

***trans*-3-Bromo-2-chloroindan-1-one**Pen-Shao Yu, Gareth D. Thomas,
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PO Box 56, Dunedin, New ZealandCorrespondence e-mail:
blackman@alkali.otago.ac.nz**Key indicators**Single-crystal X-ray study
 $T = 158$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.035
 wR factor = 0.094
Data-to-parameter ratio = 16.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Structural analysis of *trans*-3-bromo-2-chloroindan-1-one, $\text{C}_9\text{H}_6\text{BrClO}$, confirms the relative stereochemistry of the Cl and Br atoms.

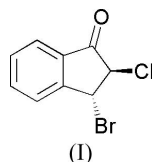
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Comment

The title compound, (I) (Fig. 1), was prepared as part of a study to investigate the stereoselectivity of radical bromination reactions of 2-chloroindanones. The structure determination was undertaken to determine the relative stereochemistry of the Cl and Br substituents. Compound (I) crystallizes as a single diastereoisomer in the non-polar space group $P2_1/n$, confirming the presence of both the *RR* and *SS* enantiomers. The benzene ring is essentially planar (average deviation = 0.0045 Å) while the five-membered ring adopts an envelope conformation, with atom C8 lying 0.361 (5) Å out of the mean plane formed by atoms C9/C1/C6/C7. The Br and Cl substituents exhibit a relative *trans* stereochemistry, and both are involved in close contacts with atoms of neighbouring molecules, with intermolecular $\text{Cl}\cdots\text{Cl}(1-x, 1-y, 1-z)$ and $\text{Br}\cdots\text{O}(x, 1+y, z)$ distances of 3.400 (2) and 3.122 (3) Å, respectively. π stacking between the benzene rings of neighbouring molecules (symmetry code: $1-x, 1-y, -z$) is also evident in the three-dimensional structure, with a centroid-to-centroid distance of 3.629 (3) Å.

**Experimental**

2-Chloroindanone was prepared by chlorination of indanone with *N*-chlorosuccinimide (NCS). The procedure involved the slow addition of NCS (1.33 g, 0.01 mol) over a 2 h period to a stirred mixture of indanone (1.50 g, 0.01 mol) and concentrated HCl (2 ml) in acetic acid (50 ml). The reaction mixture was stirred at room temperature overnight and the product was extracted into dichloromethane. The organic phase was washed with 10% aqueous sodium carbonate, dried, evaporated at reduced pressure, and the residue purified by column chromatography on silica, eluting with dichloromethane/hexane (2:1). For the synthesis of 3-bromo-2-chloroindan-1-one, a mixture of 2-chloroindanone (0.50 g, 3.0 mmol) and *N*-bromosuccinimide (0.64 g, 3.6 mmol) in dry benzene (40 ml) was heated at reflux under nitrogen for 2 h, with irradiation from a 160 W mercury gas-discharge lamp. The reaction mixture was then cooled, washed with 5% aqueous sodium carbonate (2×50 ml), dried, and evaporated under reduced pressure. The resulting residue was recrystallized from hexane to give the title compound (m.p. 369–370 K).

Analysis calculated for C₉H₆BrClO: C 44.01, H 2.46, Br 32.55, Cl 14.44%; found: C 44.11, H 2.44, Br 32.95, Cl 14.51%. ¹H NMR δ 4.70 (1H, *d*, *J* = 3.6 Hz), 5.47 (1H, *d*, *J* = 3.6 Hz), 7.56 (1H, *t*, *J* = 7.8 Hz), 7.70 (1H, *d*, *J* = 7.8 Hz), 7.79 (1H, *d*, *J* = 7.8 Hz), 7.82 (1H, *t*, *J* = 7.8 Hz).

Crystal data

C₉H₆BrClO
M_r = 245.5
 Monoclinic, *P*₂₁/*n*
a = 7.836 (1) Å
b = 8.228 (1) Å
c = 14.046 (2) Å
 β = 94.106 (5)°
V = 903.3 (2) Å³
Z = 4

D_x = 1.805 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 3310 reflections
 θ = 2.5–26.4°
 μ = 4.79 mm⁻¹
T = 158 (2) K
 Block, colourless
 0.45 × 0.45 × 0.45 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.074, *T_{max}* = 0.116
 4202 measured reflections

1784 independent reflections
 1642 reflections with *I* > 2σ(*I*)
R_{int} = 0.018
 θ_{max} = 26.3°
h = -4 → 9
k = -9 → 10
l = -17 → 17

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.035
wR(*F*²) = 0.094
S = 1.13
 1784 reflections
 109 parameters
 H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0623*P*)² + 0.531*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 1.32 e Å⁻³
 Δρ_{min} = -0.86 e Å⁻³

All H atoms were included in calculated positions (C–H distances are 0.95 Å for benzene H atoms and 1.00 Å for methine H atoms) and were refined as riding atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(parent atom). The highest peak in the difference map is 0.96 Å from atom Br1.

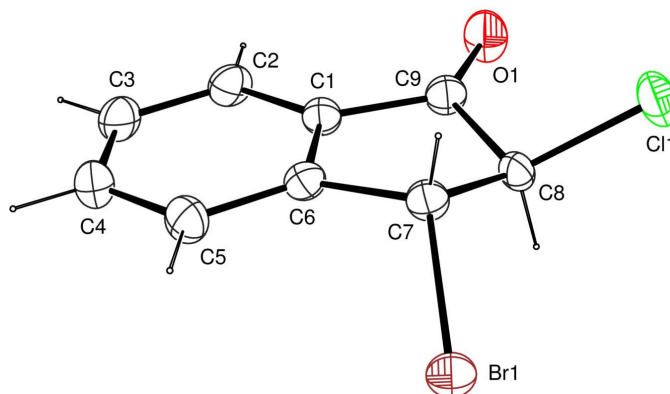


Figure 1 The structure of (I), with displacement ellipsoids drawn at the 50% probability level.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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