



Crystal structures of 6-chloroindan-1-one and 6-bromoindan-1-one exhibit different intermolecular packing interactions

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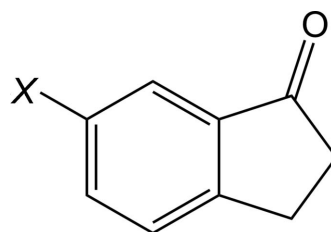
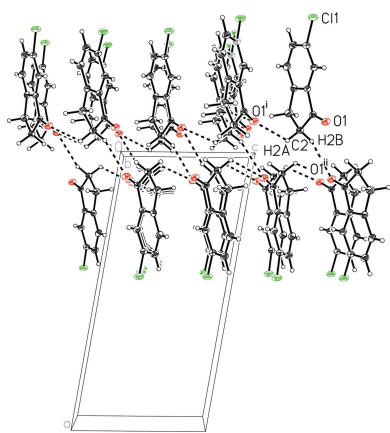
Edited by J. Simpson, University of Otago, New Zealand

Keywords: crystal structure; haloindanones; π -stacking; C—H \cdots X interactions.**CCDC references:** 1507437; 1507436**Supporting information:** this article has supporting information at journals.iucr.org/e

The two title compounds are analogs of 1-indanone that are substituted at the 6-position with chlorine and bromine. Although very similar in molecular structure, the crystal structures are not isomorphous and reveal that 6-chloroindan-1-one, C₉H₇ClO (I), and 6-bromoindan-1-one, C₉H₇BrO (II), exhibit unique intermolecular packing motifs. The molecules of the chloro analog (I) pack with a herringbone packing motif of C—H \cdots O interactions, whereas the bromo derivative (II) packs with offset face-to-face π -stacking, C—H \cdots O, C—H \cdots Br and Br \cdots O interactions. Compound (II) was refined as a two-component non-merohedral twin, BASF 0.0762 (5).

1. Chemical context

Halogenated derivatives of the common bicyclic organic framework 1-indanone have been shown to be useful in a variety of synthetic and biologically related applications (Ruiz *et al.*, 2004). A search of the Cambridge Structural Database (Version 5.31, September 2016 with updates; Groom *et al.*, 2016) returns four simple arylhalide substituted 1-indanones, although several more are commercially available. The title compounds represent two analogs of 6-haloindan-1-one that are notably not isomorphous. In addition, they are not isomorphous with the fluorine derivative 6-fluoroindan-1-one, which is one of the four that has previously been reported (Slaw & Tanski, 2014). In the chloro analog, 6-chloroindan-1-one (I), the molecules pack together *via* a series of C—H \cdots O interactions. C—H \cdots X interactions are common and have been discussed in the literature (Desiraju & Steiner, 1999), as well as specifically in the case of 1-indanone itself (Ruiz *et al.*, 2004). The bromo derivative 6-bromoindan-1-one (II) packs with offset face-to-face π -stacking (Hunter & Saunders, 1990; Lueckheide *et al.*, 2013) and several different intermolecular contacts including C—H \cdots O, C—H \cdots Br weak hydrogen bonds and Br \cdots O interactions.



(I) X = Cl

(II) X = Br

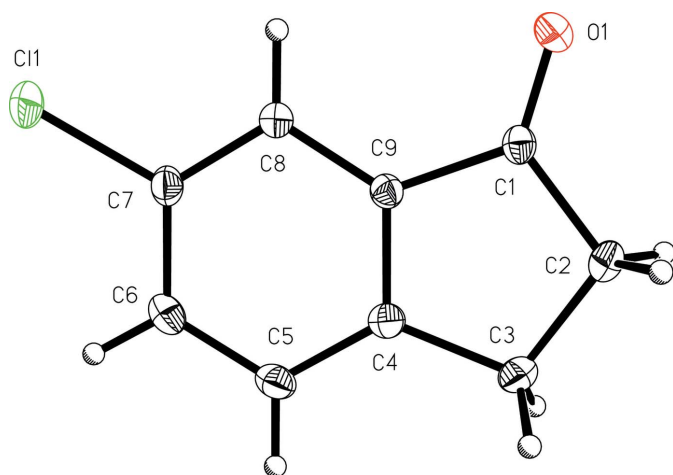


Figure 1
A view of 6-chloroindan-1-one (I) with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

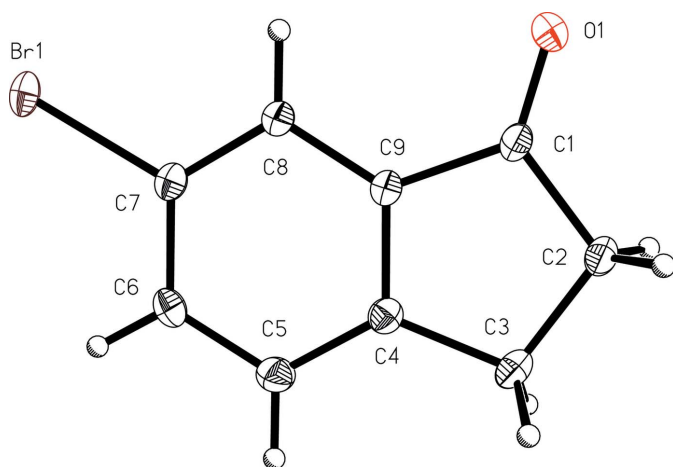


Figure 2
A view of 6-bromoindan-1-one (II) with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

The compounds 6-chloroindan-1-one (I) and 6-bromoindan-1-one (II) may be synthesized by the microwave or ultrasound-aided ring closure of 4-chloro- or 4-bromobenzenepropanoic acid, respectively, catalyzed by triflic acid in dichloromethane (Oliverio *et al.*, 2014). 6-Haloindan-1-ones have featured in the synthesis of biologically or pharmacologically active compounds. In recent examples, 6-chloroindan-1-one (I) has been employed in the total synthesis of the anticancer natural product chartarin (Unzner *et al.*, 2016), and in the synthesis of triazole-quinoline derivatives that are acetylcholinesterase inhibitors relevant to the treatment of Alzheimer's disease (Mantoani *et al.*, 2016). 6-Bromoindan-1-one has been used as the starting material for the synthesis of small molecules that inhibit cell entry by HIV-1 (Melillo *et al.*, 2016), and both 6-chloroindan-1-one and 6-bromoindan-1-one have been used as the starting material for the preparation of C-7 substituted 3,4-dihydroisoquinolin-1(2*H*)-one analogues that selectively inhibit unique poly-ADP-ribose polymerases (Morgan *et al.*, 2015).

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

<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>
C2—H2 <i>A</i> ···O1 ⁱ	0.99	2.56	3.1933 (15)	121
C2—H2 <i>B</i> ···O1 ⁱⁱ	0.99	2.59	3.5448 (14)	161

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

<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>
C3—H3 <i>B</i> ···O1 ⁱ	0.99	2.45	3.408 (4)	162
C5—H5 <i>A</i> ···O1 ⁱⁱ	0.95	2.55	3.253 (4)	131
C2—H2 <i>B</i> ···Br1 ⁱⁱⁱ	0.99	3.05	3.898 (3)	145

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

2. Structural commentary

The molecular features of 6-chloroindan-1-one (I) (Fig. 1) and 6-bromoindan-1-one (II) (Fig. 2) are similar to those reported for the analogous structure 6-fluoroindan-1-one (Slaw & Tanski, 2014), although the analogues are not isomorphous and exhibit different intermolecular packing. In the chloro derivative (I), the aryl C—Cl bond length, 1.7435 (11) Å, is similar to that found in the isomeric compound 5-chloroindan-1-one [C—Cl = 1.735 (2) Å; Ruiz *et al.*, 2006]. The aryl C—Br bond length in the bromo analog (II), 1.907 (3) Å, is similar to that found in the isomeric compound 4-bromoindan-1-one [1.894 (1) Å; Aldeborgh *et al.*, 2014]. The C=O bond lengths in 6-chloroindan-1-one (I), 1.2200 (12) Å, and 6-bromoindan-1-one (II), 1.216 (3) Å, are also very similar to those found in the other four reported structures of simple arylhalide-substituted 1-indanones: 6-fluoroindan-1-one, 1.2172 (13) Å (Slaw & Tanski, 2014); 5-fluoroindan-1-one, 1.218 (2) Å (Garcia *et al.*, 1995); 5-chloroindan-1-one, 1.210 (3) Å (Ruiz *et al.*, 2006); 4-bromoindan-1-one, 1.215 (2) Å (Aldeborgh *et al.*, 2014). These carbonyl C=O bond lengths are also similar to that found in the structure of the parent compound, 1-indanone, 1.217 (2) Å (Ruiz *et al.*, 2004). With the exception of the methylene hydrogen atoms, both (I) and (II) are nearly planar, with r.m.s. deviations from the mean planes of all non-H atoms of 0.0460 and 0.0107 Å, respectively.

3. Supramolecular features

In the crystal structure of 6-chloroindan-1-one (I), the molecules pack together *via* van der Waals contacts, specifically C—H···O interactions, without any π -stacking. The C—H···O interactions (Fig. 3 and Table 1) connect the indanone oxygen atom with methylene hydrogen atoms on neighboring molecules into a two-molecule-thick sheet parallel to the (100) plane (Fig. 4). These sheets further pack together without any notable intermolecular contacts. The closest Cl···Cl contact between the sheets, 3.728 Å, is somewhat longer than the sum of the van der Waals radii of chlorine, 3.50 Å (Bondi, 1964).

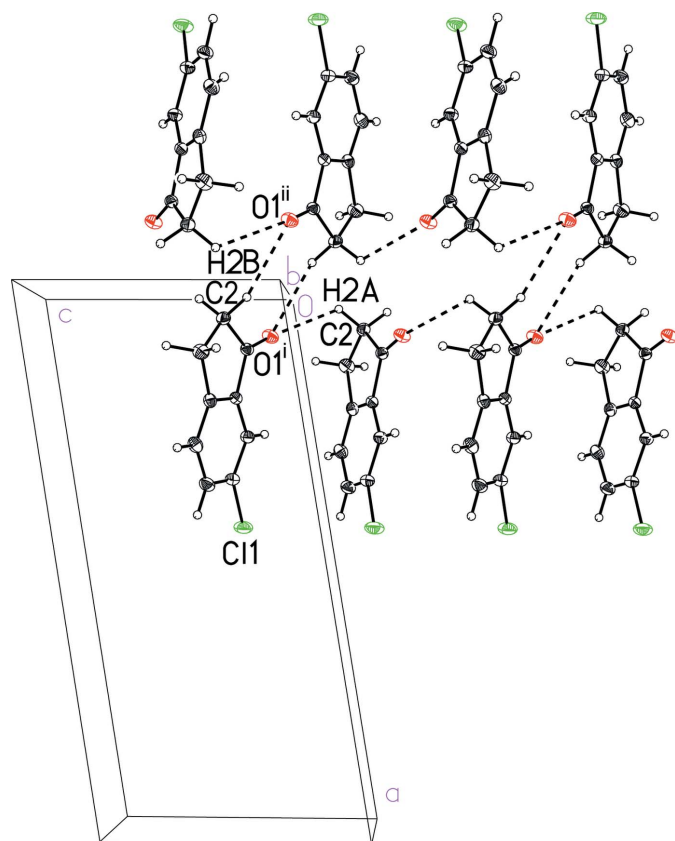


Figure 3
A view of the intermolecular C–H···O contacts in 6-chloroindan-1-one (I). See Table 1 for symmetry codes (i) and (ii). In this and subsequent figures the C–H···X interactions are shown as dashed lines.

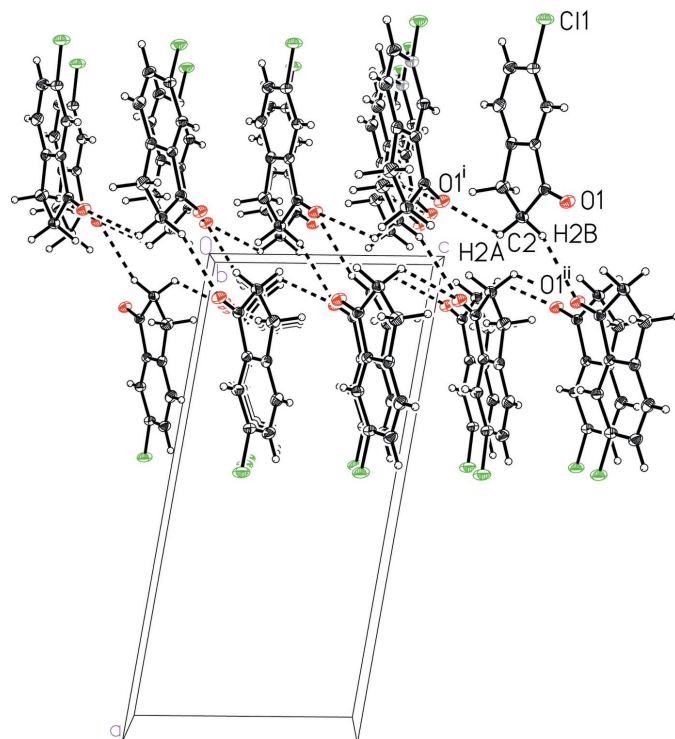


Figure 4
A view of the sheet structure in 6-chloroindan-1-one (I) formed by C–H···O contacts. See Table 1 for symmetry codes (i) and (ii).

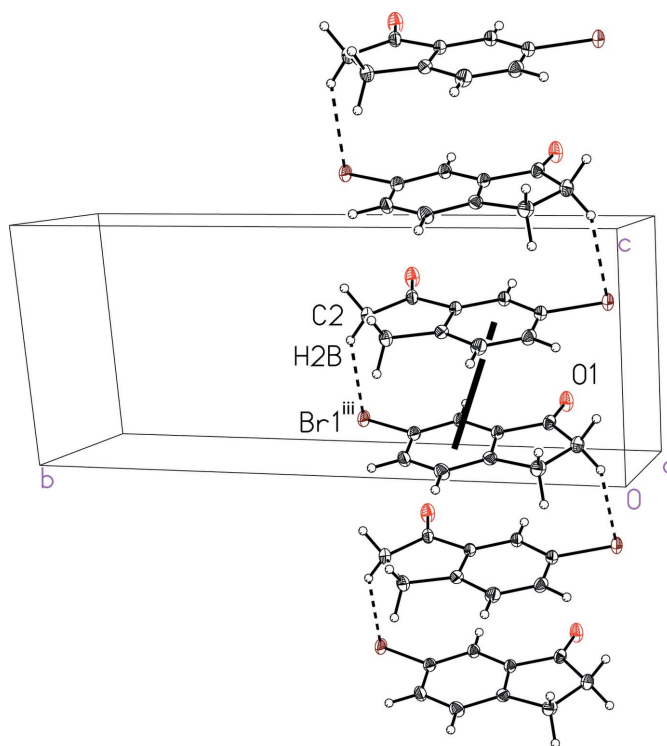


Figure 5
A view of the alternating offset face-to-face π -stacking and C–H···Br interaction in 6-bromoindan-1-one (II) with the thick black line indicating a centroid-to-centroid interaction. See Table 2 for symmetry code (iii).

The molecular packing in the bromo analog, 6-bromoindan-1-one (II), is distinct from that found in (I). The notable intermolecular interactions observed include π -stacking, Br···O, C–H···O, and C–H···Br interactions. The offset face-to-face π -stacking can be seen to extend along the crystallographic *c* axis (Fig. 5), with the molecules stacking in an alternating head-to-tail fashion featuring a C–H···Br interaction with an H···Br distance of 3.05 Å (Fig. 5 and Table 2). The π -stacking is characterized by a centroid-to-centroid distance of 3.850 (3) Å, centroid-to-plane distances of 3.530 (2) and 3.603 (2) Å, and ring offsets of 1.358 (3) and 1.536 (3) Å that result in a plane-to-plane angle of 3.1 (1)°. The π -stacked chains of (II) are linked into a three-dimensional lattice by C–H···O interactions and a Br···O contact (Fig. 6 and Table 2). The Br···O contact, at a distance of 3.018 (2) Å, is slightly shorter than the sum of the van der Waals radii, 3.37 Å (Bondi 1964). This interaction is even shorter than the Br···O contact in the isomeric 4-bromoindan-1-one [3.129 (1) Å; Aldeborgh *et al.*, 2014].

4. Database survey

A survey of the Cambridge Structural Database reveals that in addition to the two structures reported here, there are four other simple arylhalide-substituted 1-indanone structures known. These include 6-fluoroindan-1-one (Slaw & Tanski, 2014), 5-fluoroindan-1-one (Garcia *et al.*, 1995), 5-chloro-

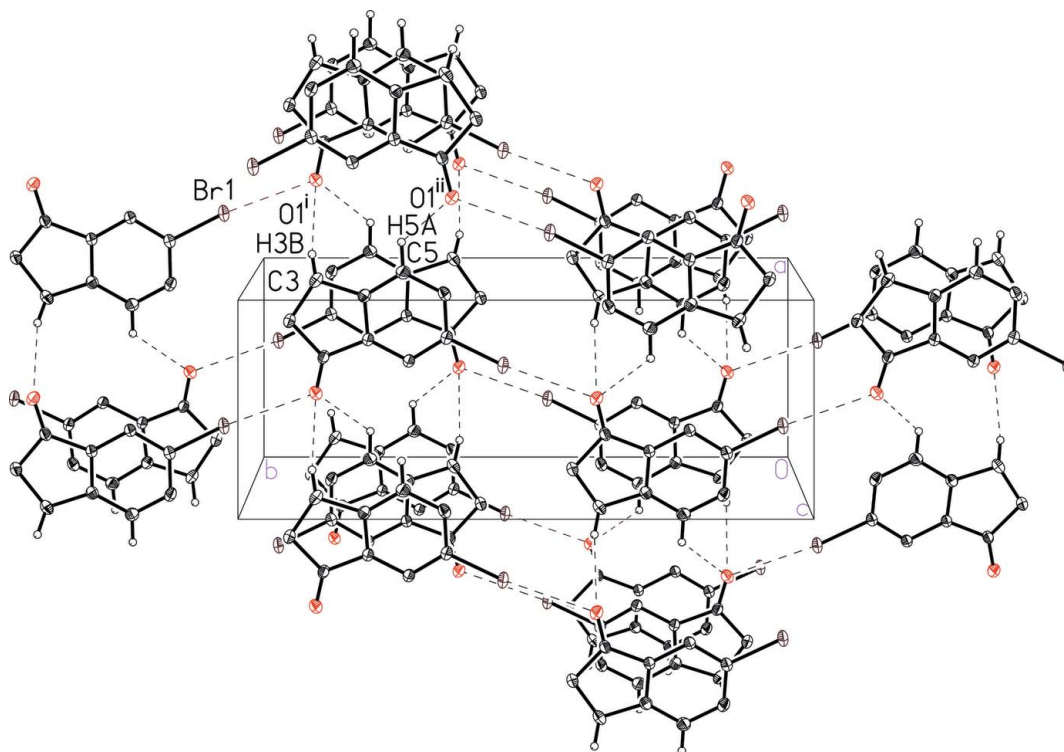


Figure 6
A view of the intermolecular C—H...O and Br...O contacts (dashed lines) in 6-bromoindan-1-one (II). See Table 2 for symmetry codes (i) and (ii).

indan-1-one (Ruiz *et al.*, 2006) and 4-bromoindan-1-one (Aldeborgh *et al.*, 2014). The crystal structure of 1-indanone itself was first reported in 1974 (Morin *et al.*, 1974) and was later described in a more detailed structural and spectroscopic analysis (Ruiz *et al.*, 2004).

5. Synthesis and crystallization

6-Chloroindan-1-one (96%) and 6-bromoindan-1-one (98%) were purchased from Aldrich Chemical Company, USA, and were used as received.

6. Analytical data

6-Chloroindan-1-one (I): ^1H NMR (Bruker Avance 300 MHz, CDCl_3): δ 2.72 (*t*, 2 H, $J = 5.9$ Hz, CH_2), 3.12 (*t*, 2H, $J = 5.9$ Hz, CH_2), 7.42 (*d*, 1 H, $J_{ortho} = 8.2$ Hz, C_{aryl}H), 7.53 (*dd*, 1H, $J_{meta} = 1.6$ Hz, $J_{ortho} = 8.1$ Hz, C_{aryl}H), 7.69 (*s*, 1 H, C_{aryl}H). ^{13}C NMR ($^{13}\text{C}\{^1\text{H}\}$, 75.5 MHz, CDCl_3): δ 25.37 (CH_2), 36.57 (CH_2), 123.45 (C_{aryl}H), 127.85 (C_{aryl}H), 133.63 (C_{aryl}), 134.50 (C_{aryl}H), 138.49 (C_{aryl}), 153.07 (C_{aryl}), 205.43 ($\text{C}=\text{O}$). IR (Thermo Nicolet iS50, KBr pellet, cm^{-1}): 3391 (*w*), 3076 (*w*), 3051 (*w*), 2964 (*w*), 2935 (*w*), 1702 (*vs*, $\text{C}=\text{O}$ *str*), 1595 (*w*), 1576 (*w*), 1466 (*m*), 1435 (*m*), 1409 (*m*), 1318 (*w*), 1285 (*w*), 1276 (*w*), 1250 (*m*), 1214 (*w*), 1187 (*m*), 1173 (*m*), 1115 (*m*), 1037 (*w*), 895 (*m*), 854 (*m*), 836 (*s*), 815 (*m*), 678 (*m*), 623 (*m*), 561 (*m*), 518 (*w*), 484 (*m*). GC/MS (Hewlett-Packard MS 5975/GC 7890): M^+ = 166 (calculated exact mass 166.02).

6-Bromoindan-1-one (II): ^1H NMR (Bruker Avance 300 MHz, CDCl_3): δ 2.71 (*t*, 2 H, $J = 5.8$ Hz, CH_2), 3.09 (*t*, 2H, $J = 5.9$ Hz, CH_2), 7.37 (*d*, 1 H, $J_{ortho} = 8.1$ Hz, C_{aryl}H), 7.65 (*dd*,

1H, $J_{meta} = 1.9$ Hz, $J_{ortho} = 8.1$ Hz, C_{aryl}H), 7.83 (*s*, 1 H, C_{aryl}H). ^{13}C NMR ($^{13}\text{C}\{^1\text{H}\}$, 75.5 MHz, CDCl_3): δ 25.37 (CH_2), 36.34 (CH_2), 121.35 (C_{aryl}), 126.46 (C_{aryl}H), 128.16 (C_{aryl}H), 137.14 (C_{aryl}H), 138.73 (C_{aryl}), 153.47 (C_{aryl}), 205.19 ($\text{C}=\text{O}$). IR (Thermo Nicolet iS50, ATR, cm^{-1}): 3394 (*w*), 3066 (*w*), 2962 (*w*), 2925 (*w*), 1698 (*vs*, $\text{C}=\text{O}$ *str*), 1598 (*w*), 1577 (*w*), 1468 (*w*), 1438 (*s*), 1417 (*w*), 1398 (*m*), 1322 (*w*), 1295 (*w*), 1279 (*w*), 1253 (*m*), 1238 (*m*), 1213 (*w*), 1191 (*s*), 1171 (*w*), 1112 (*m*), 1038 (*w*), 978 (*w*), 887 (*w*), 829 (*s*), 668 (*m*), 609 (*w*), 557 (*m*), 509 (*w*), 478 (*m*). GC/MS (Hewlett-Packard MS 5975/GC 7890): M^+ = 210 (calculated exact mass 209.97).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. After indexing with *Cell_Now* (Sheldrick, 2008), 6-bromoindan-1-one (II) was refined as a two-component non-merohedral twin, BASF 0.0762 (5). Carbon-bound hydrogen atoms were included in calculated positions and refined using a riding model at $\text{C}-\text{H} = 0.95$ and 0.99 Å and $U_{iso}(\text{H}) = 1.2U_{eq}(\text{C})$ of the aryl and methylene C atoms, respectively.

Acknowledgements

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Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C ₉ H ₇ ClO	C ₉ H ₇ BrO
<i>M_r</i>	166.60	211.06
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	125	125
<i>a</i> , <i>b</i> , <i>c</i> (Å)	16.319 (6), 6.024 (2), 7.745 (3)	6.489 (2), 17.101 (6), 7.224 (3)
β (°)	99.524 (5)	102.964 (5)
<i>V</i> (Å ³)	750.9 (5)	781.2 (5)
<i>Z</i>	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.44	5.19
Crystal size (mm)	0.28 × 0.25 × 0.14	0.40 × 0.21 × 0.05
Data collection		
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2013)	Multi-scan (<i>TWINABS</i> ; Bruker 2013)
<i>T_{min}</i> , <i>T_{max}</i>	0.84, 0.94	0.55, 0.78
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	18572, 2291, 2158	4453, 4453, 3600
<i>R_{int}</i>	0.027	0.046
(sin θ/λ) _{max} (Å ⁻¹)	0.716	0.716
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.030, 0.083, 1.08	0.030, 0.152, 1.03
No. of reflections	2291	4453
No. of parameters	100	101
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.47, -0.23	1.15, -1.15

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS* and *SHELXTL2014* (Sheldrick, 2008), *SHELXL2014/6* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008).

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supporting information

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Crystal structures of 6-chloroindan-1-one and 6-bromoindan-1-one exhibit different intermolecular packing interactions

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/6* (Sheldrick, 2015); molecular graphics: *SHELXTL2014* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL2014* (Sheldrick, 2008), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008).

(I) 6-Chloroindan-1-one

Crystal data

C₉H₇ClO

$M_r = 166.60$

Monoclinic, $P2_1/c$

$a = 16.319$ (6) Å

$b = 6.024$ (2) Å

$c = 7.745$ (3) Å

$\beta = 99.524$ (5)°

$V = 750.9$ (5) Å³

$Z = 4$

$F(000) = 344$

$D_x = 1.474$ Mg m⁻³

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

Cell parameters from 9875 reflections

$\theta = 2.5$ – 30.5 °

$\mu = 0.44$ mm⁻¹

$T = 125$ K

Block, colourless

$0.28 \times 0.25 \times 0.14$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2013)

$T_{\min} = 0.84$, $T_{\max} = 0.94$

18572 measured reflections

2291 independent reflections

2158 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 30.6$ °, $\theta_{\min} = 2.5$ °

$h = -23 \rightarrow 23$

$k = -8 \rightarrow 8$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.083$

$S = 1.08$

2291 reflections

100 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.2902P]$

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

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

$\Delta\rho_{\max} = 0.47$ e Å⁻³

$\Delta\rho_{\min} = -0.23$ e Å⁻³

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.44180 (2)	0.68505 (5)	0.30384 (4)	0.02625 (9)
O1	0.09308 (5)	0.71579 (13)	0.08199 (10)	0.02037 (16)
C1	0.12100 (5)	0.87485 (15)	0.17008 (11)	0.01444 (17)
C2	0.07137 (6)	1.06661 (17)	0.22843 (12)	0.01704 (18)
H2A	0.035	1.0136	0.3102	0.02*
H2B	0.0363	1.1363	0.1262	0.02*
C3	0.13555 (6)	1.23410 (17)	0.32015 (14)	0.02079 (19)
H3A	0.1328	1.3753	0.254	0.025*
H3B	0.1257	1.2653	0.4405	0.025*
C4	0.21867 (6)	1.12199 (16)	0.32313 (12)	0.01535 (17)
C5	0.29771 (6)	1.19687 (16)	0.39740 (13)	0.01873 (19)
H5A	0.3046	1.3374	0.4536	0.022*
C6	0.36615 (6)	1.06251 (17)	0.38780 (13)	0.01935 (19)
H6A	0.4203	1.1114	0.4374	0.023*
C7	0.35530 (6)	0.85462 (17)	0.30482 (13)	0.01715 (18)
C8	0.27767 (6)	0.77850 (16)	0.22694 (12)	0.01593 (17)
H8A	0.2708	0.6394	0.1685	0.019*
C9	0.21000 (5)	0.91673 (15)	0.23876 (11)	0.01374 (17)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.01319 (12)	0.03164 (15)	0.03279 (15)	0.00500 (8)	0.00046 (9)	-0.00599 (10)
O1	0.0170 (3)	0.0196 (3)	0.0230 (3)	-0.0015 (3)	-0.0012 (3)	-0.0029 (3)
C1	0.0134 (4)	0.0153 (4)	0.0143 (4)	0.0008 (3)	0.0012 (3)	0.0028 (3)
C2	0.0153 (4)	0.0186 (4)	0.0174 (4)	0.0039 (3)	0.0033 (3)	0.0016 (3)
C3	0.0202 (4)	0.0180 (4)	0.0233 (4)	0.0050 (4)	0.0013 (4)	-0.0045 (4)
C4	0.0174 (4)	0.0145 (4)	0.0138 (4)	0.0008 (3)	0.0017 (3)	0.0006 (3)
C5	0.0211 (4)	0.0158 (4)	0.0181 (4)	-0.0026 (3)	-0.0001 (3)	-0.0020 (3)
C6	0.0164 (4)	0.0208 (4)	0.0197 (4)	-0.0041 (3)	-0.0004 (3)	-0.0003 (3)
C7	0.0124 (4)	0.0203 (4)	0.0184 (4)	0.0012 (3)	0.0014 (3)	0.0000 (3)
C8	0.0138 (4)	0.0161 (4)	0.0175 (4)	0.0007 (3)	0.0015 (3)	-0.0017 (3)
C9	0.0133 (4)	0.0142 (4)	0.0134 (4)	0.0000 (3)	0.0011 (3)	0.0003 (3)

Geometric parameters (\AA , $^\circ$)

C11—C7	1.7435 (11)	C4—C9	1.3947 (13)
O1—C1	1.2200 (12)	C4—C5	1.3974 (14)
C1—C9	1.4834 (13)	C5—C6	1.3913 (15)

C1—C2	1.5218 (13)	C5—H5A	0.95
C2—C3	1.5406 (15)	C6—C7	1.4054 (14)
C2—H2A	0.99	C6—H6A	0.95
C2—H2B	0.99	C7—C8	1.3880 (13)
C3—C4	1.5120 (14)	C8—C9	1.3981 (13)
C3—H3A	0.99	C8—H8A	0.95
C3—H3B	0.99		
O1—C1—C9	125.95 (9)	C5—C4—C3	128.81 (9)
O1—C1—C2	126.46 (9)	C6—C5—C4	119.00 (9)
C9—C1—C2	107.58 (8)	C6—C5—H5A	120.5
C1—C2—C3	106.23 (8)	C4—C5—H5A	120.5
C1—C2—H2A	110.5	C5—C6—C7	120.09 (9)
C3—C2—H2A	110.5	C5—C6—H6A	120.0
C1—C2—H2B	110.5	C7—C6—H6A	120.0
C3—C2—H2B	110.5	C8—C7—C6	122.01 (9)
H2A—C2—H2B	108.7	C8—C7—C11	119.08 (8)
C4—C3—C2	104.73 (8)	C6—C7—C11	118.90 (7)
C4—C3—H3A	110.8	C7—C8—C9	116.67 (9)
C2—C3—H3A	110.8	C7—C8—H8A	121.7
C4—C3—H3B	110.8	C9—C8—H8A	121.7
C2—C3—H3B	110.8	C4—C9—C8	122.60 (8)
H3A—C3—H3B	108.9	C4—C9—C1	109.64 (8)
C9—C4—C5	119.62 (9)	C8—C9—C1	127.76 (9)
C9—C4—C3	111.57 (8)		
O1—C1—C2—C3	-174.57 (9)	C11—C7—C8—C9	177.06 (7)
C9—C1—C2—C3	5.05 (10)	C5—C4—C9—C8	0.96 (14)
C1—C2—C3—C4	-4.48 (10)	C3—C4—C9—C8	-178.67 (9)
C2—C3—C4—C9	2.39 (11)	C5—C4—C9—C1	-179.59 (8)
C2—C3—C4—C5	-177.19 (9)	C3—C4—C9—C1	0.78 (11)
C9—C4—C5—C6	-1.02 (14)	C7—C8—C9—C4	0.33 (14)
C3—C4—C5—C6	178.54 (9)	C7—C8—C9—C1	-179.02 (9)
C4—C5—C6—C7	-0.18 (15)	O1—C1—C9—C4	175.91 (9)
C5—C6—C7—C8	1.53 (15)	C2—C1—C9—C4	-3.71 (10)
C5—C6—C7—C11	-177.11 (8)	O1—C1—C9—C8	-4.67 (15)
C6—C7—C8—C9	-1.57 (14)	C2—C1—C9—C8	175.71 (9)

Hydrogen-bond geometry (Å, °)

<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>
C2—H2A \cdots O1 ⁱ	0.99	2.56	3.1933 (15)	121
C2—H2B \cdots O1 ⁱⁱ	0.99	2.59	3.5448 (14)	161

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

(II) 6-Bromoindan-1-one

Crystal data

C₉H₇BrO $M_r = 211.06$ Monoclinic, $P2_1/c$ $a = 6.489$ (2) Å $b = 17.101$ (6) Å $c = 7.224$ (3) Å $\beta = 102.964$ (5)° $V = 781.2$ (5) Å³ $Z = 4$ $F(000) = 416$ $D_x = 1.794$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9955 reflections

 $\theta = 2.4$ – 30.6 ° $\mu = 5.19$ mm⁻¹ $T = 125$ K

Block, colourless

 $0.40 \times 0.21 \times 0.05$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm⁻¹ φ and ω scans

Absorption correction: multi-scan

(TWINABS; Bruker 2013)

 $T_{\min} = 0.55$, $T_{\max} = 0.78$

4453 measured reflections

4453 independent reflections

3600 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.046$ $\theta_{\max} = 30.6$ °, $\theta_{\min} = 2.4$ ° $h = -9 \rightarrow 9$ $k = 0 \rightarrow 24$ $l = 0 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.152$ $S = 1.03$

4453 reflections

101 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.1149P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 1.15$ e Å⁻³ $\Delta\rho_{\min} = -1.15$ e Å⁻³

Special details

Experimental. BASF 0.0762 (5)

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

Refinement. Refined as a 2-component twin

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.34409 (5)	0.45945 (2)	0.19485 (4)	0.02009 (16)
O1	0.4929 (3)	0.12531 (11)	0.2717 (4)	0.0233 (5)
C1	0.3083 (4)	0.14135 (17)	0.2062 (4)	0.0159 (5)
C2	0.1260 (4)	0.08352 (17)	0.1453 (4)	0.0179 (5)
H2A	0.1117	0.0502	0.2537	0.021*
H2B	0.1515	0.0494	0.0419	0.021*
C3	-0.0764 (5)	0.13304 (17)	0.0755 (4)	0.0192 (6)
H3A	-0.1424	0.1212	-0.0589	0.023*

H3B	-0.1806	0.1229	0.1539	0.023*
C4	-0.0002 (4)	0.21670 (16)	0.0973 (4)	0.0154 (5)
C5	-0.1162 (5)	0.28574 (17)	0.0538 (4)	0.0194 (5)
H5A	-0.2646	0.2838	0.0039	0.023*
C6	-0.0119 (4)	0.35734 (17)	0.0842 (4)	0.0178 (5)
H6A	-0.0895	0.4045	0.0544	0.021*
C7	0.2070 (4)	0.36019 (16)	0.1585 (4)	0.0158 (5)
C8	0.3257 (4)	0.29262 (17)	0.2034 (4)	0.0155 (5)
H8A	0.474	0.2947	0.2538	0.019*
C9	0.2184 (4)	0.22114 (15)	0.1714 (4)	0.0143 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0266 (2)	0.0106 (2)	0.0232 (2)	-0.00221 (8)	0.00597 (15)	-0.00020 (8)
O1	0.0173 (10)	0.0158 (10)	0.0356 (13)	0.0027 (8)	0.0034 (9)	-0.0001 (9)
C1	0.0176 (11)	0.0125 (12)	0.0182 (13)	-0.0028 (9)	0.0054 (10)	-0.0013 (9)
C2	0.0192 (12)	0.0131 (12)	0.0217 (13)	-0.0026 (10)	0.0052 (10)	-0.0006 (10)
C3	0.0195 (13)	0.0168 (13)	0.0209 (13)	-0.0060 (10)	0.0035 (11)	-0.0010 (10)
C4	0.0165 (11)	0.0153 (12)	0.0144 (11)	-0.0011 (9)	0.0033 (9)	0.0006 (9)
C5	0.0157 (11)	0.0193 (13)	0.0220 (13)	0.0006 (10)	0.0015 (10)	0.0025 (10)
C6	0.0194 (12)	0.0145 (12)	0.0192 (13)	0.0043 (10)	0.0038 (10)	0.0026 (10)
C7	0.0184 (12)	0.0132 (11)	0.0161 (12)	-0.0018 (9)	0.0042 (10)	0.0002 (9)
C8	0.0155 (11)	0.0130 (13)	0.0177 (13)	-0.0011 (9)	0.0033 (10)	0.0002 (9)
C9	0.0156 (11)	0.0122 (11)	0.0151 (11)	-0.0010 (9)	0.0036 (9)	-0.0001 (9)

Geometric parameters (Å, °)

Br1—C7	1.907 (3)	C4—C5	1.398 (4)
O1—C1	1.216 (3)	C4—C9	1.401 (4)
C1—C9	1.483 (4)	C5—C6	1.392 (4)
C1—C2	1.529 (4)	C5—H5A	0.95
C2—C3	1.549 (4)	C6—C7	1.402 (4)
C2—H2A	0.99	C6—H6A	0.95
C2—H2B	0.99	C7—C8	1.386 (4)
C3—C4	1.510 (4)	C8—C9	1.400 (4)
C3—H3A	0.99	C8—H8A	0.95
C3—H3B	0.99		
O1—C1—C9	126.1 (3)	C9—C4—C3	111.8 (2)
O1—C1—C2	126.6 (3)	C6—C5—C4	119.3 (3)
C9—C1—C2	107.3 (2)	C6—C5—H5A	120.4
C1—C2—C3	106.6 (2)	C4—C5—H5A	120.4
C1—C2—H2A	110.4	C5—C6—C7	120.4 (3)
C3—C2—H2A	110.4	C5—C6—H6A	119.8
C1—C2—H2B	110.4	C7—C6—H6A	119.8
C3—C2—H2B	110.4	C8—C7—C6	121.5 (3)
H2A—C2—H2B	108.6	C8—C7—Br1	119.5 (2)

C4—C3—C2	104.5 (2)	C6—C7—Br1	119.0 (2)
C4—C3—H3A	110.9	C7—C8—C9	117.3 (2)
C2—C3—H3A	110.9	C7—C8—H8A	121.3
C4—C3—H3B	110.9	C9—C8—H8A	121.3
C2—C3—H3B	110.9	C8—C9—C4	122.3 (2)
H3A—C3—H3B	108.9	C8—C9—C1	127.8 (2)
C5—C4—C9	119.2 (2)	C4—C9—C1	109.9 (2)
C5—C4—C3	129.0 (2)		
O1—C1—C2—C3	179.0 (3)	Br1—C7—C8—C9	-179.01 (19)
C9—C1—C2—C3	-0.8 (3)	C7—C8—C9—C4	-0.1 (4)
C1—C2—C3—C4	0.7 (3)	C7—C8—C9—C1	179.6 (3)
C2—C3—C4—C5	179.3 (3)	C5—C4—C9—C8	-0.1 (4)
C2—C3—C4—C9	-0.4 (3)	C3—C4—C9—C8	179.6 (2)
C9—C4—C5—C6	0.3 (4)	C5—C4—C9—C1	-179.8 (2)
C3—C4—C5—C6	-179.4 (3)	C3—C4—C9—C1	-0.1 (3)
C4—C5—C6—C7	-0.3 (4)	O1—C1—C9—C8	1.1 (5)
C5—C6—C7—C8	0.1 (4)	C2—C1—C9—C8	-179.2 (3)
C5—C6—C7—Br1	179.2 (2)	O1—C1—C9—C4	-179.2 (3)
C6—C7—C8—C9	0.0 (4)	C2—C1—C9—C4	0.6 (3)

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>
C3—H3B...O1 ⁱ	0.99	2.45	3.408 (4)	162
C5—H5A...O1 ⁱⁱ	0.95	2.55	3.253 (4)	131
C2—H2B...Br1 ⁱⁱⁱ	0.99	3.05	3.898 (3)	145

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