Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Stájer, F., Frimpong-Manso, S., Bernáth, G. & Sohár, P. (1991). *J. Hetercycl. Chem.* **28**, 753–757.
Vicković, I. (1988). *CSU. Crystal Structure Utility Computer Program.* Univ. of Zagreb, Croatia.

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Homochiral Methyl (2*R*,3*S*)-3-Bromo-2-chloro-3-phenylpropanoate

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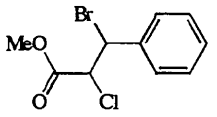
Abstract

The stereochemistries at positions 2 and 3 of the title compound, $C_{10}H_{10}BrClO_2$, have been confirmed by X-ray crystal structural analysis. The halogen atoms show an antiperiplanar arrangement.

Comment

The title compound, (I), was investigated as part of a study into the stereoselective bromination of α-halohydrocinnamates and structural determination was



(I)

undertaken to obtain the absolute stereochemistries at positions 2 and 3. There are two formula units of (I) in the asymmetric unit with the most notable difference between the angles C12—C13—Cl1 [105.0 (4)°] and C22—C23—Cl2 [110.0 (5)°]. Bond distances within the phenyl rings range from 1.364 (11) to 1.425 (10) Å. Stereochemistries at C13, C23 and C14, C24, *i.e.* positions 2 and 3 in the two molecules, were found to be *R* and *S*, respectively, with the correct choice of absolute stereochemistry confirmed by the value of the Flack parameter [0.009 (13)] (Flack, 1983). The halogen atoms in (I) are found to be essentially (+)-antiperiplanar, with the torsion angles Cl1—C13—C14—Br1 and Cl2—C23—C24—Br2 being −178.8 (5) and 176.1 (5)°, respectively. This contrasts with a similar structure of a dibromohydrocinnamate (Rappoport & Gazit, 1986) in which the halogens adopt a *gauche* conformation. The antiperiplanar arrangement in (I) obviously derives from the geometry of the intermediate, which allows insight into the mechanism of the reaction (Shaw & Tan, 1995).

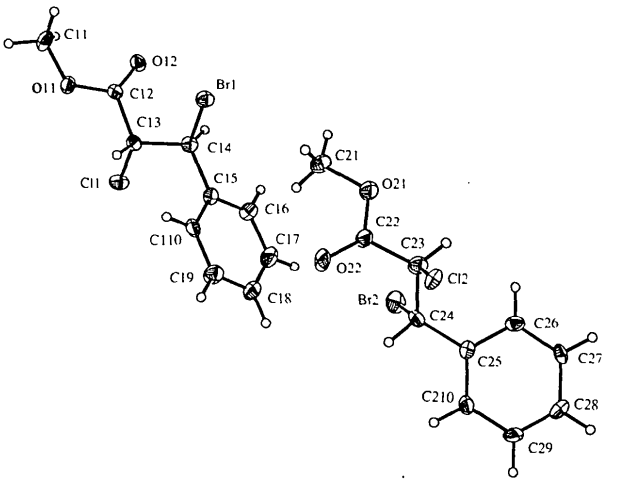


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the two independent molecules of (I) showing displacement ellipsoids drawn at the 50% probability level.

Experimental

A mixture of methyl (*R*)-2-chloro-3-phenylpropanoate (0.50 g, 2.5 mmol) and *N*-bromosuccinimide (0.54 g, 3.0 mmol) in CCl_4 (40 ml) was heated at reflux under nitrogen for 2 h, with reaction initiated by irradiation with a 160 W mercury lamp. The reaction mixture was then cooled, filtered and evaporated under reduced pressure. The resulting solid was recrystallized from hexane (Shaw & Tan, 1995).

Crystal data

$C_{10}H_{10}BrClO_2$
M_r = 277.53
Monoclinic
$P2_1$
a = 8.677 (2) Å
b = 9.438 (2) Å
c = 13.504 (3) Å
β = 96.45 (3)°
V = 1098.9 (4) Å^3
Z = 4
D_x = 1.678 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 13–16°
μ = 3.955 mm^{-1}
T = 130 (2) K
Rhomb
0.72 × 0.60 × 0.22 mm
Colourless

Data collection

Nicolet *R3M* diffractometer
ω scans
R_{int} = 0.0346
θ_{max} = 27.50°

Absorption correction: empirical ψ scans (Sheldrick, 1991)
$T_{min} = 0.632$, $T_{max} = 0.947$
2845 measured reflections
2675 independent reflections
2413 observed reflections $[I > 2\sigma(I)]$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -17 \rightarrow 17$
3 standard reflections monitored every 97 reflections
intensity variation: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0401$
$wR(F^2) = 0.1064$
$S = 0.930$
2675 reflections
255 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.019$
$\Delta\rho_{max} = 1.151$ e Å^{-3}
$\Delta\rho_{min} = -1.052$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C11	0.2636 (8)	0.5675 (10)	1.1483 (5)	0.028 (2)
O11	0.3907 (5)	0.6143 (5)	1.0959 (3)	0.0230 (10)
C12	0.4860 (7)	0.5105 (8)	1.0717 (5)	0.0191 (13)
O12	0.4658 (6)	0.3874 (5)	1.0828 (4)	0.0228 (11)
C13	0.6256 (6)	0.5761 (7)	1.0277 (4)	0.0171 (11)
Cl1	0.7709 (2)	0.6034 (2)	1.13400 (10)	0.0243 (3)
C14	0.6924 (6)	0.4767 (7)	0.9550 (4)	0.0176 (12)
Br1	0.52780 (6)	0.45769 (7)	0.84160 (4)	0.0202 (2)
C15	0.8407 (7)	0.5313 (7)	0.9178 (4)	0.0177 (12)
C16	0.9665 (7)	0.4403 (8)	0.9221 (4)	0.0222 (15)
C17	1.1032 (7)	0.4861 (9)	0.8874 (5)	0.029 (2)
C18	1.1149 (8)	0.6218 (9)	0.8488 (5)	0.029 (2)
C19	0.9817 (8)	0.7111 (9)	0.8419 (5)	0.029 (2)
C110	0.8485 (8)	0.6650 (8)	0.8765 (5)	0.0228 (13)
C21	0.7640 (8)	0.4117 (10)	0.6351 (5)	0.031 (2)
O21	0.8684 (6)	0.3859 (6)	0.5608 (4)	0.0295 (12)
C22	0.9510 (7)	0.4946 (7)	0.5373 (5)	0.0244 (14)
O22	0.9413 (6)	0.6133 (6)	0.5705 (4)	0.0370 (14)
C23	1.0632 (7)	0.4542 (9)	0.4619 (5)	0.0252 (13)
Cl2	1.01086 (14)	0.5443 (2)	0.34652 (9)	0.0177 (3)
C24	1.2262 (7)	0.5024 (8)	0.5005 (4)	0.0235 (13)
Br2	1.27458 (8)	0.40986 (8)	0.63417 (4)	0.0282 (2)
C25	1.3511 (7)	0.4646 (9)	0.4358 (4)	0.0209 (12)
C26	1.3568 (7)	0.3292 (8)	0.3927 (5)	0.0219 (14)
C27	1.4795 (8)	0.2950 (7)	0.3391 (5)	0.0202 (13)
C28	1.5968 (8)	0.3933 (9)	0.3290 (5)	0.025 (2)
C29	1.5911 (7)	0.5248 (9)	0.3705 (5)	0.026 (2)
C210	1.4672 (7)	0.5623 (8)	0.4247 (4)	0.0212 (13)

Table 2. *Selected geometric parameters* (Å, °)

C11—O11	1.444 (7)	C21—O21	1.446 (7)
O11—C12	1.346 (8)	O21—C22	1.311 (8)
C12—O12	1.187 (9)	C22—O22	1.213 (9)
C12—C13	1.538 (8)	C22—C23	1.535 (8)
C13—C14	1.520 (8)	C23—C24	1.520 (8)
C13—Cl1	1.819 (6)	C23—Cl2	1.788 (7)
C14—C15	1.523 (8)	C24—C25	1.509 (8)
C14—Br1	1.982 (5)	C24—Br2	2.007 (7)
C15—C16	1.385 (9)	C25—C26	1.407 (11)
C15—C110	1.385 (10)	C25—C210	1.386 (10)
C16—C17	1.391 (9)	C26—C27	1.390 (8)
C17—C18	1.391 (11)	C27—C28	1.396 (10)
C18—C19	1.425 (10)	C28—C29	1.364 (11)
C19—C110	1.365 (9)	C29—C210	1.412 (8)
C14—C13—Cl1	108.4 (4)	C24—C23—Cl2	107.0 (5)
C12—C13—Cl1	105.0 (4)	C22—C23—Cl2	110.0 (5)
C13—C14—Br1	105.1 (4)	C25—C24—Br2	109.1 (4)
C15—C14—Br1	109.8 (4)	C23—C24—Br2	105.7 (4)

Data collection, cell refinement and data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1198). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Rappoport, Z. & Gazit, A. (1986). *J. Org. Chem.* **51**, 4112–4131.
Shaw, J. P. & Tan, E. W. (1995). In preparation.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1994). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

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(13-Methyl-1,4,7,8,13,13b-hexahydro[1′,2′]-oxazepino[2′,3′:1,2]pyrido[3,4-*b*]indol-1-yl)-methanol and its Unusual Packing Style

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Abstract

The asymmetric unit of the crystal of (13-methyl-1,4,7,8,13,13b-hexahydro[1′,2′]oxazepino[2′,3′:1,2]-pyrido[3,4-*b*]indol-1-yl)methanol, $C_{17}H_{20}N_2O_2$, is

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