

- 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. Heterocycl. Chem.* **28**, 753–757.
- Vicković, I. (1988). *CSU. Crystal Structure Utility Computer Program*. Univ. of Zagreb, Croatia.

Acta Cryst. (1995). **C51**, 134–135

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

JOANNE P. SHAW, ENG WUI TAN
AND ALLAN G. BLACKMAN

*Department of Chemistry, University of Otago,
PO Box 56, Dunedin, New Zealand*

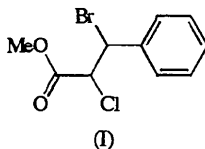
(Received 13 May 1994; accepted 14 July 1994)

Abstract

The stereochemistries at positions 2 and 3 of the title compound, C₁₀H₁₀BrClO₂, 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



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—C11 [105.0 (4)°] and C22—C23—C12 [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 C11—C13—C14—Br1 and C12—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).

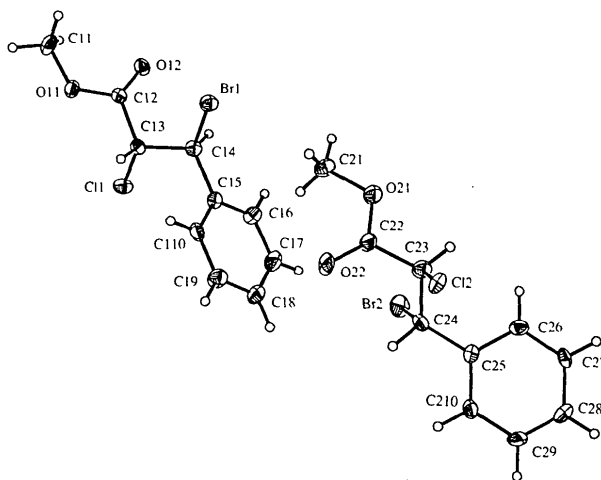


Fig. 1. ORTEP (Johnson, 1965) drawing of the two independent molecules of (I) showing displacement ellipsoids drawn at the 50% probability level.

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 CCl₄ (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

C₁₀H₁₀BrClO₂

M_r = 277.53

Monoclinic

*P*2₁

a = 8.677 (2) Å

b = 9.438 (2) Å

c = 13.504 (3) Å

β = 96.45 (3)°

V = 1098.9 (4) Å³

Z = 4

D_x = 1.678 Mg m^{−3}

Data collection

Nicolet R3M diffractometer

ω scans

Mo *K* α radiation

λ = 0.71073 Å

Cell parameters from 24 reflections

θ = 13–16°

μ = 3.955 mm^{−1}

T = 130 (2) K

Rhombohedral

0.72 × 0.60 × 0.22 mm

Colourless

*R*_{int} = 0.0346

θ_{max} = 27.50°

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

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\max} = 0.019$
 $R[F^2 > 2\sigma(F^2)] = 0.0401$ $\Delta\rho_{\max} = 1.151 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.1064$ $\Delta\rho_{\min} = -1.052 \text{ e } \text{\AA}^{-3}$
 $S = 0.930$ Extinction correction: none
 2675 reflections Atomic scattering factors
 255 parameters from *International Tables*
 H-atom parameters for *Crystallography* (1992,
 refined Vol. C, Tables 4.2.6.8 and
 $w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ 6.1.1.4)
 where $P = (F_o^2 + 2F_c^2)/3$

C14—C13—C11	108.4 (4)	C24—C23—C12	107.0 (5)
C12—C13—C11	105.0 (4)	C22—C23—C12	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: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994).

We thank Professor W. T. Robinson, University of Canterbury, for data collection, and Associate Professor J. Simpson and Dr L. R. Hanton for assistance with crystallography. This work is supported by a University of Otago Division of Sciences Research Grant.

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.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	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)
C11	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)
C12	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 ($\text{\AA}$, $^\circ$)

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—C11	1.819 (6)	C23—C12	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)

References

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 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). *SHELXS86. 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). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). **C51**, 135–139

(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

HIROFUMI OHISHI,* YASUHIKO SAKAMOTO,†
 SHINYA HARUSAWA, RYUJI YONEDA AND
 TAKUSHI KURIHARA

Osaka University of Pharmaceutical Sciences,
 2-10-65 Kawai, Matsubara, Osaka 580, Japan

(Received 5 March 1993; accepted 4 August 1994)

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

† Present address: Research Laboratories Nipponshoji Kaisha, 24-3 Sho 2-Chome, Ibaraki, Osaka 567, Japan.