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Structure Determination of (1*S*;3*S*)-3-Methoxy-1-phenyl-2-(*p*-tolylsulfinyl)-2-propen-1-ol (C₁₇H₁₈O₃S)

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Abstract

The structure determination of C₁₇H₁₈O₃S based on X-ray single-crystal diffraction shows the *S* absolute configuration of the 1C atom and the *E* geometry of the double bond.

Comment

Optically active sulfoxides are used with great success in stereoselective syntheses (Posner, 1988). In connection with our interest in using chiral vinyl sulfoxide in asymmetric synthesis, we sought new strategies for constructing systems containing a vinyl sulfoxide moiety as chiral center.

To this end, we recently reported the first method for obtaining enantiomerically pure (*R*)-2-methoxyvinyl *p*-tolyl sulfoxide (Guillot & Maignan, 1991) and we tried direct deprotonation by trapping the resulting vinyl an-

ion with aldehyde. Condensation with benzaldehyde produced a 20:80 mixture of readily separable diastereoisomeric alcohols. The structure of these alcohols could not be assigned by analysis of their ¹H NMR data. In order to obtain definitive information on the absolute configuration of the C atom bearing the alcohol and the stereochemistry of the double bond, we tried to obtain single crystals with a view to determination of their structure by X-ray diffraction.

This paper deals with the X-ray structure determination of (1*S*;3*S*)-3-methoxy-1-phenyl-2-(*p*-tolylsulfinyl)-2-propen-1-ol (C₁₇H₁₈O₃S). The perspective view obtained reveals the *S* absolute configuration of the 1C atom (C9 in the structure description) and the *E* geometry of the double bond.

Experimental

Crystal data

C₁₇H₁₈O₃S
M_r = 302.39
 Orthorhombic
*P*2₁2₁2₁
a = 7.0123 (5) Å
b = 7.5297 (9) Å
c = 29.094 (2) Å
V = 1536 (1) Å³
Z = 4
D_x = 1.31 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 32 reflections
 θ = 14–15.5°
 μ = 0.216 mm⁻¹
T = 293 K
 Plate
 0.361 × 0.228 × 0.095 mm
 Colorless

Data collection

Stoe Siemens AED-2 diffractometer
 ω/2θ scans
 Absorption correction: none
 4208 measured reflections
 2206 independent reflections
 1210 observed reflections
 [*I* > 3.0σ(*I*)]

θ_{max} = 27.5°
 |*h*| = 0 → 9
 |*k*| = 0 → 9
 |*l*| = 0 → 37
 (two centrosymmetric sets were recorded)
 3 standard reflections
 frequency: 60 min
 intensity variation: 5%

Refinement

Refinement on *F*
 Final *R* = 0.028
wR = 0.029
 1210 reflections
 234 parameters
 All H-atom parameters refined with a common thermal parameter
w = 2.193/[σ²(*F*) + 0.0002*F*²]
 (Δ/σ)_{max} = 0.002

Δρ_{max} = 0.161 e Å⁻³
 Δρ_{min} = -0.179 e Å⁻³
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) for S and O atoms; from Cromer & Mann (1968) for C and H atoms

Data collection: *DIF4* (Stoe & Co., 1987). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Co., 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985; Robinson & Sheldrick, 1988) option *PATT*. Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP* (Johnson, 1965).

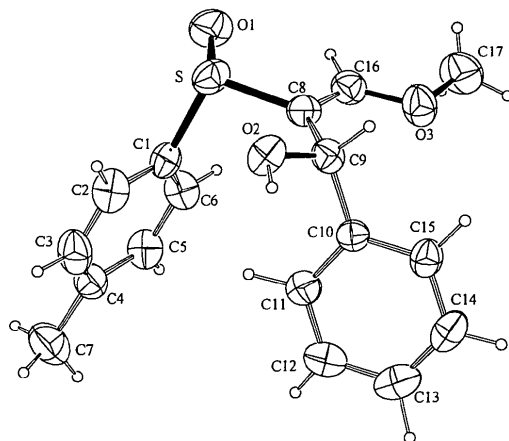


Fig. 1. ORTEP plot of C₁₇H₁₈O₃S. For clarity, the thermal parameters of the H atoms were divided by ten.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
S1	0.0606 (1)	0.9747 (1)	0.1036 (1)	0.0446 (5)
O1	0.2625 (3)	1.0043 (4)	0.1195 (1)	0.059 (2)
O2	-0.3423 (4)	0.9542 (3)	0.1068 (1)	0.047 (1)
O3	-0.0605 (4)	0.8572 (4)	0.2325 (1)	0.052 (2)
C1	0.0638 (5)	0.7695 (4)	0.0723 (1)	0.040 (2)
C2	0.1624 (5)	0.6231 (5)	0.0890 (1)	0.046 (2)
C3	0.1690 (5)	0.4697 (6)	0.0631 (1)	0.047 (2)
C4	0.0792 (5)	0.4613 (6)	0.0202 (1)	0.046 (2)
C5	-0.0170 (6)	0.6087 (7)	0.0053 (1)	0.053 (2)
C6	-0.0255 (6)	0.7626 (6)	0.0303 (1)	0.050 (2)
C7	0.0943 (9)	0.2954 (8)	-0.0089 (2)	0.069 (3)
C8	-0.0694 (5)	0.9065 (4)	0.1538 (1)	0.037 (2)
C9	-0.2866 (5)	0.8912 (5)	0.1505 (1)	0.036 (2)
C10	-0.3618 (5)	0.7053 (4)	0.1597 (1)	0.034 (2)
C11	-0.4812 (5)	0.6749 (5)	0.1967 (1)	0.040 (2)
C12	-0.5620 (6)	0.5106 (5)	0.2042 (1)	0.052 (2)
C13	-0.5226 (6)	0.3713 (6)	0.1745 (2)	0.057 (3)
C14	-0.4025 (6)	0.4003 (6)	0.1374 (1)	0.053 (2)
C15	-0.3238 (5)	0.5653 (5)	0.1303 (1)	0.045 (2)
C16	0.0287 (5)	0.8948 (5)	0.1925 (1)	0.044 (2)
C17	0.0577 (9)	0.8626 (8)	0.2727 (2)	0.063 (3)

Table 2. Geometric parameters (\AA , $^\circ$)

S1—O1	1.506 (2)	C16—O3	1.351 (4)
S1—C1	1.794 (4)	O3—C17	1.434 (7)
S1—C8	1.797 (3)	C8—C9	1.530 (5)
C1—C2	1.389 (5)	C9—O2	1.412 (4)
C2—C3	1.380 (5)	C9—C10	1.520 (5)
C3—C4	1.398 (4)	C10—C11	1.383 (5)
C4—C5	1.371 (6)	C11—C12	1.378 (5)
C5—C6	1.370 (6)	C12—C13	1.387 (6)
C6—C1	1.374 (5)	C13—C14	1.386 (6)
C4—C7	1.513 (7)	C14—C15	1.375 (6)
C8—C16	1.322 (4)	C15—C10	1.382 (5)
O1—S1—C1	105.8 (2)	C9—C8—C16	124.5 (3)
S1—C1—C6	118.6 (3)	C8—C16—O3	120.4 (3)
S1—C1—C2	120.8 (2)	C16—O3—C17	115.4 (3)
C1—C2—C3	119.3 (3)	C8—C9—O2	107.8 (3)
C2—C3—C4	120.7 (4)	O2—C9—C10	111.8 (3)
C4—C5—C6	122.6 (3)	C8—C9—C10	113.8 (3)
C5—C6—C1	119.0 (4)	C9—C10—C15	121.8 (3)
C6—C1—C2	120.5 (3)	C9—C10—C11	120.0 (3)
C3—C4—C7	120.5 (4)	C10—C11—C12	121.4 (3)
C7—C4—C5	121.7 (3)	C11—C12—C13	119.9 (3)
C5—C4—C3	117.9 (4)	C12—C13—C14	119.1 (4)
O1—S1—C8	105.0 (2)	C13—C14—C15	120.1 (4)
S1—C8—C16	116.6 (3)	C14—C15—C10	121.4 (3)
S1—C8—C9	118.4 (2)		

A colorless parallelepipedic crystal, obtained by slow evaporation at room temperature of a pentane/ether solution, was isolated. Its quality was tested with Laue photographs.

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55782 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1027]

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Structure of 5-Amino-1,3,4-thiadiazole-2-sulfonamide, an Inhibitor of the Enzyme Carbonic Anhydrase

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Abstract

Bond lengths and angles indicate a strong interaction between the NH_2 group and the thiadiazole ring. The sulfonamido moiety adopts a distorted arrangement around the S atom. Structural features of the compound are compared with those of acetazolamide, *N*-[5-sulfamoyl-1,3,4-thiadiazol-2(3*H*)-ylidene]-acetamide (H_2acm), methazolamide, *N*-[3-methyl-5-sulfamoyl-1,3,4-thiadiazol-2(3*H*)-ylidene]acetamide (Hmacm), and 5-amino-1,3,4-thiadiazole-2-thiol (Hatm).