N3—C4—C5	123.3 (2)	O5*D*—C5*D*—C4*D*	111.4 (3)
C6—C5—C4	115.8 (2)	O6*D*—C6*D*—C4*D*	109.8 (2)
O6*D*—C6—C5	126.7 (2)	O6*D*—C6*D*—C1*D*	105.5 (2)
O6*D*—C6—N1	111.6 (2)	C4*D*—C6*D*—C1*D*	107.1 (2)
C5—C6—N1	121.7 (2)	C6—O6*D*—C6*D*	109.8 (2)
N1—C1*D*—C2*D*	113.0 (2)		

C2—N1—C1*D*—C2*D*	−71.5 (3)
C1*D*—C2*D*—C3*D*—C4*D*	−38.4 (3)
C2—N1—C1*D*—C6*D*	176.3 (3)
C2*D*—C3*D*—C4*D*—C5*D*	162.6 (3)
C6—N1—C1*D*—C2*D*	111.9 (3)
C2*D*—C3*D*—C4*D*—C6*D*	37.2 (3)
C6—N1—C1*D*—C6*D*	−0.2 (3)
O3*D*—C3*D*—C4*D*—C5*D*	−75.4 (3)
N1—C6—O6*D*—C6*D*	−1.4 (3)
O3*D*—C3*D*—C4*D*—C6*D*	159.3 (2)
C5—C6—O6*D*—C6*D*	178.6 (3)
C3*D*—C4*D*—C5*D*—O5*D*	178.7 (3)
N1—C1*D*—C2*D*—C3*D*	−84.9 (3)
C6*D*—C4*D*—C5*D*—O5*D*	−62.0 (4)
C6*D*—C1*D*—C2*D*—C3*D*	24.6 (3)
C3*D*—C4*D*—C6*D*—C1*D*	−22.1 (3)
N1—C1*D*—C6*D*—C4*D*	116.4 (2)
C3*D*—C4*D*—C6*D*—O6*D*	92.0 (3)
N1—C1*D*—C6*D*—O6*D*	−0.5 (3)
C5*D*—C4*D*—C6*D*—C1*D*	−146.2 (3)
C2*D*—C1*D*—C6*D*—C4*D*	−1.5 (3)
C5*D*—C4*D*—C6*D*—O6*D*	−32.1 (3)
C2*D*—C1*D*—C6*D*—O6*D*	−118.4 (2)
C1*D*—C6*D*—O6*D*—C6	1.2 (3)
C1*D*—C2*D*—C3*D*—O3*D*	−158.0 (2)
C4*D*—C6*D*—O6*D*—C6	−113.9 (3)

Table 2. *Contact distances (Å)*

O*W*···O5*D*	2.793 (4)	N3···O3*D*^{iii}	2.871 (3)
O2···N4^{i}	2.893 (4)	O*W*···N3^{iv}	3.168 (4)
N4···O5*D*^{ii}	2.821 (4)	O*W*···O3*D*^{v}	2.821 (4)

Symmetry codes: (i) $1+x, y, z$; (ii) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iii) $2-x, -y, 2-z$; (iv) $1-x, -y, 1-z$; (v) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$.

Intensities were measured with a scan rate of 4° min^{-1} in 2θ and a scan width of $d(2\theta) = (1.2 + 0.15\tan\theta)°$. Background intensities were measured for 4 s at each side of a scan. The initial *E* map gave a partial structure around the pyrimidine skeleton. The positions of the remaining non-H atoms were located stepwise from the subsequent Fourier syntheses. The structure was refined by the block-diagonal least-squares procedure and the full-matrix least-squares refinement was carried out with *SHELXL*93 (Sheldrick, 1993).

Data collection: *Rigaku/AFC Diffractometer Control Software* (Rigaku Co. Ltd, 1997). Cell refinement: *Rigaku/AFC Diffractometer Control Software*. Data reduction: *UNICS* (Universal Crystallographic Computation Program System Osaka, 1979). Program(s) used to solve structure: *MULTAN*87 (Debaerdemaeker *et al.*, 1987). Molecular graphics: *ORTEP*II (Johnson, 1976) and *ORTEP*III (Burnett & Johnson, 1996).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1077). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 982–984

Methyl Ester of the Bioactive Metabolite of Thromboxane A2 Receptor Antagonist ON-579

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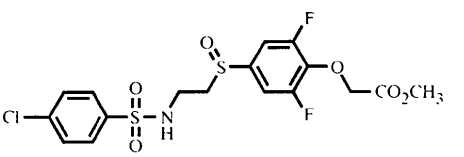
(*Received 10 July 1997; accepted 6 January 1998*)

Abstract

The title compound, methyl (*RS*)-{4-[2-(4-chlorophenylsulfonylamino)ethylsulfinyl]-2,6-difluorophenoxy}acetate, $C_{17}H_{16}ClF_2NO_6S_2$, crystallizes in space group $P2_1/c$. In the crystal, the enantiomeric molecules, related by a center of symmetry, form pairs joined by N—H···O hydrogen bonds.

Comment

In the course of our investigation of the pharmacodynamics of 4-[2-(4-chlorophenylsulfonylamino)ethylthio]-2,6-difluorophenoxyacetic acid (ON-579), which is a novel thromboxane A2 antagonist (Sato *et al.*, 1995), the corresponding sulfoxide, namely ON-579M2, was detected as a major and bioactive metabolite in animal urines. This paper reports the crystal structure of the synthetic racemate, (I), of the title compound.



(I)

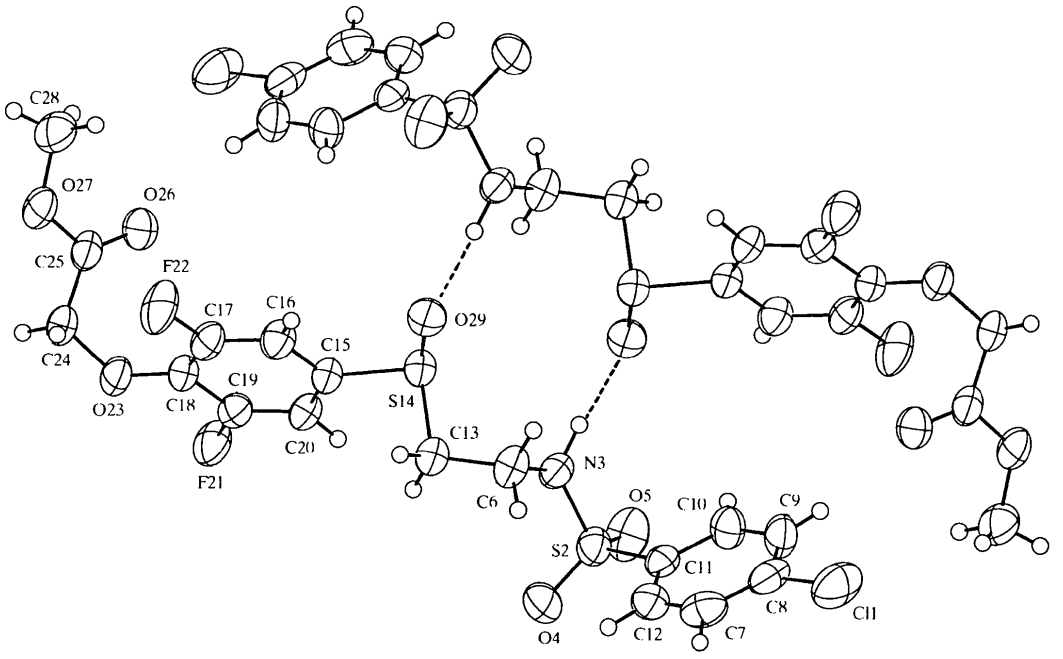


Fig. 1. The molecular structure of a pair of enantiomers showing 50% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976). Hydrogen bonds are shown as dotted lines.

As shown in Fig. 1, each enantiomer forms head-to-tail molecular pairs. An intermolecular hydrogen bond was observed between the H atom attached to the N atom (N3) of the sulfonamido group and the O atom (O29) of the sulfinyl group of the adjacent molecule [N3—H3 0.97 (2), H3···O29^{i} 1.88 (2), N3···O29^{i} 2.847 (2) Å and N3—H3···O29^{i} 177 (1)°; symmetry code: (i) $-x$, $-y$, $-z$].

Experimental

The title compound was prepared by oxidation of ON-579 methyl ester with *m*-CPBA (*m*-chloroperbenzoic acid), followed by hydrolysis with NaOH, and was crystallized from ethyl acetate.

Crystal data

$C_{17}H_{16}ClF_2NO_6S_2$
M_r = 467.90
Monoclinic
$P2_1/c$
a = 10.250 (4) Å
b = 19.183 (5) Å
c = 10.077 (5) Å
β = 99.05 (3)°
V = 1957 (1) Å^3
Z = 4
D_x = 1.588 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 20 reflections
θ = 27.5–30.0°
μ = 4.234 mm^{-1}
T = 288 K
Plate
0.4 × 0.3 × 0.3 mm
Yellow

Data collection

MacScience MXC18 diffractometer
$\theta/2\theta$ scans
Absorption correction: spherical (Mackay *et al.*, 1997)
T_{min} = 0.338, T_{max} = 0.468
3593 measured reflections
3218 independent reflections
2787 reflections with $I > 3\sigma(I)$
R_{int} = 0.034
θ_{max} = 63.81°
h = −11 → 11
k = 0 → 22
l = 0 → 11
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F
R = 0.052
wR = 0.048
S = 3.267
2787 reflections
327 parameters
Only coordinates of H atoms refined
Weights based on counting statistics
$(\Delta/\sigma)_{max}$ = 0.0834
$\Delta\rho_{max}$ = 0.74 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 1.904
Scattering factors from Waasmaier & Kirfel (1995)

Table 1. *Selected geometric parameters (Å, °)*

Cl1—C8	1.731 (2)	S14—O29	1.498 (2)
S2—N3	1.612 (2)	C15—C16	1.395 (3)
S2—O4	1.421 (2)	C15—C20	1.369 (3)
S2—O5	1.442 (2)	C16—C17	1.375 (3)
S2—C11	1.749 (2)	C17—C18	1.362 (3)
N3—C6	1.474 (3)	C17—F22	1.370 (2)
C6—C13	1.533 (3)	C18—C19	1.409 (3)
C7—C8	1.379 (3)	C18—O23	1.359 (2)
C7—C12	1.381 (3)	C19—C20	1.365 (3)
C8—C9	1.374 (3)	C19—F21	1.341 (2)
C9—C10	1.361 (3)	O23—C24	1.438 (3)
C10—C11	1.391 (3)	C24—C25	1.477 (3)
C11—C12	1.390 (3)	C25—O26	1.202 (2)
C13—S14	1.804 (2)	C25—O27	1.342 (2)
S14—C15	1.797 (2)	O27—C28	1.442 (3)

N3—S2—O4	108.3 (1)	C15—S14—O29	106.9 (1)
N3—S2—O5	105.9 (1)	S14—C15—C16	119.1 (2)
N3—S2—C11	106.8 (1)	S14—C15—C20	119.8 (2)
O4—S2—O5	119.9 (1)	C16—C15—C20	120.9 (2)
O4—S2—C11	108.0 (1)	C15—C16—C17	117.3 (2)
O5—S2—C11	107.2 (1)	C16—C17—C18	124.7 (2)
S2—N3—C6	119.6 (2)	C16—C17—F22	116.9 (2)
N3—C6—C13	110.2 (2)	C18—C17—F22	118.4 (2)
C8—C7—C12	119.8 (2)	C17—C18—C19	115.2 (2)
C11—C8—C7	120.1 (2)	C17—C18—O23	130.2 (2)
C11—C8—C9	119.3 (2)	C19—C18—O23	114.6 (2)
C7—C8—C9	120.6 (2)	C18—C19—C20	122.7 (2)
C8—C9—C10	119.9 (2)	C18—C19—F21	116.6 (2)
C9—C10—C11	120.7 (2)	C20—C19—F21	120.6 (2)
S2—C11—C10	119.5 (2)	C15—C20—C19	119.1 (2)
S2—C11—C12	121.1 (2)	C18—O23—C24	121.5 (2)
C10—C11—C12	119.2 (2)	O23—C24—C25	113.1 (2)
C7—C12—C11	119.7 (2)	C24—C25—O26	127.1 (2)
C6—C13—S14	109.6 (2)	C24—C25—O27	109.6 (2)
C13—S14—C15	97.7 (1)	O26—C25—O27	123.3 (2)
C13—S14—O29	105.4 (1)	C25—O27—C28	117.0 (2)

The *MAXUS* software package (Mackay *et al.*, 1997) was used throughout the analysis.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1071). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 984–985

1-(1,3-Benzothiazol-2-yl)-3,3,3-trichloro-2-propanol

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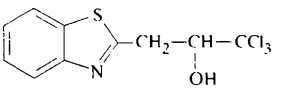
(*Received 26 October 1997; accepted 26 January 1998*)

Abstract

In the title compound, $C_{10}H_8Cl_3NOS$, the two planar benzene and thiazole rings are nearly coplanar. The dihedral angle between them is 1.20 (8)°. Strong intermolecular O—H···N hydrogen bonds link the molecules into infinite chains.

Comment

Derivatives of benzothiazole belong to a series of compounds which have especially remarkable biological properties (Varkonda *et al.*, 1985). We report here on the crystal structure of 1-(1,3-benzothiazol-2-yl)-3,3,3-trichloro-2-propanol, (I).



(I)

The mean value of 1.377 (4) Å for the six C—C bonds in the benzene ring is significantly short of the value of 1.397 Å expected from neutron diffraction (Bacon *et al.*, 1964). The two C—S distances [1.713 (3) and 1.733 (3) Å] in the thiazole ring have values intermediate between those reported for C_{sp^3}—S single [1.81 Å] and double [1.61 Å] bonds (Khan *et al.*, 1988). The bond distances C4—N1 [1.272 (3) Å] and N1—C5 [1.394 (3) Å] are in agreement with those found in related compounds (Bhatia *et al.*, 1991; Teo *et al.*, 1995). The benzothiazole nucleus adopts an almost planar conformation, with a dihedral angle between the individual planes of the benzene and thiazole rings of 1.20 (8)°. The crystal packing is dominated by O—H···N hydrogen bonds [O1···N1^{i} 2.807 (3), H1···N1^{i} 1.64 (4) Å, O—H···N^{i} 166 (2)°; symmetry code: (i) x, $-y$, $\frac{1}{2}+z$], which link the molecules into infinite chains in the **b** direction.

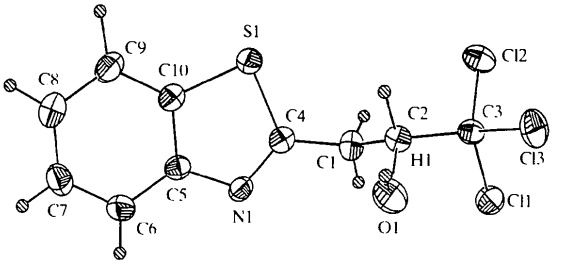


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title molecule, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii.

Experimental

Full details of the synthetic procedure have been published by Ettel *et al.* (1950). Single crystals were prepared by crystallization from ethanol.