

C4A	0.2564 (3)	0.7835 (2)	0.4536 (2)	0.0477 (8)
C5A	0.1894 (3)	0.7132 (2)	0.4246 (2)	0.0495 (8)
C3	0.2943 (3)	0.8075 (2)	0.5561 (2)	0.0428 (8)
C4	0.1814 (3)	0.8179 (2)	0.5909 (2)	0.0509 (9)
C5	0.2265 (3)	0.8398 (2)	0.6948 (2)	0.0578 (9)
C6	0.3091 (3)	0.9050 (2)	0.7119 (3)	0.0691 (11)
C7	0.4212 (3)	0.8958 (2)	0.6772 (2)	0.0632 (10)
C8	0.3754 (3)	0.8739 (2)	0.5729 (2)	0.0544 (9)
C9	0.2267 (3)	0.6528 (2)	0.4937 (2)	0.0462 (8)
C10	0.3670 (3)	0.6326 (2)	0.5132 (2)	0.0605 (9)
C11	0.4076 (4)	0.5718 (2)	0.5812 (3)	0.0794 (12)
C12	0.3211 (4)	0.5089 (2)	0.5450 (3)	0.0897 (13)
C13	0.1808 (4)	0.5274 (2)	0.5243 (3)	0.0779 (12)
C14	0.1400 (3)	0.5894 (2)	0.4573 (2)	0.0627 (10)
S2B	0.18490 (11)	0.81074 (5)	0.93464 (7)	0.0729 (3)
O1B	0.1819 (2)	0.83606 (12)	1.03662 (15)	0.0653 (7)
O2B	0.0530 (3)	0.7934 (2)	0.8778 (2)	0.1016 (10)
O3B	0.2621 (2)	0.73947 (11)	0.97614 (14)	0.0580 (6)
C4B	0.2307 (3)	0.7164 (2)	1.0588 (2)	0.0485 (8)
C5B	0.2317 (3)	0.7842 (2)	1.1124 (2)	0.0513 (9)
C17	0.3188 (3)	0.6569 (2)	1.1082 (2)	0.0457 (8)
C18	0.3174 (3)	0.5984 (2)	1.0387 (2)	0.0556 (9)
C19	0.3988 (4)	0.5360 (2)	1.0883 (3)	0.0657 (10)
C20	0.3592 (4)	0.5089 (2)	1.1682 (3)	0.0735 (11)
C21	0.3595 (4)	0.5664 (2)	1.2376 (2)	0.0717 (11)
C22	0.2784 (3)	0.6286 (2)	1.1890 (2)	0.0605 (9)
C23	0.3592 (3)	0.8108 (2)	1.1835 (2)	0.0460 (8)
C24	0.4638 (3)	0.8256 (2)	1.1414 (2)	0.0549 (9)
C25	0.5884 (3)	0.8486 (2)	1.2176 (3)	0.0656 (10)
C26	0.5660 (4)	0.9124 (2)	1.2695 (3)	0.0699 (11)
C27	0.4620 (4)	0.8996 (2)	1.3108 (3)	0.0693 (11)
C28	0.3367 (3)	0.8755 (2)	1.2354 (2)	0.0614 (10)

Table 2. Selected geometric parameters (Å, °)

S2A—O2A	1.443 (3)	S2B—O2B	1.445 (3)
S2A—O3A	1.607 (2)	S2B—O3B	1.612 (2)
S2A—O1A	1.608 (2)	S2B—O1B	1.613 (2)
O1A—C5A	1.465 (4)	O1B—C5B	1.462 (4)
O3A—C4A	1.454 (4)	O3B—C4B	1.464 (3)
C4A—C5A	1.522 (4)	C4B—C5B	1.521 (4)
O2A—S2A—O3A	105.6 (2)	O2B—S2B—O3B	108.8 (2)
O2A—S2A—O1A	108.9 (2)	O2B—S2B—O1B	106.3 (2)
O3A—S2A—O1A	94.05 (11)	O3B—S2B—O1B	93.95 (11)
C5A—O1A—S2A	109.6 (2)	C5B—O1B—S2B	114.0 (2)
C4A—O3A—S2A	114.0 (2)	C4B—O3B—S2B	109.5 (2)
O3A—C4A—C5A	103.8 (2)	O3B—C4B—C5B	103.2 (2)
O1A—C5A—C4A	102.7 (2)	O1B—C5B—C4B	103.6 (2)
O2A—S2A—O1A—C5A	79.5 (2)	O2B—S2B—O1B—C5B	104.5 (2)
O3A—S2A—O1A—C5A	−28.6 (2)	O3B—S2B—O1B—C5B	−6.5 (2)
O2A—S2A—O3A—C4A	−105.7 (2)	O2B—S2B—O3B—C4B	−79.6 (2)
O1A—S2A—O3A—C4A	5.3 (2)	O1B—S2B—O3B—C4B	29.1 (2)
S2A—O3A—C4A—C5A	17.9 (3)	S2B—O3B—C4B—C5B	−41.7 (3)
S2A—O1A—C5A—C4A	41.6 (3)	S2B—O1B—C5B—C4B	−16.6 (3)
O3A—C4A—C5A—O1A	−35.3 (3)	O3B—C4B—C5B—O1B	34.6 (3)

Data collection and reduction and cell refinement used *CAD-4/PC* software (Enraf–Nonius, 1989). The structure was solved using *SHELXS86* (Sheldrick, 1990), refinement was carried out using *SHELXL92* (Sheldrick, 1992) and the molecular graphics were prepared using *PLUTON* (Spek, 1993). *SHELXL93* (Sheldrick, 1993) was used to prepare material for publication.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5ar-*p*-Tolyl-6at,7t,8,9,10t,10at-hexahydro-11H,13H-7,10-methano[1,3]benzoxazino-[2,3-*b*][1,3]benzoxazin-13-one

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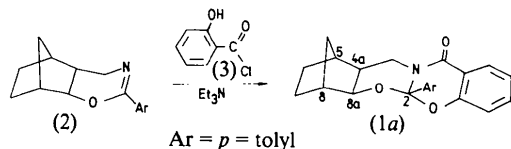
Abstract

The structure of the pentacyclic title compound, $C_{23}H_{23}NO_3$, was established by X-ray diffraction. The *exo* positions of the aryl group, indicated by the NMR data, were confirmed by the relevant torsion angles: $C11—O10—C2—C19 = -79.7 (3)^\circ$, $C4—N3—C2—C19 = -98.5 (3)^\circ$ and $C13—N3—C2—C19 = 86.2 (4)^\circ$. The position of the aryl group is stabilized by a $C24—H24 \cdots N3$ interaction [$H24 \cdots N3$ 2.480 (2), $C24 \cdots N3$ 2.855 (4) Å, $C24—H24 \cdots N3$ 98.7 (2)°]. The conformations of the benzoxazine rings I and II are twist-boat (1T_3) and screw-boat (1S_6), respectively.

Comment

Partially saturated condensed pentacyclic compounds have been synthesized by 4π cycloaddition, with pharmacological and stereochemical aims (Stájer, Frimpong-Manso, Bernáth & Sohár, 1991). The title compound (1a) was prepared from the

norbornane-di-*exo*-condensed dihydro-1,3-oxazine (2) and oxoketene obtained *in situ* by the reaction of salicyloyl chloride (3) and triethylamine. The product, consisting of two components, (1a) and (1b), was separated by chromatography and crystallization. The 1H and ^{13}C NMR data showed that compounds (1a) and (1b) are diastereomers. In (1a) the *p*-tolyl group is in the *exo* position, whereas in (1b) it assumes the *endo* position. This was confirmed by the X-ray analysis presented here. In contrast, because of the poor quality of the single crystals obtained, elucidation of the structure of (1b) was not possible by X-ray diffraction analysis.



As shown by the puckering parameters (Cremer & Pople, 1975) and asymmetry factors (Kálmán, Czugler & Simon, 1982) for the benzoxazine rings and the rings in the norbornane unit (Table 3), the benzoxazine rings I and II have twist-boat (1T_3) and screw-boat (1S_6) conformations, respectively. The norbornane skeleton is similar to the respective parts of related compounds (Kapor, Ribár, Divjaković, Stájer, Bernáth & Argay, 1987; Kapor, Argay, Stájer & Bernáth, 1990) and is *cis* annelated with the benzoxazine ring I [$H4a-C4a-C8a-H8a = 5.8(5)^\circ$]. A weak intermolecular hydrogen bond $C5-H5\cdots O18^i$ [$H5\cdots O18 = 2.478(4)$, $C5\cdots O18$

$3.450(6)$ Å, $C5-H5\cdots O18 = 148.4(2)^\circ$; symmetry code: (i) $-x, -y, 1-z$] leads to the formation of dimers which pack along the direction of the cube diagonal in the crystal lattice (Fig. 2). The C11-C17 and C19-C24 phenyl rings are planar and the best planes make an angle of $84.10(8)^\circ$ with each other.

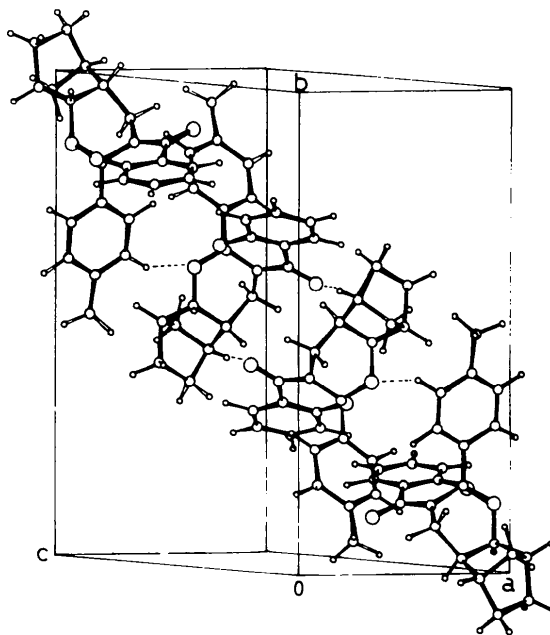


Fig. 2. Packing diagram of the unit cell.

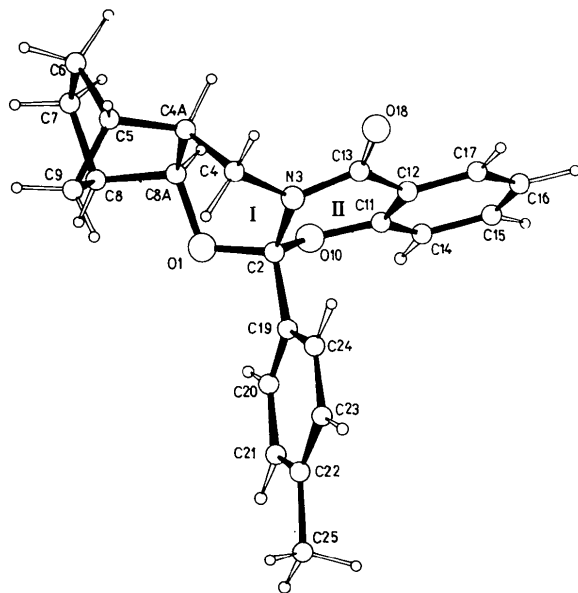


Fig. 1. Perspective view of the title molecule showing the atomic numbering. The H atoms are shown but not labelled.

Experimental

Crystal data

$C_{23}H_{23}NO_3$
 $M_r = 361.44$
 Monoclinic
 $P2_1/c$
 $a = 9.150(4)$ Å
 $b = 17.465(6)$ Å
 $c = 12.098(5)$ Å
 $\beta = 106.89(2)^\circ$
 $V = 1850(1)$ Å³
 $Z = 4$
 $D_x = 1.298$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.7107$ Å
 Cell parameters from 25 reflections
 $\theta = 4.80-7.44^\circ$
 $\mu = 0.08$ mm⁻¹
 $T = 293$ K
 Plate-like
 $0.65 \times 0.40 \times 0.15$ mm
 Colourless

Data collection

Philips PW1100 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 2191 measured reflections
 2093 independent reflections
 2036 observed reflections
 $[F > 4\sigma(F)]$

$R_{int} = 0.0104$
 $\theta_{max} = 28^\circ$
 $h = -11 \rightarrow 11$
 $k = 0 \rightarrow 22$
 $l = 0 \rightarrow 15$
 3 standard reflections
 frequency: 120 min
 intensity variation: 8.44%

Refinement

Refinement on F $R = 0.050$ $wR = 0.058$ $S = 0.870$ 2067 [$F > 3\sigma(F)$] reflections

246 parameters

Only H-atom U 's refined $w = 1/[\sigma^2(F) + 0.000611F^2]$ $(\Delta/\sigma)_{\max} = 0.407 (U_{11}, C4)$ $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = 0.18 \text{ e } \text{\AA}^{-3}$

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

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

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

	x	y	z	U_{eq}
O1	0.1794 (2)	0.1342 (1)	0.2226 (2)	0.0439 (5)
C2	0.0510 (4)	0.1717 (2)	0.2337 (3)	0.0452 (7)
N3	-0.0100 (3)	0.1296 (1)	0.3145 (2)	0.0540 (7)
C4	0.0994 (4)	0.0782 (2)	0.3930 (3)	0.0636 (10)
C4a	0.1381 (4)	0.0150 (2)	0.3190 (3)	0.0549 (8)
C5	0.2866 (4)	-0.0289 (2)	0.3734 (3)	0.0690 (10)
C6	0.2861 (5)	-0.0995 (2)	0.2987 (4)	0.0846 (13)
C7	0.3030 (5)	-0.0651 (2)	0.1853 (4)	0.0717 (11)
C8	0.3230 (4)	0.0213 (2)	0.2123 (4)	0.0532 (8)
C8a	0.1661 (4)	0.0515 (2)	0.2096 (3)	0.0456 (7)
C9	0.4062 (4)	0.0214 (2)	0.3419 (3)	0.0653 (9)
O10	-0.0581 (2)	0.1721 (1)	0.1205 (2)	0.0470 (5)
C11	-0.2065 (3)	0.1897 (2)	0.1169 (3)	0.0481 (8)
C12	-0.2582 (4)	0.1737 (2)	0.2110 (3)	0.0548 (9)
C13	-0.1595 (4)	0.1334 (2)	0.3115 (3)	0.0612 (9)
C14	-0.3012 (4)	0.2183 (2)	0.0154 (3)	0.0609 (9)
C15	-0.4526 (4)	0.2314 (2)	0.0076 (4)	0.0775 (12)
C16	-0.5073 (4)	0.2159 (2)	0.1006 (5)	0.0844 (14)
C17	-0.4119 (5)	0.1875 (2)	0.2017 (4)	0.0741 (12)
O18	-0.2065 (3)	0.1020 (2)	0.3857 (3)	0.0946 (9)
C19	0.0991 (3)	0.2527 (2)	0.2718 (2)	0.0411 (7)
C20	0.1624 (4)	0.2975 (2)	0.2028 (3)	0.0533 (9)
C21	0.2181 (4)	0.3692 (2)	0.2380 (3)	0.0546 (8)
C22	0.2125 (3)	0.3991 (2)	0.3431 (3)	0.0477 (7)
C23	0.1438 (4)	0.3559 (2)	0.4090 (3)	0.0533 (8)
C24	0.0878 (4)	0.2832 (2)	0.3746 (3)	0.0477 (8)
C25	0.2822 (4)	0.4767 (2)	0.3839 (3)	0.0715 (10)

Table 2. Selected geometric parameters ($\text{\AA}$, $^\circ$)

O1—C2	1.385 (4)	O10—C11	1.380 (4)
O1—C8a	1.454 (4)	C11—C12	1.382 (6)
C2—N3	1.458 (5)	C11—C14	1.375 (4)
C2—O10	1.441 (4)	C12—C13	1.467 (5)
C2—C19	1.513 (5)	C12—C17	1.399 (6)
N3—C4	1.469 (4)	C13—O18	1.231 (5)
N3—C13	1.359 (5)	C14—C15	1.380 (5)
C4—C4a	1.526 (5)	C15—C16	1.384 (8)
C4a—C5	1.532 (5)	C16—C17	1.372 (6)
C4a—C8a	1.556 (5)	C19—C20	1.388 (5)
C5—C6	1.528 (5)	C19—C24	1.384 (5)
C5—C9	1.535 (5)	C20—C21	1.372 (5)
C6—C7	1.546 (7)	C21—C22	1.389 (5)
C7—C8	1.544 (5)	C22—C23	1.375 (5)
C8—C8a	1.521 (5)	C22—C25	1.518 (5)
C8—C9	1.531 (5)	C23—C24	1.387 (5)
C2—O1—C8a	115.7 (2)	C5—C9—C8	94.3 (3)
O1—C2—N3	109.1 (3)	C2—O10—C11	115.8 (2)
O1—C2—O10	106.4 (3)	O10—C11—C12	119.9 (3)
O1—C2—C19	107.0 (3)	O10—C11—C14	117.9 (3)
N3—C2—O10	110.2 (3)	C12—C11—C14	122.1 (3)
N3—C2—C19	113.7 (3)	C11—C12—C13	120.3 (3)
O10—C2—C19	110.2 (3)	C11—C12—C17	118.4 (3)
C2—N3—C4	114.8 (3)	C13—C12—C17	120.8 (3)
C2—N3—C13	122.4 (3)	N3—C13—C12	114.7 (3)
C4—N3—C13	122.6 (3)	N3—C13—O18	121.4 (3)
N3—C4—C4a	107.1 (3)	C12—C13—O18	123.8 (3)

C4—C4a—C5	116.0 (3)	C11—C14—C15	118.8 (3)
C4—C4a—C8a	109.0 (3)	C14—C15—C16	120.3 (4)
C5—C4a—C8a	103.1 (3)	C15—C16—C17	120.6 (4)
C4a—C5—C6	107.4 (3)	C12—C17—C16	119.9 (4)
C4a—C5—C9	102.4 (3)	C2—C19—C20	118.6 (3)
C6—C5—C9	101.2 (3)	C2—C19—C24	122.9 (3)
C5—C6—C7	103.1 (3)	C20—C19—C24	118.5 (3)
C6—C7—C8	103.2 (3)	C19—C20—C21	120.7 (3)
C7—C8—C8a	106.3 (3)	C20—C21—C22	121.2 (3)
C7—C8—C9	102.2 (3)	C21—C22—C23	118.0 (3)
C8a—C8—C9	102.0 (3)	C21—C22—C25	120.6 (3)
O1—C8a—C4a	110.1 (3)	C23—C22—C25	121.4 (3)
O1—C8a—C8	107.1 (3)	C22—C23—C24	121.3 (3)
C4a—C8a—C8	103.1 (3)	C19—C24—C23	120.3 (3)

Table 3. Puckering parameters and asymmetry factors

Ring A (benzoxazine I): N3, C4, C4a, C8a, O1, C2. Ring B (benzoxazine II): O10, C11, C12, C13, N3, C2. Ring C: C4a, C5, C9, C8, C8a. Ring D: C5, C6, C7, C8, C9. Ring E C4a, C5, C6, C7, C8, C8a.

Ring	Q ($\text{\AA}$)	θ ($^\circ$)	φ ($^\circ$)	fC_o ($\text{\AA}$)	fC_2 ($\text{\AA}$)	cN_d
A	0.736 (4)	86.9 (3)	44.8 (3)	0.160 (22)	0.093 (13)	$^1T_3 \rightarrow B_{3,6}$
				[N3—C2]	[C2]	
B	0.393 (4)	66.4 (5)	-42.0 (4)	0.111 (28)	0.061 (15)	1S_6
				[C12]	[C12—C13]	
C	0.578 (4)	—	-105.8 (3)	0.022 (4)	—	E
				[C9]		
D	0.585 (4)	—	-32.3 (4)	0.038 (6)	—	E
				[C9]		
E	0.974 (4)	90.5 (2)	56.9 (3)	0.042 (44)	—	$B_{2,5}$
				[C5]		
				0.043 (44)		
				[C6—C7]		

Data were corrected for Lorentz and polarization factors. The positions of the H atoms were generated from the assumed geometry and refined isotropically to values of the overall isotropic displacement parameters U_1 and U_2 of 0.081 (2) and 0.123 (9) $\text{\AA}^2$ (methyl H atoms), respectively. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and refined with *SHELX76* (Sheldrick, 1976). Conformational calculations were performed using the program *RING* (Párkányi, 1979). *CSU* (Vicković, 1988) was used to prepare the material for publication. All calculations were carried out on a PC/AT computer.

This work was supported by the Serbian Ministry of Science under Condensed Matter Project 0127.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, distances involving H atoms and torsion angles have been deposited with the IUCr (Reference: KA1072). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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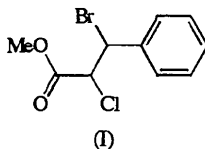
(Received 13 May 1994; accepted 14 July 1994)

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).

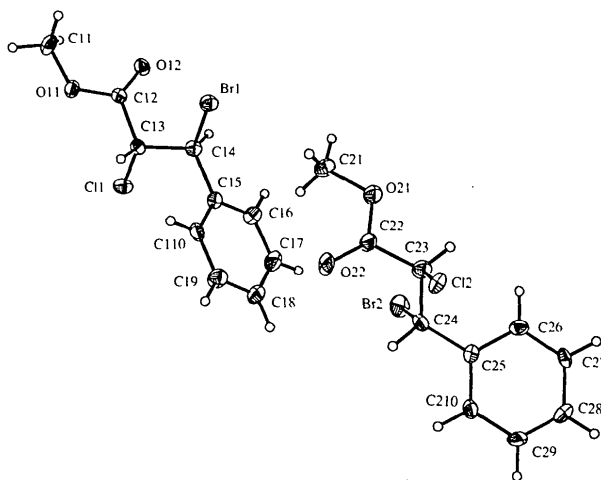


Fig. 1. ORTEP (Johnson, 1965) drawing of the two independent molecules of (I) showing displacement ellipsoids drawn at the 50% probability level.

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°