

(2*S*,4*R*,5*S*)-5-Allyl-4-hydroxytetrahydro-2-furylmethyl *p*-toluenesulfonate

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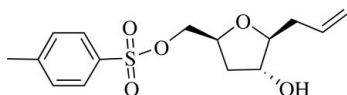
Received 8 May 2009; accepted 26 May 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.055; wR factor = 0.126; data-to-parameter ratio = 8.1.

In the title compound, $\text{C}_{15}\text{H}_{20}\text{O}_5\text{S}$, the tetrahydrofuran ring shows an envelope conformation. The crystal packing is stabilized by an intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond, generating a ribbon structure along the a axis. Two weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions are also observed.

Related literature

For the synthesis of chiral tetrahydrofurans bearing an allyl group at the C1 position, see: Romero *et al.* (2006); Sartillo-Melendez *et al.* (2006); Hernández-García *et al.* (2009); Paz-Morales *et al.* (2009). For ring conformation analysis, see: Cremer & Pople (1975).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{20}\text{O}_5\text{S}$	$V = 813.7$ (3) Å ³
$M_r = 312.37$	$Z = 2$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 5.9420$ (12) Å	$\mu = 0.22$ mm ⁻¹
$b = 16.966$ (3) Å	$T = 293$ K
$c = 8.1980$ (19) Å	$0.6 \times 0.4 \times 0.3$ mm
$\beta = 100.09$ (2)°	

Data collection

Bruker P4 diffractometer	$R_{\text{int}} = 0.030$
Absorption correction: none	3 standard reflections
2127 measured reflections	every 97 reflections
1575 independent reflections	intensity decay: 7%
1099 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.126$	$\Delta\rho_{\text{max}} = 0.26$ e Å ⁻³
$S = 1.07$	$\Delta\rho_{\text{min}} = -0.23$ e Å ⁻³
1575 reflections	Absolute structure: Flack (1983), 97
194 parameters	Friedel pairs
1 restraint	Flack parameter: -0.1 (2)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O5}-\text{H5O}\cdots\text{O1}^i$	0.96 (11)	1.83 (11)	2.782 (7)	171 (10)
$\text{C5}-\text{H5B}\cdots\text{O4}^i$	0.97	2.49	3.188 (8)	129
$\text{C13}-\text{H13}\cdots\text{O3}^{ii}$	0.93	2.46	3.350 (8)	161

Symmetry codes: (i) $x + 1, y, z$; (ii) $x, y, z + 1$.

Data collection: XSCANS (Siemens, 1994); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SIR2004 (Burla *et al.*, 2005); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

We gratefully acknowledge financial support from CONACYT (grant No. 62203 and Graduate Scholarship 159362) and Facultad de Ciencias Químicas (BUAP).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2418).

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supplementary materials

Acta Cryst. (2009). E65, o1456 [doi:10.1107/S160053680902011X]

(2*S*,4*R*,5*S*)-5-Allyl-4-hydroxytetrahydro-2-furylmethyl *p*-toluenesulfonate

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Comment

During the course of our investigations led to the synthesis of chiral tetrahydrofurans bearing an allyl group at C1 position (Romero *et al.*, 2006; Sartillo-Melendez *et al.*, 2006; Hernández-García *et al.*, 2009; Paz-Morales *et al.*, 2009), the title compound was obtained and separated by crystallization from the its C1-epimer.

In the crystal structure, the tosyl ring and terminal double bond reach a close distance from each one ($C7\cdots C13$ and $C8\cdots C12 = ca. 3.9 \text{ \AA}$), with allyl π orbital being perpendicular for those of the aromatic ring. The furane ring (O1/C1–C4) shows an envelope conformation on atom C2 with puckering parameters (Cremer & Pople, 1975) $q_2 = 0.348 (8) \text{ \AA}$ and $\varphi_2 = 72.3 (12)^\circ$. The molecules are linked by hydrogen bond [$O1\cdots O5 = 2.782 (7) \text{ \AA}$] interactions, building a ribbon structure along the [100] direction.

Experimental

The title compound was obtained from a solution of 1,2-*O*-isopropylidene- α -D-xylofuranose derivative (1.0 mmol) in 10 ml of dry CH_2Cl_2 . This was treated with allyltrimethylsilane (6.0 mmol) at room temperature over 10 min and the reaction mixture was cooled at 0 °C, then BF_3OEt_2 (6.0 mmol) was added dropwise. The reaction mixture was warmed at room temperature over 6 hrs and was treated with saturated aqueous solution of NaHCO_3 (10 ml). The aqueous layer was extracted three times with CH_2Cl_2 (20 ml). The organic phase was dried with MgSO_4 , concentrated and purified by flash chromatography on silica gel (hexane: ethyl acetate). The absolute configuration was established by the structure determination of 1,2-*O*-isopropylidene- α -D-xylofuranose of known absolute configuration of starting material.

Refinement

The H atom bonded to O atom was located in a difference map and the positional parameters were refined, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. C-bound H atoms were placed in geometrical idealized positions ($\text{C—H} = 0.93\text{--}0.98 \text{ \AA}$) and were refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

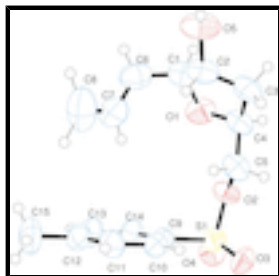


Fig. 1. The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

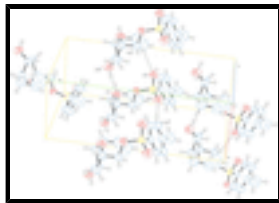


Fig. 2. The packing of the title compound, showing molecules connected by O5—H···O1¹ hydrogen bonds and intermolecular weak interactions (dashed lines).

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Crystal data

$C_{15}H_{20}O_5S$	$F_{000} = 332$
$M_r = 312.37$	$D_x = 1.275 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
Hall symbol: P 2yb	$\lambda = 0.71073 \text{ \AA}$
$a = 5.9420 (12) \text{ \AA}$	Cell parameters from 40 reflections
$b = 16.966 (3) \text{ \AA}$	$\theta = 4.8\text{--}24.7^\circ$
$c = 8.1980 (19) \text{ \AA}$	$\mu = 0.22 \text{ mm}^{-1}$
$\beta = 100.09 (2)^\circ$	$T = 293 \text{ K}$
$V = 813.7 (3) \text{ \AA}^3$	Prism, colorless
$Z = 2$	$0.6 \times 0.4 \times 0.3 \text{ mm}$

Data collection

Bruker P4 diffractometer	$R_{\text{int}} = 0.030$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.5^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 2.4^\circ$
$T = 293 \text{ K}$	$h = -1 \rightarrow 7$
$2\theta/\omega$ scans	$k = -1 \rightarrow 20$
Absorption correction: none	$l = -9 \rightarrow 9$
2127 measured reflections	3 standard reflections
1575 independent reflections	every 97 reflections
1099 reflections with $I > 2\sigma(I)$	intensity decay: 7%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.055$	$w = 1/[\sigma^2(F_o^2) + (0.0261P)^2 + 0.5527P]$
$wR(F^2) = 0.126$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1575 reflections	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

194 parameters
 1 restraint
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map
 Extinction correction: none
 Absolute structure: Flack (1983), 97 Friedel pairs
 Flack parameter: -0.1 (2)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.2153 (3)	0.44181 (11)	0.35980 (19)	0.0535 (4)
O4	-0.0229 (6)	0.4463 (4)	0.3656 (5)	0.0664 (12)
O2	0.2892 (7)	0.5318 (3)	0.3599 (6)	0.0586 (12)
C9	0.3645 (10)	0.4054 (4)	0.5478 (7)	0.0456 (14)
O1	0.5274 (7)	0.6525 (3)	0.5384 (7)	0.0707 (15)
O3	0.2936 (8)	0.4018 (3)	0.2256 (5)	0.0673 (14)
O5	1.1076 (9)	0.7133 (4)	0.5830 (9)	0.092 (2)
C10	0.5693 (10)	0.3653 (4)	0.5528 (8)	0.0488 (15)
H10	0.6287	0.3575	0.4564	0.059*
C4	0.5632 (11)	0.6353 (4)	0.3722 (9)	0.0615 (19)
H4	0.4558	0.6660	0.2926	0.074*
C11	0.6847 (12)	0.3367 (4)	0.7038 (9)	0.0611 (19)
H11	0.8222	0.3100	0.7077	0.073*
C14	0.2741 (12)	0.4168 (4)	0.6894 (8)	0.0614 (19)
H14	0.1377	0.4441	0.6860	0.074*
C2	0.9228 (11)	0.6608 (4)	0.5423 (10)	0.065 (2)
H2	0.9719	0.6073	0.5770	0.077*
C5	0.5240 (11)	0.5496 (4)	0.3390 (9)	0.0626 (19)
H5A	0.5446	0.5370	0.2271	0.075*
H5B	0.6317	0.5186	0.4158	0.075*
C3	0.8076 (12)	0.6601 (5)	0.3629 (10)	0.075 (2)
H3A	0.8799	0.6225	0.2994	0.090*
H3B	0.8110	0.7119	0.3135	0.090*
C13	0.3905 (13)	0.3868 (5)	0.8364 (9)	0.070 (2)
H13	0.3274	0.3931	0.9317	0.084*
C12	0.5976 (12)	0.3476 (5)	0.8486 (8)	0.065 (2)
C1	0.7298 (11)	0.6868 (5)	0.6323 (10)	0.068 (2)
H1	0.7159	0.7442	0.6244	0.082*

supplementary materials

C15	0.7242 (14)	0.3163 (6)	1.0118 (9)	0.092 (3)
H15A	0.7423	0.2603	1.0040	0.138*
H15B	0.6388	0.3280	1.0980	0.138*
H15C	0.8719	0.3408	1.0370	0.138*
C7	0.765 (2)	0.5786 (7)	0.8486 (12)	0.100 (3)
H7	0.6437	0.5466	0.8007	0.120*
C6	0.7504 (16)	0.6637 (5)	0.8113 (11)	0.088 (3)
H6A	0.8857	0.6888	0.8728	0.106*
H6B	0.6196	0.6849	0.8526	0.106*
C8	0.938 (2)	0.5455 (8)	0.9454 (12)	0.135 (5)
H8A	1.0609	0.5760	0.9949	0.162*
H8B	0.9375	0.4915	0.9645	0.162*
H5O	1.26 (2)	0.695 (7)	0.578 (14)	0.162*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0460 (8)	0.0574 (9)	0.0559 (9)	-0.0012 (9)	0.0053 (7)	0.0015 (10)
O4	0.038 (2)	0.080 (3)	0.081 (3)	-0.001 (3)	0.010 (2)	0.008 (4)
O2	0.044 (2)	0.050 (3)	0.084 (3)	-0.003 (2)	0.017 (2)	0.008 (2)
C9	0.046 (3)	0.043 (3)	0.048 (3)	-0.006 (3)	0.008 (3)	0.002 (3)
O1	0.035 (2)	0.087 (4)	0.093 (4)	-0.009 (3)	0.020 (2)	-0.010 (3)
O3	0.078 (3)	0.079 (3)	0.046 (2)	-0.003 (3)	0.013 (2)	-0.015 (2)
O5	0.050 (3)	0.082 (4)	0.146 (5)	-0.010 (3)	0.022 (4)	-0.015 (4)
C10	0.042 (4)	0.052 (4)	0.054 (4)	-0.004 (3)	0.013 (3)	-0.005 (3)
C4	0.049 (4)	0.052 (4)	0.082 (5)	0.005 (3)	0.009 (4)	0.016 (4)
C11	0.053 (4)	0.059 (5)	0.068 (4)	0.008 (4)	0.000 (3)	0.009 (4)
C14	0.062 (4)	0.073 (5)	0.051 (4)	0.010 (4)	0.013 (3)	-0.006 (3)
C2	0.033 (3)	0.051 (4)	0.109 (6)	-0.001 (3)	0.012 (4)	0.001 (4)
C5	0.048 (4)	0.061 (4)	0.083 (5)	0.004 (4)	0.021 (4)	0.012 (4)
C3	0.056 (4)	0.072 (5)	0.102 (6)	-0.012 (4)	0.028 (4)	0.018 (5)
C13	0.068 (5)	0.089 (6)	0.055 (4)	0.007 (4)	0.017 (4)	-0.007 (4)
C12	0.062 (5)	0.072 (5)	0.057 (4)	-0.013 (4)	0.000 (3)	-0.001 (4)
C1	0.041 (4)	0.061 (5)	0.104 (6)	0.007 (3)	0.017 (4)	0.003 (4)
C15	0.091 (6)	0.109 (7)	0.066 (5)	-0.001 (6)	-0.010 (4)	0.028 (5)
C7	0.117 (9)	0.100 (8)	0.080 (6)	-0.020 (7)	0.014 (6)	-0.011 (6)
C6	0.076 (6)	0.083 (7)	0.108 (7)	-0.011 (5)	0.022 (5)	-0.017 (6)
C8	0.181 (12)	0.135 (10)	0.088 (7)	0.042 (10)	0.023 (8)	0.016 (7)

Geometric parameters (\AA , $^\circ$)

S1—O4	1.427 (4)	C2—C1	1.533 (9)
S1—O3	1.437 (5)	C2—H2	0.9800
S1—O2	1.589 (5)	C5—H5A	0.9700
S1—C9	1.751 (6)	C5—H5B	0.9700
O2—C5	1.467 (7)	C3—H3A	0.9700
C9—C14	1.375 (8)	C3—H3B	0.9700
C9—C10	1.389 (8)	C13—C12	1.386 (10)
O1—C1	1.433 (8)	C13—H13	0.9300

O1—C4	1.445 (8)	C12—C15	1.512 (9)
O5—C2	1.408 (8)	C1—C6	1.503 (11)
O5—H5O	0.96 (11)	C1—H1	0.9800
C10—C11	1.392 (9)	C15—H15A	0.9600
C10—H10	0.9300	C15—H15B	0.9600
C4—C5	1.490 (10)	C15—H15C	0.9600
C4—C3	1.526 (9)	C7—C8	1.311 (14)
C4—H4	0.9800	C7—C6	1.475 (13)
C11—C12	1.388 (9)	C7—H7	0.9300
C11—H11	0.9300	C6—H6A	0.9700
C14—C13	1.378 (9)	C6—H6B	0.9700
C14—H14	0.9300	C8—H8A	0.9300
C2—C3	1.509 (10)	C8—H8B	0.9300
O4—S1—O3	120.5 (3)	H5A—C5—H5B	108.5
O4—S1—O2	103.0 (3)	C2—C3—C4	103.1 (6)
O3—S1—O2	109.1 (3)	C2—C3—H3A	111.1
O4—S1—C9	110.0 (3)	C4—C3—H3A	111.1
O3—S1—C9	109.0 (3)	C2—C3—H3B	111.1
O2—S1—C9	104.0 (3)	C4—C3—H3B	111.1
C5—O2—S1	117.7 (4)	H3A—C3—H3B	109.1
C14—C9—C10	120.9 (6)	C14—C13—C12	122.9 (7)
C14—C9—S1	118.7 (5)	C14—C13—H13	118.6
C10—C9—S1	120.3 (5)	C12—C13—H13	118.6
C1—O1—C4	109.9 (5)	C13—C12—C11	117.5 (6)
C2—O5—H5O	119 (7)	C13—C12—C15	122.0 (7)
C9—C10—C11	119.3 (6)	C11—C12—C15	120.5 (7)
C9—C10—H10	120.4	O1—C1—C6	109.6 (6)
C11—C10—H10	120.4	O1—C1—C2	104.6 (6)
O1—C4—C5	109.0 (6)	C6—C1—C2	117.1 (7)
O1—C4—C3	106.9 (6)	O1—C1—H1	108.4
C5—C4—C3	112.3 (6)	C6—C1—H1	108.4
O1—C4—H4	109.6	C2—C1—H1	108.4
C5—C4—H4	109.6	C12—C15—H15A	109.5
C3—C4—H4	109.6	C12—C15—H15B	109.5
C12—C11—C10	121.0 (7)	H15A—C15—H15B	109.5
C12—C11—H11	119.5	C12—C15—H15C	109.5
C10—C11—H11	119.5	H15A—C15—H15C	109.5
C9—C14—C13	118.4 (6)	H15B—C15—H15C	109.5
C9—C14—H14	120.8	C8—C7—C6	123.7 (12)
C13—C14—H14	120.8	C8—C7—H7	118.1
O5—C2—C3	116.0 (7)	C6—C7—H7	118.1
O5—C2—C1	108.8 (6)	C7—C6—C1	116.7 (8)
C3—C2—C1	102.8 (6)	C7—C6—H6A	108.1
O5—C2—H2	109.7	C1—C6—H6A	108.1
C3—C2—H2	109.7	C7—C6—H6B	108.1
C1—C2—H2	109.7	C1—C6—H6B	108.1
O2—C5—C4	107.5 (5)	H6A—C6—H6B	107.3
O2—C5—H5A	110.2	C7—C8—H8A	120.0
C4—C5—H5A	110.2	C7—C8—H8B	120.0

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O2—C5—H5B	110.2	H8A—C8—H8B	120.0
C4—C5—H5B	110.2		
O4—S1—O2—C5	-174.5 (5)	O5—C2—C3—C4	-151.9 (6)
O3—S1—O2—C5	-45.4 (5)	C1—C2—C3—C4	-33.3 (8)
C9—S1—O2—C5	70.8 (5)	O1—C4—C3—C2	21.1 (8)
O4—S1—C9—C14	-26.7 (6)	C5—C4—C3—C2	-98.3 (8)
O3—S1—C9—C14	-160.7 (5)	C9—C14—C13—C12	1.9 (12)
O2—S1—C9—C14	83.0 (6)	C14—C13—C12—C11	-2.0 (12)
O4—S1—C9—C10	152.9 (5)	C14—C13—C12—C15	179.0 (8)
O3—S1—C9—C10	18.9 (6)	C10—C11—C12—C13	0.9 (11)
O2—S1—C9—C10	-97.4 (5)	C10—C11—C12—C15	179.9 (7)
C14—C9—C10—C11	-0.4 (9)	C4—O1—C1—C6	-148.3 (6)
S1—C9—C10—C11	-179.9 (5)	C4—O1—C1—C2	-21.9 (8)
C1—O1—C4—C5	122.2 (6)	O5—C2—C1—O1	158.0 (6)
C1—O1—C4—C3	0.6 (8)	C3—C2—C1—O1	34.5 (8)
C9—C10—C11—C12	0.2 (10)	O5—C2—C1—C6	-80.5 (9)
C10—C9—C14—C13	-0.6 (10)	C3—C2—C1—C6	156.1 (7)
S1—C9—C14—C13	178.9 (6)	C8—C7—C6—C1	120.2 (11)
S1—O2—C5—C4	-170.4 (5)	O1—C1—C6—C7	59.6 (11)
O1—C4—C5—O2	58.8 (7)	C2—C1—C6—C7	-59.3 (12)
C3—C4—C5—O2	177.0 (5)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5O \cdots O1 ⁱ	0.96 (11)	1.83 (11)	2.782 (7)	171 (10)
C5—H5B \cdots O4 ⁱ	0.97	2.49	3.188 (8)	129
C13—H13 \cdots O3 ⁱⁱ	0.93	2.46	3.350 (8)	161

Symmetry codes: (i) $x+1, y, z$; (ii) $x, y, z+1$.

Fig. 1

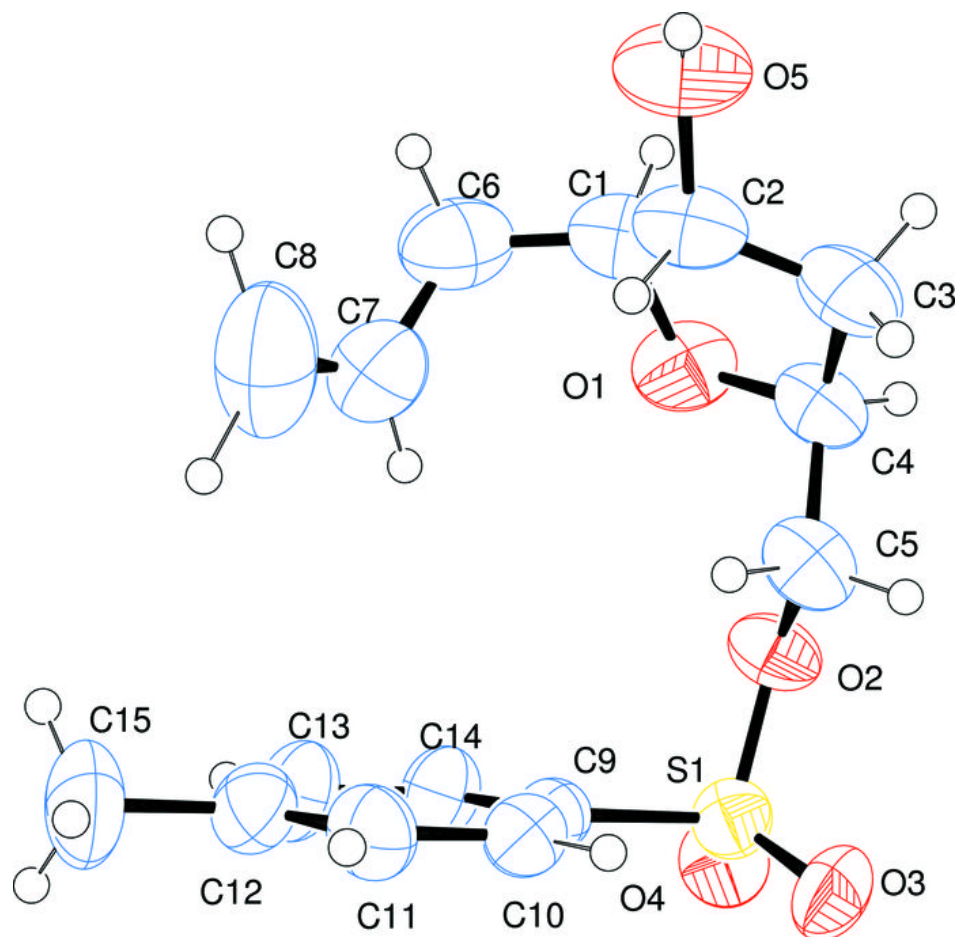


Fig. 2

