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Homochiral Methyl (*S*)-2-Benzenesulfonyloxy-4-bromo-4-methylpentanoate

Joanne P. Shaw, Eng Wui Tan and Allan G. Blackman

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

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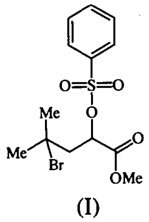
Abstract

The stereochemistry at position 2 of the title compound, $C_{13}H_{17}BrO_5S$, has been confirmed as (*S*).

Comment

The title compound, (I), was investigated as part of a study into the regioselective bromination of 2-hydroxy-4-methylpentanoic acid derivatives. This structure determination was undertaken to confirm that the stereochemistry at the 2 position (*i.e.* C5) was unaffected by the bromination reaction. The compound crystallized in the monoclinic space group $P2_1$ and refinement of the structure showed the presence of only one enantiomer. The stereochemistry at C5 was found to be (*S*), with the correct choice of 'handedness' verified by the value of the Flack (1983) parameter [χ = 0.01 (2)]. Close intermolecular contacts occur between Br1 and H10(2 − *x*, *y* + 1/2, 2 − *z*) [2.989 (7) Å], and O2 and H9(2 − *x*, *y* + 1/2, 2 − *z*) [2.438 (8) Å]. A similar regioselective (and stereoselective) bromination of a cinnamate derivative has been reported previously (Shaw, Tan & Blackman, 1995).



(I)

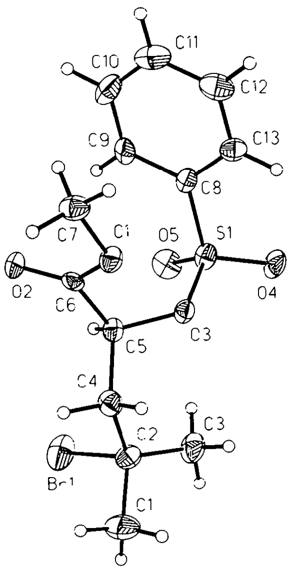


Fig. 1. *ORTEP* (Johnson, 1965) drawing of (I) showing displacement ellipsoids drawn at the 50% probability level.

Experimental

A mixture of methyl (*S*)-2-benzenesulfonyloxy-4-methylpentanoate (0.32 g, 1.1 mmol) and *N*-bromosuccinimide (0.24 g, 1.3 mmol) in CCl_4 (40 ml) was heated at reflux under nitrogen for 8 h, the reaction being 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 dichloromethane and hexane (Shaw & Tan, 1995). The compound crystallizes as large blocks which were used for the analysis, as smaller crystals could not be grown.

Crystal data

$C_{13}H_{17}BrO_5S$
M_r = 365.24
Monoclinic
$P2_1$
a = 10.352 (2) Å
b = 7.1190 (10) Å
c = 10.652 (2) Å
β = 104.89 (3)°
V = 758.6 (2) Å^3
Z = 2
D_x = 1.599 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 15 reflections
θ = 12–17°
μ = 2.862 mm^{-1}
T = 130 (2) K
Block
0.7 × 0.6 × 0.3 mm
Colourless

Data collection

Nicolet *R*3*M* diffractometer
ω scans
Absorption correction:
ψ scans
$T_{\min} = 0.590$, $T_{\max} = 0.907$
1520 measured reflections
1438 independent reflections
1214 observed reflections
$[I > 2\sigma(I)]$

$R_{\rm int} = 0.0664$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 8$
$l = -12 \rightarrow 12$
3 standard reflections
monitored every 97 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0400$
$wR(F^2) = 0.0829$
$S = 1.068$
1438 reflections
184 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.002$
$\Delta\rho_{\max} = 0.615$ e Å^{-3}
$\Delta\rho_{\min} = -0.504$ 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)

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

	x	y	z	$U_{\rm eq}$
Br1	0.89489 (7)	0.15280 (14)	0.55164 (6)	0.0430 (3)
S1	0.5895 (2)	−0.0750 (2)	0.8244 (2)	0.0229 (4)
O1	0.7964 (4)	0.2711 (7)	1.0333 (4)	0.0222 (11)
O2	0.9771 (4)	0.1211 (7)	1.0025 (4)	0.0251 (12)
O3	0.6447 (4)	0.1306 (7)	0.8099 (4)	0.0198 (10)
O4	0.4485 (4)	−0.0553 (8)	0.7762 (5)	0.0307 (13)
O5	0.6600 (5)	−0.2061 (8)	0.7659 (5)	0.0343 (13)
C1	0.7813 (8)	0.5164 (13)	0.5454 (7)	0.040 (2)
C2	0.7624 (7)	0.3239 (12)	0.6000 (6)	0.028 (2)
C3	0.6267 (6)	0.2451 (12)	0.5387 (6)	0.034 (2)
C4	0.7995 (7)	0.3369 (11)	0.7479 (6)	0.024 (2)
C5	0.7873 (5)	0.1597 (13)	0.8217 (5)	0.0212 (13)
C6	0.8659 (6)	0.1800 (11)	0.9635 (5)	0.0189 (14)
C7	0.8637 (7)	0.2963 (12)	1.1696 (6)	0.027 (2)
C8	0.6309 (6)	−0.1103 (9)	0.9921 (6)	0.021 (2)
C9	0.7515 (6)	−0.1997 (10)	1.0527 (7)	0.024 (2)
C10	0.7843 (7)	−0.2118 (12)	1.1874 (7)	0.037 (2)
C11	0.7013 (8)	−0.1408 (11)	1.2567 (7)	0.036 (2)
C12	0.5830 (8)	−0.0564 (12)	1.1960 (7)	0.035 (2)
C13	0.5467 (7)	−0.0415 (10)	1.0631 (6)	0.026 (2)

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

Br1—C2	1.999 (8)	C2—C3	1.497 (9)
S1—O5	1.424 (5)	C2—C4	1.526 (9)
S1—O4	1.424 (5)	C4—C5	1.509 (11)
S1—O3	1.593 (5)	C5—C6	1.527 (7)
S1—C8	1.745 (6)	C8—C13	1.383 (9)
O1—C6	1.329 (8)	C8—C9	1.402 (9)
O1—C7	1.452 (7)	C9—C10	1.390 (9)
O2—C6	1.195 (7)	C10—C11	1.365 (11)
O3—C5	1.464 (6)	C11—C12	1.368 (11)
C1—C2	1.521 (12)	C12—C13	1.372 (10)
O5—S1—O4	120.0 (3)	O3—C5—C4	106.9 (5)
O5—S1—O3	108.8 (3)	O3—C5—C6	111.6 (4)
O4—S1—O3	103.6 (3)	C4—C5—C6	110.0 (6)
O5—S1—C8	109.6 (3)	O2—C6—O1	126.1 (5)
O4—S1—C8	110.0 (3)	O2—C6—C5	122.1 (6)
O3—S1—C8	103.4 (3)	O1—C6—C5	111.8 (5)
C6—O1—C7	115.4 (5)	C13—C8—C9	121.4 (6)
C5—O3—S1	120.0 (5)	C13—C8—S1	119.1 (5)
C3—C2—C1	111.7 (6)	C9—C8—S1	119.5 (5)
C3—C2—C4	115.6 (6)	C10—C9—C8	117.1 (7)
C1—C2—C4	108.5 (6)	C11—C10—C9	121.0 (7)
C3—C2—Br1	106.8 (5)	C10—C11—C12	121.2 (7)
C1—C2—Br1	106.7 (5)	C11—C12—C13	119.7 (8)
C4—C2—Br1	107.2 (5)	C12—C13—C8	119.5 (7)
C5—C4—C2	117.1 (6)		

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1251). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Phenyl Carbamidonitrile

QING-CHUAN YANG,* DAN-MEI HUANG, HUI-YING CHEN AND YOU-QI TANG

Department of Chemistry, Peking University, Beijing 100871, People's Republic of China

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Abstract

The molecule of 3-phenyl-1-ureidonitrile, $C_8H_7N_3O$, has an extended conformation with a nearly planar carbamidonitrile group (to within 0.04 Å). The angle between the phenyl ring plane and the plane of the carbamidonitrile group is 27.6 (4)°. The carbamido N(7) and N(10) atoms are sp^2 hybridized. Except for the nitrile C(11)≡N(12) triple bond, the remaining C—N

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