Table 4. *Selected geometric parameters* (Å, °) *for* (17)

C(1)—O(13)	1.44 (1)	C(6)—C(7)	1.53 (1)
C(1)—C(12)	1.50 (2)	C(7)—O(15)	1.19 (1)
C(1)—C(2)	1.59 (1)	C(7)—C(8)	1.48 (1)
C(2)—C(3)	1.54 (2)	C(8)—C(9)	1.52 (1)
C(2)—C(8)	1.56 (1)	C(9)—O(13)	1.42 (1)
C(3)—O(14)	1.44 (1)	C(9)—C(10)	1.51 (1)
C(3)—C(4)	1.51 (2)	C(10)—C(11)	1.32 (1)
C(4)—O(5)	1.44 (2)	C(11)—C(12)	1.46 (1)
O(5)—C(6)	1.42 (1)	C(12)—O(16)	1.24 (1)
C(6)—O(14)	1.38 (1)		
O(13)—C(1)—C(12)	107.4 (9)	O(15)—C(7)—C(6)	121.3 (12)
O(13)—C(1)—C(2)	104.4 (9)	C(8)—C(7)—C(6)	114.7 (10)
C(12)—C(1)—C(2)	108.8 (10)	C(7)—C(8)—C(9)	108.8 (9)
C(3)—C(2)—C(8)	113.5 (9)	C(7)—C(8)—C(2)	115.2 (9)
C(3)—C(2)—C(1)	112.5 (9)	C(9)—C(8)—C(2)	102.0 (9)
C(8)—C(2)—C(1)	102.6 (9)	O(13)—C(9)—C(10)	108.0 (9)
O(14)—C(3)—C(4)	102.2 (10)	O(13)—C(9)—C(8)	104.4 (9)
O(14)—C(3)—C(2)	107.4 (9)	C(10)—C(9)—C(8)	111.6 (10)
C(4)—C(3)—C(2)	113.9 (10)	C(11)—C(10)—C(9)	120.8 (12)
O(5)—C(4)—C(3)	104.4 (12)	C(10)—C(11)—C(12)	118.3 (12)
C(6)—O(5)—C(4)	104.4 (10)	O(16)—C(12)—C(11)	121.9 (13)
O(14)—C(6)—O(5)	109.5 (10)	O(16)—C(12)—C(1)	122.8 (12)
O(14)—C(6)—C(7)	110.5 (9)	C(11)—C(12)—C(1)	115.2 (10)
O(5)—C(6)—C(7)	108.3 (11)	C(9)—O(13)—C(1)	102.9 (8)
O(15)—C(7)—C(8)	123.8 (12)	C(6)—O(14)—C(3)	100.6 (8)

Table 5. *Puckering parameters*

Compound	Ring Atoms*	Q (Å)	θ (°)	φ (°)
(9)	1,2,3,4,5,6	0.513	51.9	1.8
(9)	1,2,6a,1a,2a,6	0.610	0.0	0.0
(17)	14,3,4,5,6	0.397	–	188.0
(17)	14,6,7,8,2,3	0.623	136.1	175.9
(17)	13,1,2,8,9	0.447	–	164.6
(17)	13,1,12,11,10,9	0.610	54.3	6.6

* In the order of calculation (Cremer & Pople, 1975).

Preliminary photographs and the last collected shell of data indicated no significant data observable beyond θ = 18.5° for compound (17).

All H atoms were refined with isotropic displacement parameters; in (17), one common factor [final value 0.031 (11) $Å^2$] was used. All other atoms were refined with anisotropic displacement parameters. In (17), H(6), H(10) and H(11) were treated as riding atoms on C(6), C(10) and C(11), respectively, with C—H = 0.98 Å.

Programs used were *SHELXS*86 (Sheldrick, 1985) for direct-methods structure solution, *SHELXL*93 (Sheldrick, 1993) for refinement and *ORTEP*II (Johnson, 1971) for diagrams.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KH1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2*S*,4*R*,5*R*)-(–)-3-Isopropyl-4-methyl-5-phenyl-1,2,3-oxathiazolidine 2-Oxide

Paul D. Robinson

Department of Geology, Southern Illinois University, Carbondale, IL 62901, USA

Duy H. Hua, Jinshan Chen and Nadege Lagneau

Department of Chemistry, Kansas State University, Manhattan, KS 66506, USA

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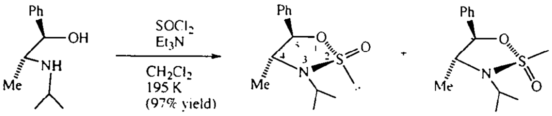
Abstract

The present structure determination, coupled with the known configuration of the starting amino alcohol, confirms the absolute configuration at the S atom of the title compound, $C_{12}H_{17}NO_2S$, and its 2*R* diastereoisomer. The five-membered oxathiazolidine 2-oxide ring has an envelope conformation with atom N3 deviating by 0.598 (3) Å from the least-squares plane formed by atoms O1, S2, C4 and C5. The bond lengths S2—O6 and S2—N3 are respectively shorter than the bond lengths S=O and S—N of sulfinamides.

Comment

In studies of the synthesis of chiral sulfoxides utilizing the displacement reactions of 1,2,3-oxathiazolidine 2-oxides with organometallic reagents (Benson & Snyder, 1991; Wudl & Lee, 1973), the title compound (1) and its 2*R* diastereoisomer (2) were prepared from the coupling reaction of (1*R*,2*R*)-(–)-2-(*N*-isopropylamino)-1-phenylpropanol, (3), thionyl chloride and triethylamine in methylene chloride.



(3) (1) (2)

The reaction provided a 97% yield of (1) and (2) in a ratio of 3:1. Fortunately, compound (1) is a white solid while (2) is an oil, so that (1) was easily isolated by crystallization from an ether/ethyl acetate solution to give large white prisms. On column chromatographic separation, the mother liquor gave pure (1) and (2). When the above coupling reaction was carried out at 195 K using 2,6-dimethylpyridine instead of triethylamine as base, compounds (1) and (2) were formed in a ratio of 1:3. In the previously reported preparation of the 4,5-*cis* 3-methyl analogue, *i.e.* (4*S*,5*R*)-3,4-dimethyl-5-phenyl-1,2,3-oxathiazolidine 2-oxide, utilizing triethylamine as base, the ratio of 2*S* to 2*R* isomers was 1:9 (Benson & Snyder, 1991). These conflicting results, along with the structural assignments of all 1,2,3-oxathiazolidine 2-oxides described so far, were based solely on the ^{1}H NMR chemical shift of the C5 H atom [the resonance of the C5 H atom of the 2*R* isomer (S═O and the H atom are *cis* with respect to each other) appears at lower field compared with that of the 2*S* isomer]. It was necessary, therefore, to determine the stereochemistry at the S atom of (1) and (2) unambiguously by single-crystal X-ray analysis. Furthermore, unlike the stereochemistry of the structures published by Benson & Snyder (1991), the C4 methyl and C5 phenyl groups of both compounds (1) and (2) are *trans* and there is an isopropyl group on the N atom.

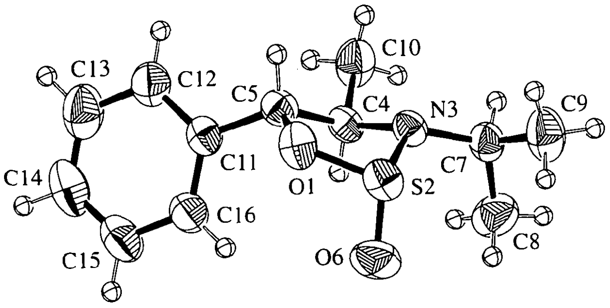


Fig. 1. The molecular configuration and atom-numbering scheme of the title compound with displacement ellipsoids at the 50% probability level. H atoms are shown as unlabeled isotropic spheres of arbitrary radii.

A view of molecule (1) with the numbering scheme adopted is given in Fig. 1. The configurations at atoms C4 and C5 are the same as those of the starting amino alcohol (3) (Hua, Chan-Yu-King, Ostrander & McKie, 1989), the absolute configuration of which had been determined, thereby establishing the absolute configuration of (1). The five-membered oxathiazolidine ring has the envelope conformation with N3 deviating by 0.598 (3) Å from the least-squares plane formed by atoms O1, S2, C4 and C5. The N3 isopropyl group is *trans* to the C4 methyl group, with a C7—N3—C4—C10 torsion angle of −57.6 (3)°. The bond lengths S2—O6 and S2—N3 of 1.456 (2) and 1.676 (2) Å are respectively shorter than the S═O [1.484 (5) Å] and S—N [1.714 (4) Å] distances of sulfinamides (Robinson, Hua, Chen & Saha, 1991).

Experimental

The title compound, (1), and its 2*R* diastereoisomer, (2), displayed satisfactory ^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz), UV, IR and low-resolution mass spectra (EI and CI). The synthesis of (1) and (2) was achieved by adding thionyl chloride (0.38 ml, 5.2 mmol) dropwise to a cold (195 K) solution of (1*R*,2*R*)-(−)-2-(*N*-isopropylamino)-1-phenylpropanol, (3), (1.0 g, 5.2 mmol) in triethylamine (1.8 ml) and methylene chloride (20 ml) under argon. After the solution was stirred at 195 K for 1 h, water was added and the mixture was extracted twice with methylene chloride. The methylene chloride layer was isolated, dried ($MgSO_4$) and concentrated to give 1.243 g of crude product. The ^{1}H NMR spectrum of this product indicated it to be a mixture of (1) and (2) (ratio 3:1). Crystallization of the crude product from ether/ethyl acetate (1:1) solution gave 0.605 g of pure (1); m.p. 400–401 K, $[\alpha]_D^{22} = -99.4°$ (c = 1.0, ethyl acetate). The mother liquor was concentrated and column chromatographed on silica gel, using hexane and ether as eluant, to give 0.302 g of (1) (R_f = 0.6; toluene:ether = 9:1) and 0.298 g of (2) (R_f = 0.4). Compound (2) was an oil, $[\alpha]_D^{22}$ = +61.8° (c = 1, ethyl acetate).

Crystal data

$C_{12}H_{17}NO_2S$
M_r = 239.33
Orthorhombic
$P2_12_12_1$
a = 7.136 (1) Å
b = 7.221 (1) Å
c = 24.405 (3) Å
V = 1257.7 (5) Å^3
Z = 4
D_x = 1.264 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 12.6–13.3°
μ = 0.232 mm^{-1}
T = 296 K
Equant fragment
0.46 × 0.41 × 0.30 mm
Colorless

Data collection

Rigaku AFC-5S diffractometer
ω scans (rate 4° min^{-1} in ω)
Absorption correction: none
1341 measured reflections
1341 independent reflections
1219 observed reflections [$I > \sigma(I)$]

θ_{max} = 25°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 29$
3 standard reflections monitored every 100 reflections
intensity decay: 0.6%

Refinement

Refinement on F
R = 0.031
wR = 0.036
S = 1.54
1219 reflections
145 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

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

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

	x	y	z	U_{eq}
S2	0.8883 (1)	0.25851 (9)	0.54815 (2)	0.0511 (2)
O1	1.0470 (3)	0.1106 (2)	0.56745 (7)	0.0528 (4)
O6	0.7320 (3)	0.2339 (3)	0.58579 (8)	0.073 (1)
N3	1.0247 (3)	0.4371 (2)	0.56643 (7)	0.040 (1)
C4	1.1289 (3)	0.3847 (3)	0.61623 (8)	0.040 (1)
C5	1.1920 (4)	0.1889 (3)	0.60281 (9)	0.043 (1)
C7	0.9432 (3)	0.6263 (3)	0.5636 (1)	0.047 (1)
C8	0.8028 (5)	0.6700 (4)	0.6084 (1)	0.069 (1)
C9	0.8626 (4)	0.6594 (4)	0.5066 (1)	0.071 (1)
C10	1.2914 (4)	0.5122 (4)	0.6281 (1)	0.063 (1)
C11	1.2221 (4)	0.0686 (3)	0.6523 (1)	0.043 (1)
C12	1.4011 (4)	0.0235 (4)	0.6689 (1)	0.057 (1)
C13	1.4263 (5)	−0.0773 (4)	0.7172 (1)	0.075 (1)
C14	1.2765 (6)	−0.1313 (4)	0.7479 (1)	0.075 (1)
C15	1.0977 (5)	−0.0897 (4)	0.7307 (1)	0.068 (1)
C16	1.0709 (3)	0.0104 (4)	0.6832 (1)	0.052 (1)

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

S2—O1	1.626 (2)	C7—C8	1.515 (4)
S2—O6	1.456 (2)	C7—C9	1.525 (4)
S2—N3	1.676 (2)	C11—C12	1.379 (4)
O1—C5	1.461 (3)	C11—C16	1.382 (3)
N3—C4	1.474 (3)	C12—C13	1.397 (4)
N3—C7	1.487 (3)	C13—C14	1.362 (4)
C4—C5	1.520 (3)	C14—C15	1.376 (4)
C4—C10	1.508 (3)	C15—C16	1.381 (4)
C5—C11	1.503 (3)		
O1—S2—O6	105.7 (1)	C4—C5—C11	114.0 (2)
O1—S2—N3	91.37 (9)	N3—C7—C8	114.6 (2)
O6—S2—N3	111.8 (1)	N3—C7—C9	109.5 (2)
S2—O1—C5	114.2 (1)	C8—C7—C9	112.1 (2)
S2—N3—C4	108.4 (1)	C5—C11—C12	120.3 (2)
S2—N3—C7	117.9 (2)	C5—C11—C16	120.2 (2)
C4—N3—C7	118.1 (2)	C12—C11—C16	119.4 (2)
N3—C4—C5	102.2 (2)	C11—C12—C13	119.4 (3)
N3—C4—C10	112.9 (2)	C12—C13—C14	120.8 (3)
C5—C4—C10	112.4 (2)	C13—C14—C15	119.9 (2)
O1—C5—C4	106.1 (2)	C14—C15—C16	119.9 (3)
O1—C5—C11	110.6 (2)	C11—C16—C15	120.7 (3)

The absolute configuration was deduced from the known configuration of the starting material, (3), and confirmed by refinement of the alternative enantiomer (R = 0.032, wR = 0.038). Weak reflections [$I < 10\sigma(I)$] were rescanned (maximum of three rescans) and the counts accumulated to improve accuracy. The H atoms were placed at assumed positions (C—H = 0.95 Å, $U = 1.2U_{eq}$ of the associated atom) and fixed.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *TEXSAN ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data, and a stereoscopic packing diagram have been deposited with the IUCr (Reference: FG1054). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,3-Diphenylmaleic Anhydride, an Analogue of *cis*-Stilbene

Minjoong Yoon,* Yong Hee Kim and Dae Won Cho

Department of Chemistry, Chungnam National University, Taejeon 305-764, Korea

Il-Hwan Suh,* Jin-Ho Lee, Bo-Young Ryu and Jeong-Ran Park

Department of Physics, Chungnam National University, Taejeon 305-764, Korea

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Abstract

The asymmetric unit of the title structure, $C_{16}H_{10}O_3$, consists of two molecules which have similar conformations. Each of the two pentagonal rings is planar, while the average dihedral angle between the two phenyl rings within each molecule is 57.4°. The molecules are held together by van der Waals forces, with the shortest intermolecular distance being 2.914 (11) Å for $O(3')\cdots C(4)(\frac{1}{2}-x,\ -y,\ \frac{1}{2}+z)$.

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