-Amino-3*H*-isobenzofuran-1-one (1.49 g, 10 mmol) was diazotized with $NaNO_2$ (0.828 g, 12 mmol) and concentrated HCl (10 ml) to yield the diazonium salt. This was further treated with CuBr (1.71 g, 12 mmol) in aqueous HBr (5 ml) to give the title compound, which was recrystallized from acetonitrile (m.p. 433–436 K) (Bigler *et al.*, 1977).

Crystal data

$C_8H_5BrO_2$
M_r = 213.03
Monoclinic, $P2_1/n$
a = 15.4063 (19) Å
b = 6.0861 (6) Å
c = 15.4426 (16) Å
β = 93.950 (9)°
V = 1444.5 (3) Å^3
Z = 8
D_x = 1.959 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 076 reflections
θ = 3.4–25.7°
μ = 5.63 mm^{-1}
T = 173 (2) K
Plate, colourless
0.31 × 0.27 × 0.08 mm

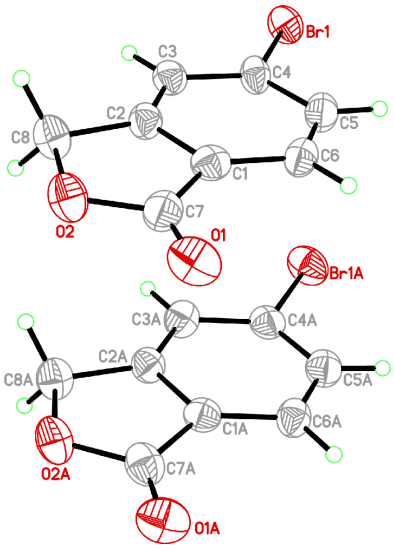


Figure 1
Perspective view of the asymmetric unit of the title compound with the atom numbering; displacement ellipsoids are shown at the 50% probability level.

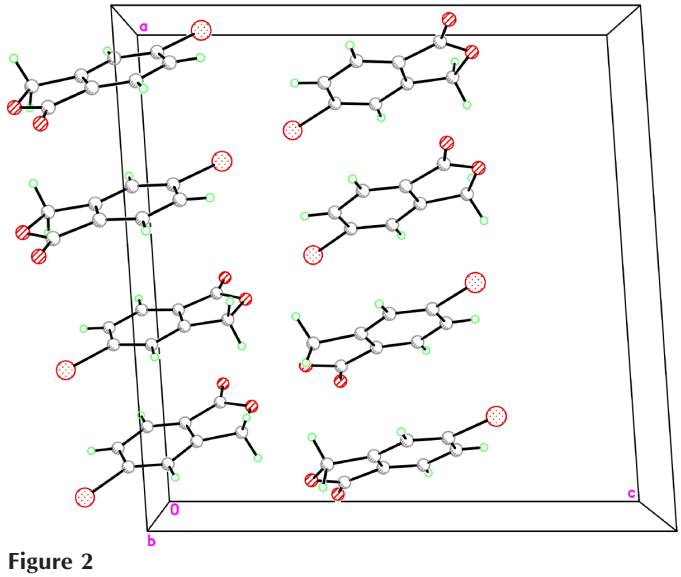


Figure 2
Packing diagram of the title compound, projected on to the *ac* plane.

Data collection

Stoe IPDS-II two-circle diffractometer	2707 independent reflections
ω scans	1990 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)	$R_{int} = 0.092$
$T_{min} = 0.187$, $T_{max} = 0.635$	$\theta_{max} = 25.6°$
17 904 measured reflections	$h = -18 \rightarrow 18$
	$k = -7 \rightarrow 7$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.049$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.122$	$\Delta\rho_{max} = 1.03$ e Å^{-3}
$S = 0.97$	$\Delta\rho_{min} = -0.71$ e Å^{-3}
2707 reflections	Extinction correction: *SHELXL*97
200 parameters	Extinction coefficient: 0.0027 (7)
H-atom parameters constrained	

H atoms were positioned geometrically and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C)] using a riding model, with C—H = 0.99 and 0.95 Å for methylene and aromatic CH groups, respectively. The highest peak in the final difference electron-density map is situated 1.07 Å from atom O1A.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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