

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(+)-(5*S*)-5-[(1*S*)-2-Benzoyl-1-hydroxyethyl]-1,2,3,4-tetrahydrofuran-2-oneJulie A. Cuthbert,^a Dennis K. Taylor^{b‡} and Edward R. T. Tiekink^{c*}^aDepartment of Chemistry, The University of Adelaide, 5005 South Australia, Australia, ^bDepartment of Wine and Horticulture, The University of Adelaide, Waite Campus, Glen Osmond, 5064 South Australia, Australia, and ^cDepartment of Chemistry, The University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA

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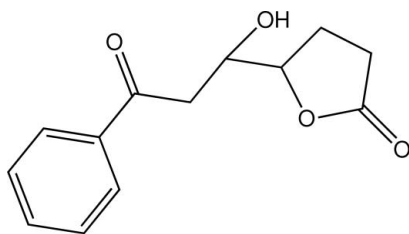
Received 15 June 2007; accepted 25 June 2007

Key indicators: single-crystal X-ray study; $T = 223$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.044; wR factor = 0.122; data-to-parameter ratio = 16.8.

The title compound, $\text{C}_{13}\text{H}_{14}\text{O}_4$, displays a kinked conformation in which the hydroxy group is orientated over the furanone ring. Molecules aggregate into linear supramolecular chains *via* $\text{O}-\text{H}\cdots\text{O}$ interactions, and $\text{C}-\text{H}\cdots\text{O}$ contacts link these chains into two-dimensional arrays.

Related literature

For related literature, see: Avery *et al.* (2005); Jung *et al.* (2002); Yates & Anderson (1963).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{14}\text{O}_4$
 $M_r = 234.24$
 Monoclinic, $P2_1/n$
 $a = 14.6504$ (16) Å
 $b = 5.5565$ (6) Å
 $c = 14.7565$ (15) Å
 $\beta = 106.628$ (2)°

$V = 1151.0$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 223$ (2) K
 $0.47 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2000)
 $T_{\min} = 0.857$, $T_{\max} = 1$

7754 measured reflections
 2635 independent reflections
 2124 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.122$
 $S = 1.04$
 2635 reflections
 157 parameters

1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1
 Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1O}\cdots\text{O12}^i$	0.84	2.04	2.8750 (16)	174
$\text{C2}-\text{H2A}\cdots\text{O11}$	0.98	2.46	2.8640 (19)	104
$\text{C14}-\text{H14A}\cdots\text{O3}^{ii}$	0.98	2.59	3.154 (2)	117
$\text{C14}-\text{H14B}\cdots\text{O12}^i$	0.98	2.56	3.383 (2)	142
$\text{C36}-\text{H36}\cdots\text{O1}^{iii}$	0.94	2.38	3.2787 (19)	159

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976) and DIAMOND (Brandenburg, 2006); software used to prepare material for publication: SHELXL97.

We are grateful to the Australian Research Council for financial support. We also thank the The University of Adelaide, Faculty of Science, for a postgraduate scholarship to JAC.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2239).

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supplementary materials

Acta Cryst. (2007). E63, o3349 [doi:10.1107/S160053680703084X]

(+)-(5*S*)-5-[(1*S*)-2-Benzoyl-1-hydroxyethyl]-1,2,3,4-tetrahydrofuran-2-one

J. A. Cuthbert, D. K. Taylor and E. R. T. Tiekink

Comment

The title compound (I) was investigated during studies of intramolecular cyclizations of 1,2-dioxines containing tethered hydroxyl groups (Avery *et al.*, 2005). The molecular structure (Fig. 1) shows the O1-hydroxyl group to lie over the five-membered furanone ring; the latter is twisted about the C13—C14 bond. Overall the molecule adopts a kinked shape. The C₆H₅C(=O)CH₂CH residue has an open or extended configuration as seen in the values of the C1/C2/C3/C31 and C2/C3/C31/C32 torsion angles of 164.79 (12) and 165.98 (13) °, respectively. The kink in the molecule occurs about the C1—C15 bond as reflected in the C2/C1/C15/O11 torsion angle of −55.15 (14)°. The dihedral angle formed between the six- and five-membered rings is 18.52 (8)°. Molecules aggregate in the crystal structure *via* O—H⋯O contacts involving the hydroxyl group and O12-carbonyl to form a linear supramolecular chain aligned along the *b* axis (Fig. 2 & Table 1); reinforcing this chain are C14—H⋯O12 contacts. The chains are connected into double-chains *via* additional phenyl C36—H⋯O1 and methylene-C14—H⋯O3 interactions (Table 1); the lactone-O11 atom is involved in an intramolecular C—H⋯O contact with a methylene C2—H atom. Double chains stack along the (1 0 − 1) plane being separated by hydrophobic interactions (Fig. 3).

Experimental

The title compound was prepared in 27% yield following literature procedures (Avery *et al.*, 2005; Jung *et al.*, 2002; Yates & Anderson, 1963). Solid (I) was recrystallized heptane/dichloromethane to give white needles with m.p. 366–367 K. *R*_f 0.28 (3:2 ethyl acetate:hexanes). Elemental analysis found: C 66.73, H, 5.99%; C₁₃H₁₄O₄ requires: C 66.66, H, 6.02%. IR (nujol) 3144–3650, 2253 (w), 1780, 1769, 1747, 1732, 1689, 1682, 1671, 1597, 1580 cm^{−1}. ¹H NMR (600 MHz, p.p.m.) δ 2.30–2.41 (m, 3H), 2.50 (ddd, *J* = 17.4, 10.2, 6.0 Hz, 1H), 2.73 (ddd, *J* = 17.4, 9.6, 7.2 Hz, 1H), 3.25 (dd, *J* = 18.0, 3.0 Hz, 1H), 3.41 (dd, *J* = 18.0, 9.0 Hz, 1H), 4.32 (ddd, *J* = 9.0, 3.0, 3.0 Hz, 1H), 4.58 (ddd, *J* = 8.4, 6.0, 3.0 Hz, 1H), 7.47–7.50 (m, 2H), 7.96–7.98 (m, 2H), 7.59–7.62 (m, 1H). ¹³C NMR (75 MHz, p.p.m.) δ 23.9, 28.4, 41.6, 69.3, 81.6, 128.1, 128.8, 133.8, 136.4, 177.5, 199.7. MS (EI) *m/z* (%): 235 ([*M*+H]⁺, 20), 217 (35), 157 (37), 149 (58), 120 (14), 105 (100), 77 (88), 51 (52).

Refinement

All C-bound H atoms were included in the riding-model approximation, with C—H = 0.94 to 0.99 Å, and with *U*_{iso}(H) = 1.2*U*_{eq}(C). The hydroxyl-H atoms were located from a difference map and included so that O—H = 0.84 Å and *U*_{iso}(H) = 1.5*U*_{eq}(O).

Figures

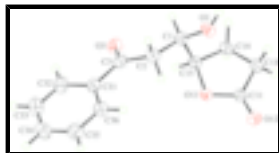


Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 35% probability level.

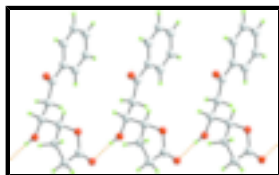


Fig. 2. View of the supramolecular chain in (I) mediated by hydrogen bonds, shown as orange-dashed lines. Colour code: red (oxygen), grey (carbon) and green (hydrogen).

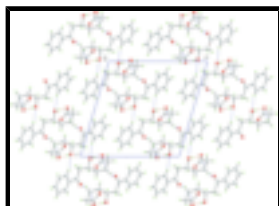


Fig. 3. View of the unit-cell contents of (I) highlighting the stacking of double layers along the (1 0 - 1) plane. Hydrogen bonding contacts are shown as orange- (O—H...O) and blue- (C—H...O) dashed lines.

(+)-(5S)-5-[(1S)-2-Benzoyl-1-hydroxyethyl]-1,2,3,4-tetrahydrofuran-2-one

Crystal data

$C_{13}H_{14}O_4$

$M_r = 234.24$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 14.6504$ (16) Å

$b = 5.5565$ (6) Å

$c = 14.7565$ (15) Å

$\beta = 106.628$ (2)°

$V = 1151.0$ (2) Å³

$Z = 4$

$F_{000} = 496$

$D_x = 1.352$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 2654 reflections

$\theta = 2.3$ – 29.0 °

$\mu = 0.10$ mm⁻¹

$T = 223$ (2) K

Block, colourless

$0.47 \times 0.18 \times 0.16$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 223$ (2) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2000)

$T_{\min} = 0.857$, $T_{\max} = 1$

7754 measured reflections

2635 independent reflections

2124 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 27.5$ °

$\theta_{\text{min}} = 1.7$ °

$h = -19 \rightarrow 18$

$k = -7 \rightarrow 7$

$l = -11 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.2723P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2635 reflections	$(\Delta/\sigma)_{\max} < 0.001$
157 parameters	$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.39803 (7)	0.15817 (19)	0.47990 (7)	0.0425 (3)
H1O	0.3653	0.0391	0.4868	0.058*
O3	0.41520 (8)	0.2218 (2)	0.20524 (7)	0.0477 (3)
O11	0.30843 (7)	0.58579 (17)	0.38984 (7)	0.0375 (3)
O12	0.27976 (8)	0.77243 (19)	0.51112 (8)	0.0473 (3)
C1	0.38118 (10)	0.1995 (2)	0.38123 (9)	0.0355 (3)
H1	0.3724	0.0431	0.3477	0.043*
C2	0.46917 (10)	0.3235 (3)	0.36818 (10)	0.0391 (3)
H2A	0.4735	0.4856	0.3953	0.047*
H2B	0.5258	0.2339	0.4035	0.047*
C3	0.46945 (10)	0.3429 (3)	0.26603 (10)	0.0367 (3)
C12	0.26556 (10)	0.6016 (2)	0.45865 (10)	0.0369 (3)
C13	0.20198 (10)	0.3884 (3)	0.45679 (11)	0.0410 (3)
H13A	0.1371	0.4401	0.4534	0.049*
H13B	0.2265	0.2884	0.5132	0.049*
C14	0.20369 (10)	0.2517 (3)	0.36801 (10)	0.0389 (3)
H14A	0.1457	0.2826	0.3164	0.047*

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H14B	0.2096	0.0781	0.3801	0.047*
C15	0.29174 (10)	0.3504 (2)	0.34409 (9)	0.0345 (3)
H15	0.2776	0.3701	0.2748	0.041*
C31	0.53744 (9)	0.5110 (3)	0.24057 (10)	0.0369 (3)
C32	0.55196 (12)	0.4892 (3)	0.15187 (11)	0.0505 (4)
H32	0.5200	0.3685	0.1101	0.061*
C33	0.61305 (13)	0.6438 (4)	0.12478 (13)	0.0596 (5)
H33	0.6232	0.6269	0.0650	0.071*
C34	0.65933 (13)	0.8236 (4)	0.18532 (13)	0.0571 (5)
H34	0.7007	0.9292	0.1666	0.069*
C35	0.64510 (12)	0.8480 (3)	0.27262 (13)	0.0538 (4)
H35	0.6762	0.9715	0.3134	0.065*
C36	0.58489 (11)	0.6910 (3)	0.30106 (11)	0.0451 (4)
H36	0.5762	0.7067	0.3615	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0462 (6)	0.0428 (6)	0.0341 (5)	-0.0025 (5)	0.0043 (4)	0.0065 (4)
O3	0.0490 (6)	0.0508 (6)	0.0414 (6)	-0.0100 (5)	0.0098 (5)	-0.0109 (5)
O11	0.0423 (5)	0.0283 (5)	0.0437 (5)	-0.0019 (4)	0.0149 (4)	0.0028 (4)
O12	0.0564 (6)	0.0373 (6)	0.0507 (6)	-0.0020 (5)	0.0193 (5)	-0.0053 (5)
C1	0.0394 (7)	0.0322 (7)	0.0313 (7)	0.0001 (5)	0.0044 (5)	-0.0001 (5)
C2	0.0343 (7)	0.0434 (8)	0.0355 (7)	0.0002 (6)	0.0034 (6)	-0.0010 (6)
C3	0.0332 (6)	0.0358 (7)	0.0384 (7)	0.0041 (5)	0.0058 (5)	-0.0036 (6)
C12	0.0373 (7)	0.0322 (7)	0.0402 (7)	0.0035 (5)	0.0097 (6)	0.0057 (6)
C13	0.0407 (7)	0.0385 (8)	0.0450 (8)	-0.0017 (6)	0.0142 (6)	0.0071 (6)
C14	0.0354 (7)	0.0340 (7)	0.0433 (8)	-0.0041 (5)	0.0049 (6)	0.0030 (6)
C15	0.0386 (7)	0.0311 (6)	0.0307 (6)	-0.0021 (5)	0.0049 (5)	0.0009 (5)
C31	0.0314 (6)	0.0399 (7)	0.0373 (7)	0.0040 (6)	0.0062 (5)	-0.0002 (6)
C32	0.0486 (9)	0.0595 (10)	0.0434 (8)	-0.0045 (8)	0.0134 (7)	-0.0072 (7)
C33	0.0581 (10)	0.0759 (13)	0.0493 (10)	-0.0048 (9)	0.0230 (8)	0.0004 (9)
C34	0.0487 (9)	0.0627 (11)	0.0619 (11)	-0.0070 (8)	0.0190 (8)	0.0097 (9)
C35	0.0497 (9)	0.0521 (10)	0.0567 (10)	-0.0106 (8)	0.0107 (8)	-0.0037 (8)
C36	0.0439 (8)	0.0483 (9)	0.0417 (8)	-0.0041 (7)	0.0101 (6)	-0.0035 (7)

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

O1—C1	1.4241 (16)	C13—H13B	0.9800
O1—H1O	0.8401	C14—C15	1.533 (2)
O3—C3	1.2169 (17)	C14—H14A	0.9800
O11—C12	1.3409 (17)	C14—H14B	0.9800
O11—C15	1.4600 (16)	C15—H15	0.9900
O12—C12	1.2047 (18)	C31—C32	1.390 (2)
C1—C15	1.5196 (19)	C31—C36	1.388 (2)
C1—C2	1.522 (2)	C32—C33	1.380 (3)
C1—H1	0.9900	C32—H32	0.9400
C2—C3	1.5123 (19)	C33—C34	1.381 (3)
C2—H2A	0.9800	C33—H33	0.9400

C2—H2B	0.9800	C34—C35	1.369 (3)
C3—C31	1.490 (2)	C34—H34	0.9400
C12—C13	1.503 (2)	C35—C36	1.388 (2)
C13—C14	1.521 (2)	C35—H35	0.9400
C13—H13A	0.9800	C36—H36	0.9400
C1—O1—H1O	108.3	C15—C14—H14A	111.0
C12—O11—C15	110.87 (10)	C13—C14—H14B	111.0
O1—C1—C15	109.70 (11)	C15—C14—H14B	111.0
O1—C1—C2	107.29 (11)	H14A—C14—H14B	109.0
C15—C1—C2	112.27 (12)	O11—C15—C1	108.32 (10)
O1—C1—H1	109.2	O11—C15—C14	104.76 (11)
C15—C1—H1	109.2	C1—C15—C14	114.52 (11)
C2—C1—H1	109.2	O11—C15—H15	109.7
C3—C2—C1	113.73 (11)	C1—C15—H15	109.7
C3—C2—H2A	108.8	C14—C15—H15	109.7
C1—C2—H2A	108.8	C32—C31—C36	119.00 (14)
C3—C2—H2B	108.8	C32—C31—C3	118.46 (13)
C1—C2—H2B	108.8	C36—C31—C3	122.53 (13)
H2A—C2—H2B	107.7	C33—C32—C31	120.36 (16)
O3—C3—C31	120.22 (13)	C33—C32—H32	119.8
O3—C3—C2	120.52 (13)	C31—C32—H32	119.8
C31—C3—C2	119.26 (12)	C32—C33—C34	120.11 (16)
O12—C12—O11	120.42 (13)	C32—C33—H33	119.9
O12—C12—C13	128.68 (13)	C34—C33—H33	119.9
O11—C12—C13	110.90 (12)	C35—C34—C33	120.09 (16)
C12—C13—C14	104.28 (11)	C35—C34—H34	120.0
C12—C13—H13A	110.9	C33—C34—H34	120.0
C14—C13—H13A	110.9	C34—C35—C36	120.20 (16)
C12—C13—H13B	110.9	C34—C35—H35	119.9
C14—C13—H13B	110.9	C36—C35—H35	119.9
H13A—C13—H13B	108.9	C35—C36—C31	120.22 (15)
C13—C14—C15	104.01 (11)	C35—C36—H36	119.9
C13—C14—H14A	111.0	C31—C36—H36	119.9
O1—C1—C2—C3	170.38 (11)	C13—C14—C15—O11	-22.70 (13)
C15—C1—C2—C3	-69.02 (16)	C13—C14—C15—C1	95.83 (13)
C1—C2—C3—O3	-15.6 (2)	O3—C3—C31—C32	-13.6 (2)
C1—C2—C3—C31	164.79 (12)	C2—C3—C31—C32	165.98 (13)
C15—O11—C12—O12	171.93 (12)	O3—C3—C31—C36	164.87 (14)
C15—O11—C12—C13	-8.76 (15)	C2—C3—C31—C36	-15.5 (2)
O12—C12—C13—C14	172.88 (14)	C36—C31—C32—C33	0.3 (2)
O11—C12—C13—C14	-6.36 (15)	C3—C31—C32—C33	178.88 (16)
C12—C13—C14—C15	17.70 (14)	C31—C32—C33—C34	-0.8 (3)
C12—O11—C15—C1	-102.61 (13)	C32—C33—C34—C35	0.3 (3)
C12—O11—C15—C14	20.04 (14)	C33—C34—C35—C36	0.6 (3)
O1—C1—C15—O11	64.04 (13)	C34—C35—C36—C31	-1.1 (3)
C2—C1—C15—O11	-55.15 (14)	C32—C31—C36—C35	0.7 (2)
O1—C1—C15—C14	-52.45 (15)	C3—C31—C36—C35	-177.84 (14)
C2—C1—C15—C14	-171.64 (12)		

supplementary materials

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1O \cdots O12 ⁱ	0.84	2.04	2.8750 (16)	174
C2—H2A \cdots O11	0.98	2.46	2.8640 (19)	104
C14—H14A \cdots O3 ⁱⁱ	0.98	2.59	3.154 (2)	117
C14—H14B \cdots O12 ⁱ	0.98	2.56	3.383 (2)	142
C36—H36 \cdots O1 ⁱⁱⁱ	0.94	2.38	3.2787 (19)	159

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1/2, y+1/2, -z+1/2$; (iii) $-x+1, -y+1, -z+1$.

Fig. 1

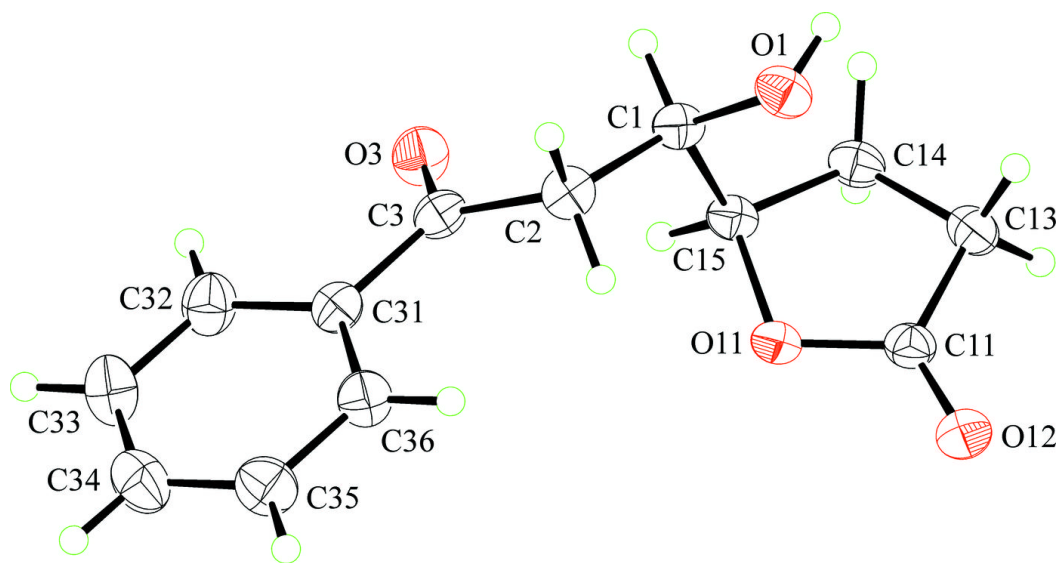


Fig. 2

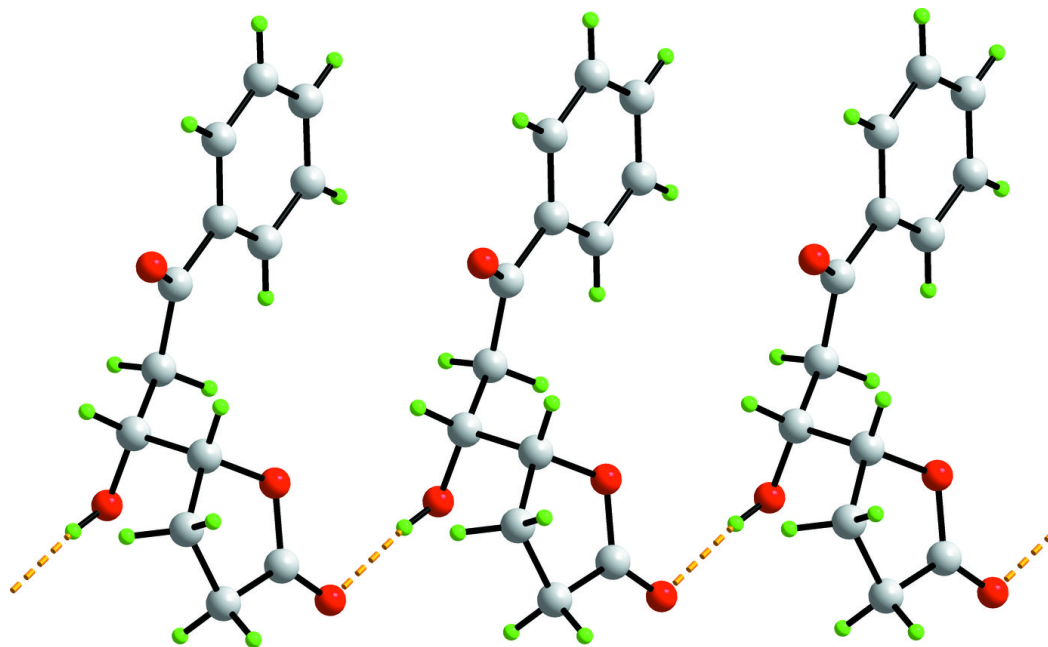


Fig. 3

