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A Dimeric (Phenylsulfonyl)oxazolidine

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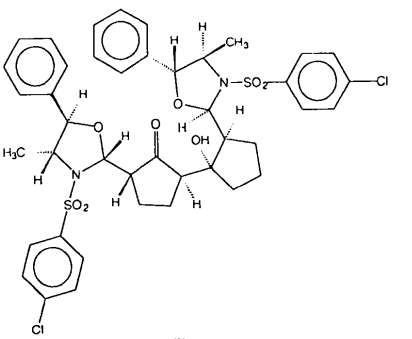
Abstract

We report the crystal structure of (1*R*,3*R*)-2[(2*R*,4*R*,-5*R*)-3-(*p*-chlorophenylsulfonyl)-4-methyl-5-phenyl-1,3-oxazolidin-2-yl]-5-{2-(1*R*,2*S*)-[(2*R*,4*R*,5*R*)-3-(*p*-chlorophenylsulfonyl)-4-methyl-5-phenyl-1,3-oxazolidin-2-yl]-1-hydroxycyclopentyl}-1-cyclopentanone, $C_{42}H_{44}Cl_2N_2O_8S_2$ (1). The chlorophenyl-sulfonyl substituent is *cis* to the phenyl ring on each oxazolidine heterocycle. Each chlorophenyl-sulfonyl substituent adopts a folded conformation by which the aromatic ring shields the heterocycle. The oxazolidine rings have envelope conformations with C(3) and O(17) out of their respective planes. An intramolecular hydrogen bond exists between the hydroxyl group O(11) and the ketone O(7) [O(11)···O(7) 2.840 Å, O(11)—H(11)···O(7) 134.0°].

Comment

The title compound (I) is the product of a diastereoselective side reaction occurring during the preparation of the trimethylsilyl enol ether from the corresponding 2-(2-oxocyclopentyl)-5-phenyl-1,3-oxazolidine (Conde-Frieboes & Hoppe, 1992). It is generated as a pure diastereomer bearing ten stereogenic centres, whose relative configuration was determined using NOE derived distance restraints and distance–geometry calculations (Crippen & Havel, 1988; Mierke & Reggelin, 1992; Reggelin, Köck, Conde-Frieboes & Mierke, 1994). The X-ray structure analysis was carried out in order to confirm the stereochemical results from these calculations.



(I)

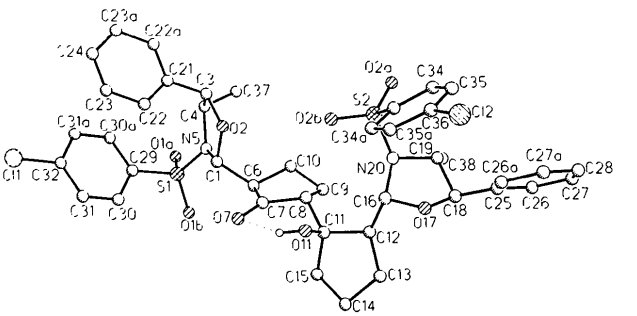


Fig. 1. Perspective view of the title compound with the atom-numbering scheme (only the hydroxy H atom is shown).

Experimental

Crystal data

$C_{42}H_{44}Cl_2N_2O_8S_2$
M_r = 839.8
Monoclinic
*C*2
a = 30.314 (4) Å
b = 7.372 (1) Å
c = 23.336 (4) Å
β = 126.31 (1)°
V = 4202 (1) Å^3
Z = 4
D_x = 1.327 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 30–40°
μ = 2.76 mm^{-1}
T = 293 K
Transparent block
0.3 × 0.3 × 0.1 mm
Colourless
Crystal source: from methanol/chloroform

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: empirical
T_{min} = 0.75, T_{max} = 1.00
4993 measured reflections
3342 independent reflections
3144 observed reflections [$F > 4\sigma(F)$]
R_{int} = 0.020
θ_{max} = 60°
h = −34 → 11
k = −8 → 0
l = −24 → 26
3 standard reflections
frequency: 92 min
intensity variation: none

Refinement

Refinement on *F*
R = 0.038
wR = 0.048
S = 1.86
Extinction correction: empirical
$F^* = F[1 + (0.002\chi \times F^2/\sin 2\theta)]^{-1/4}$

3144 reflections
514 parameters
$w = 1/[\sigma^2(F) + 0.000325F^2]$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Extinction coefficient:
$\chi = 0.0010$ (1)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
C(1)	0.6557 (1)	0.6220 (6)	0.5594 (2)	0.048 (2)
O(2)	0.6741 (1)	0.6354 (5)	0.5164 (1)	0.067 (1)
C(3)	0.7201 (1)	0.7527 (7)	0.5476 (2)	0.053 (2)
C(4)	0.7171 (2)	0.8770 (6)	0.5971 (2)	0.053 (2)
C(37)	0.6927 (2)	1.0606 (7)	0.5651 (3)	0.077 (3)
N(5)	0.6813 (1)	0.7746 (5)	0.6095 (1)	0.049 (1)
S(1)	0.6992 (1)	0.7500†	0.6899 (1)	0.058 (1)
O(1*A*)	0.7187 (1)	0.9232 (5)	0.7239 (2)	0.076 (2)
O(1*B*)	0.6539 (1)	0.6662 (6)	0.6832 (1)	0.074 (2)
C(29)	0.7540 (2)	0.5960 (7)	0.7342 (2)	0.057 (2)
C(30)	0.7448 (2)	0.4138 (8)	0.7388 (2)	0.069 (3)
C(30*A*)	0.8072 (2)	0.6537 (8)	0.7626 (2)	0.070 (2)
C(31)	0.7865 (2)	0.2916 (8)	0.7696 (3)	0.079 (3)
C(31*A*)	0.8489 (2)	0.5298 (10)	0.7927 (2)	0.084 (3)
C(32)	0.8388 (2)	0.3504 (9)	0.7964 (3)	0.079 (3)
Cl(1)	0.8920 (1)	0.1966 (3)	0.8358 (1)	0.124 (1)
C(21)	0.7737 (1)	0.6513 (6)	0.5833 (2)	0.049 (2)
C(22)	0.7791 (2)	0.4701 (7)	0.6018 (2)	0.064 (3)
C(22*A*)	0.8191 (2)	0.7420 (9)	0.5987 (3)	0.077 (3)
C(23)	0.8296 (3)	0.3846 (10)	0.6363 (3)	0.091 (4)
C(23*A*)	0.8697 (2)	0.6541 (12)	0.6330 (3)	0.095 (4)
C(24)	0.8742 (3)	0.4755 (13)	0.6518 (3)	0.098 (4)
C(6)	0.5935 (1)	0.6273 (6)	0.5121 (2)	0.048 (2)
C(7)	0.5697 (1)	0.4581 (6)	0.4665 (2)	0.048 (2)
O(7)	0.5860 (1)	0.3059 (5)	0.4885 (2)	0.069 (2)
C(8)	0.5231 (1)	0.5097 (6)	0.3914 (2)	0.048 (2)
C(9)	0.5087 (2)	0.7024 (6)	0.3993 (2)	0.057 (2)
C(10)	0.5647 (2)	0.7831 (6)	0.4594 (2)	0.058 (2)
C(11)	0.4784 (1)	0.3628 (6)	0.3547 (2)	0.047 (2)
O(11)	0.5017 (1)	0.2031 (4)	0.3464 (1)	0.057 (1)
C(12)	0.4272 (1)	0.4087 (6)	0.2797 (2)	0.049 (2)
C(13)	0.3851 (1)	0.2676 (8)	0.2667 (2)	0.065 (2)
C(14)	0.4024 (2)	0.2041 (8)	0.3399 (2)	0.076 (3)
C(15)	0.4538 (2)	0.3132 (7)	0.3936 (2)	0.061 (2)
C(16)	0.4368 (1)	0.4056 (6)	0.2225 (2)	0.050 (2)
O(17)	0.3885 (1)	0.3690 (5)	0.1533 (1)	0.063 (1)
C(18)	0.3602 (2)	0.5350 (7)	0.1245 (2)	0.065 (2)
C(19)	0.4050 (2)	0.6791 (7)	0.1541 (2)	0.071 (2)
C(38)	0.3945 (3)	0.8534 (9)	0.1800 (4)	0.113 (4)
N(20)	0.4551 (1)	0.5862 (6)	0.2142 (2)	0.056 (2)
S(2)	0.5084 (1)	0.5896 (2)	0.2122 (1)	0.064 (1)
O(2*A*)	0.5113 (2)	0.7709 (6)	0.1927 (2)	0.092 (2)
O(2*B*)	0.5529 (1)	0.5144 (6)	0.2774 (1)	0.076 (2)
C(33)	0.4936 (2)	0.4428 (8)	0.1433 (2)	0.067 (2)
C(34)	0.4673 (2)	0.5045 (10)	0.0750 (2)	0.086 (3)
C(34*A*)	0.5041 (2)	0.2574 (8)	0.1577 (3)	0.073 (3)
C(35)	0.4495 (3)	0.3771 (15)	0.0210 (3)	0.116 (4)
C(35*A*)	0.4865 (2)	0.1384 (10)	0.1041 (3)	0.093 (4)
C(36)	0.4587 (3)	0.2001 (13)	0.0370 (4)	0.104 (4)
Cl(2)	0.4309 (1)	0.0487 (5)	−0.0334 (1)	0.193 (2)
C(25)	0.3232 (2)	0.5228 (9)	0.0437 (2)	0.074 (2)
C(26)	0.2822 (2)	0.6451 (12)	0.0053 (3)	0.117 (4)
C(26*A*)	0.3296 (2)	0.3926 (11)	0.0092 (2)	0.096 (3)
C(27)	0.2493 (3)	0.6414 (14)	−0.0684 (3)	0.137 (4)
C(27*A*)	0.2959 (2)	0.3956 (14)	−0.0656 (3)	0.118 (4)
C(28)	0.2571 (3)	0.5175 (16)	−0.1034 (3)	0.127 (4)

† Coordinate fixed to define origin.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—O(2)	1.410 (6)	C(1)—N(5)	1.470 (5)
C(1)—C(6)	1.519 (5)	O(2)—C(3)	1.422 (5)
C(3)—C(4)	1.519 (7)	C(3)—C(21)	1.513 (6)
C(4)—C(37)	1.511 (7)	C(4)—N(5)	1.485 (7)
N(5)—S(1)	1.624 (4)	S(1)—O(1*A*)	1.433 (4)
S(1)—O(1*B*)	1.429 (4)	S(1)—C(29)	1.756 (4)
C(29)—C(30)	1.389 (8)	C(29)—C(30*A*)	1.399 (6)
C(30)—C(31)	1.361 (7)	C(30*A*)—C(31*A*)	1.368 (8)
C(31)—C(32)	1.385 (8)	C(31*A*)—C(32)	1.372 (10)
C(32)—Cl(1)	1.725 (6)	C(21)—C(22)	1.383 (7)
C(21)—C(22*A*)	1.373 (7)	C(22)—C(23)	1.390 (8)
C(22*A*)—C(23*A*)	1.400 (8)	C(23)—C(24)	1.353 (11)
C(23*A*)—C(24)	1.369 (13)	C(6)—C(7)	1.516 (6)
C(6)—C(10)	1.524 (6)	C(7)—O(7)	1.211 (6)
C(7)—C(8)	1.510 (4)	C(8)—C(9)	1.528 (6)
C(8)—C(11)	1.537 (5)	C(9)—C(10)	1.544 (5)
C(11)—O(11)	1.443 (6)	C(11)—C(12)	1.540 (4)
C(11)—C(15)	1.522 (8)	C(12)—C(13)	1.530 (7)
C(12)—C(16)	1.526 (7)	C(13)—C(14)	1.534 (7)
C(14)—C(15)	1.527 (6)	C(16)—O(17)	1.421 (4)
C(16)—N(20)	1.499 (6)	O(17)—C(18)	1.415 (6)
C(18)—C(19)	1.528 (7)	C(18)—C(25)	1.522 (5)
C(19)—C(38)	1.530 (10)	C(19)—N(20)	1.490 (5)
N(20)—S(2)	1.643 (4)	S(2)—O(2*A*)	1.431 (5)
S(2)—O(2*B*)	1.419 (3)	S(2)—C(33)	1.763 (6)
C(33)—C(34)	1.371 (7)	C(33)—C(34*A*)	1.398 (8)
C(34)—C(35)	1.398 (11)	C(34*A*)—C(35*A*)	1.352 (9)
C(35)—C(36)	1.340 (14)	C(35*A*)—C(36)	1.344 (10)
C(36)—Cl(2)	1.737 (9)	C(25)—C(26)	1.357 (8)
C(25)—C(26*A*)	1.339 (10)	C(26)—C(27)	1.387 (8)
C(26*A*)—C(27*A*)	1.407 (7)	C(27)—C(28)	1.334 (14)
C(27*A*)—C(28)	1.321 (12)		
O(2)—C(1)—N(5)	105.6 (3)	O(2)—C(1)—C(6)	108.9 (3)
N(5)—C(1)—C(6)	113.7 (4)	C(1)—O(2)—C(3)	111.1 (3)
O(2)—C(3)—C(4)	105.8 (4)	O(2)—C(3)—C(21)	112.9 (4)
C(4)—C(3)—C(21)	114.1 (3)	C(3)—C(4)—C(37)	113.4 (4)
C(3)—C(4)—N(5)	102.8 (3)	C(37)—C(4)—N(5)	110.7 (4)
C(1)—N(5)—C(4)	109.6 (4)	C(1)—N(5)—S(1)	119.5 (3)
C(4)—N(5)—S(1)	119.9 (2)	N(5)—S(1)—O(1*A*)	106.6 (2)
N(5)—S(1)—O(1*B*)	106.2 (2)	O(1*A*)—S(1)—O(1*B*)	120.1 (3)
N(5)—S(1)—C(29)	107.8 (2)	O(1*A*)—S(1)—C(29)	108.3 (2)
O(1*B*)—S(1)—C(29)	107.3 (2)	S(1)—C(29)—C(30)	120.5 (3)
S(1)—C(29)—C(30*A*)	120.4 (4)	C(30)—C(29)—C(30*A*)	119.1 (4)
C(29)—C(30)—C(31)	121.0 (5)	C(29)—C(30*A*)—C(31*A*)	119.7 (5)
C(30)—C(31)—C(32)	119.2 (6)	C(30*A*)—C(31*A*)—C(32)	120.2 (5)
C(31)—C(32)—C(31*A*)	120.8 (5)	C(31)—C(32)—Cl(1)	119.6 (5)
C(31*A*)—C(32)—Cl(1)	119.6 (4)	C(3)—C(21)—C(22)	122.6 (4)
C(3)—C(21)—C(22*A*)	119.1 (4)	C(22)—C(21)—C(22*A*)	118.3 (4)
C(21)—C(22)—C(23)	120.3 (5)	C(21)—C(22*A*)—C(23*A*)	120.9 (6)
C(22)—C(23)—C(24)	121.0 (7)	C(22*A*)—C(23*A*)—C(24)	119.8 (6)
C(23)—C(24)—C(23*A*)	119.7 (6)	C(1)—C(6)—C(7)	111.1 (4)
C(1)—C(6)—C(10)	118.5 (4)	C(7)—C(6)—C(10)	104.3 (3)
C(6)—C(7)—O(7)	124.1 (3)	C(6)—C(7)—C(8)	109.7 (3)
O(7)—C(7)—C(8)	126.2 (4)	C(7)—C(8)—C(9)	103.4 (3)
C(7)—C(8)—C(11)	112.3 (4)	C(9)—C(8)—C(11)	120.2 (3)
C(8)—C(9)—C(10)	103.3 (4)	C(6)—C(10)—C(9)	103.9 (3)
C(8)—C(11)—O(11)	108.1 (3)	C(8)—C(11)—C(12)	116.2 (4)
O(11)—C(11)—C(12)	105.7 (3)	C(8)—C(11)—C(15)	114.7 (4)
O(11)—C(11)—C(15)	109.6 (4)	C(12)—C(11)—C(15)	102.0 (3)
C(11)—C(12)—C(13)	103.8 (3)	C(11)—C(12)—C(16)	113.8 (3)
C(13)—C(12)—C(16)	112.6 (3)	C(12)—C(13)—C(14)	106.9 (3)
C(13)—C(14)—C(15)	105.2 (4)	C(11)—C(15)—C(14)	105.1 (4)
C(12)—C(16)—O(17)	113.3 (4)	C(12)—C(16)—N(20)	112.1 (4)
O(17)—C(16)—N(20)	103.5 (3)	C(16)—O(17)—C(18)	107.6 (3)
O(17)—C(18)—C(19)	105.1 (3)	O(17)—C(18)—C(25)	109.4 (4)
C(19)—C(18)—C(25)	114.0 (5)	C(18)—C(19)—C(38)	115.3 (5)
C(18)—C(19)—N(20)	104.1 (4)	C(38)—C(19)—N(20)	110.4 (4)
C(16)—N(20)—C(19)	106.3 (3)	C(16)—N(20)—S(2)	117.6 (3)
C(19)—N(20)—S(2)	116.0 (4)	N(20)—S(2)—O(2*A*)	105.6 (3)
N(20)—S(2)—O(2*B*)	106.9 (2)	O(2*A*)—S(2)—O(2*B*)	120.5 (2)
N(20)—S(2)—C(33)	107.1 (2)	O(2*A*)—S(2)—C(33)	108.3 (3)
O(2*B*)—S(2)—C(33)	107.7 (2)	S(2)—C(33)—C(34)	121.0 (5)
S(2)—C(33)—C(34*A*)	118.8 (4)	C(34)—C(33)—C(34*A*)	119.8 (5)
C(33)—C(34)—C(35)	118.2 (7)	C(33)—C(34*A*)—C(35*A*)	120.2 (5)
C(34)—C(35)—C(36)	119.8 (6)	C(34*A*)—C(35*A*)—C(36)	119.3 (7)
C(35)—C(36)—C(35*A*)	122.5 (7)	C(35)—C(36)—Cl(2)	117.2 (5)

C(35A)—C(36)—Cl(2)	120.2 (7)	C(18)—C(25)—C(26)	119.6 (6)
C(18)—C(25)—C(26A)	121.6 (5)	C(26)—C(25)—C(26A)	118.8 (4)
C(25)—C(26)—C(27)	120.7 (8)	C(25)—C(26A)—C(27A)	118.6 (6)
C(26)—C(27)—C(28)	121.0 (7)	C(26A)—C(27A)—C(28)	122.8 (8)
C(27)—C(28)—C(27A)	118.0 (6)		

Intensities were corrected for Lorentz, polarization and absorption effects. The structure was solved by extracting the positions of the Cl and S atoms from a sharpened Patterson map and extending the structure with a tangent expansion. All H atoms were located by difference synthesis and refined with displacement factors of H fixed to $U(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$ or $1.5U_{eq}(O)$ using a riding model with C—H = 0.96 Å. The coordinates of the hydroxyl H atom were refined with the O—H distance restrained to 0.8500 (1) Å. Refinement was carried out with *XLS* (Sheldrick, 1987). The absolute configuration was determined by η refinement (Rogers, 1981) starting from both configurations [$\eta = 1.0$ (1) for the correct structure]. The molecular plot was prepared with *XP* (*SHELXTL-Plus*; Sheldrick, 1991).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SH1080). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A γ-Hydroxyvinylsulfoximine, $C_{23}H_{31}NO_3S$

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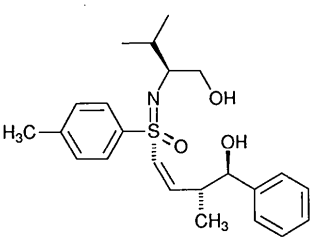
Abstract

The structure of [*S*(S),1*Z*,*N*(1*S*),3*R*,4*R*]-1-{*N*-[1-(hydroxymethyl)-2-methylpropyl]-*S*-(*p*-tolyl)sulfonimidoyl}-3-methyl-4-phenyl-1-buten-4-ol {or *N*-[1-(hydroxymethyl)-2-methylpropyl]-*S*-(4-hydroxy-3-methyl-4-phenyl-1-butenyl)-*S*-(*p*-tolyl)sulfoximide} has been determined. The C=C double bond is *cis* configured. Both hydroxyl groups form intramolecular hydrogen bonds.

Comment

The geometrical parameters of the two intramolecular hydrogen bonds are as follows: O(41)···O(61) 2.915 (3), H(41)···O(61) 2.080 (9) Å, O(41)—H(41)···O(61) 168 (1)° and O(61)···O(1) 2.902 (3), H(61)···O(1) 2.088 (8) Å, O(61)—H(61)···O(6) 160 (1)°. The title compound, (I), is the product of a diastereoselective γ-hydroxyalkylation of an enantiomerically pure crotylsulfoximine (Reggelin & Weinberger, 1994).



(I)

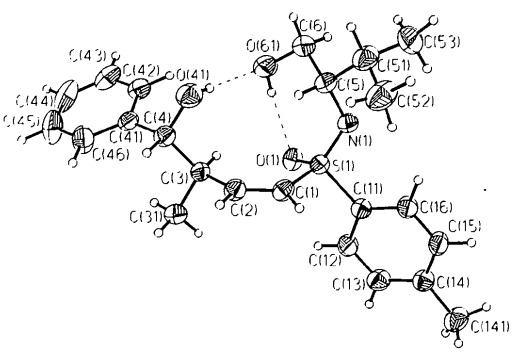


Fig. 1. Perspective view with the atom-numbering scheme, with heavy atoms represented as 30% probability ellipsoids and H atoms as spheres of arbitrary radii.

Experimental

Crystal data

$C_{23}H_{31}NO_3S$
$M_r = 401.5$
Orthorhombic
$P2_12_12_1$
$a = 9.707$ (1) Å
$b = 10.116$ (1) Å
$c = 23.262$ (2) Å
$V = 2284.2$ (4) Å^3

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 13$–42°
$\mu = 1.39$ mm^{-1}
$T = 293$ K
Transparent block