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2-*O*-(4,4′-Dimethylbenzhydryl)-L-erythronolactone

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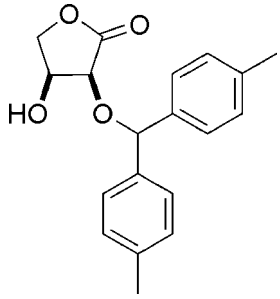
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.006 Å; R factor = 0.059; wR factor = 0.171; data-to-parameter ratio = 10.2.

The high regioselectivity of the $SnCl_2$-catalyzed reaction of diaryldiazomethanes with vicinal diols was demonstrated by the reaction of diazo[bis(4-methylphenyl)]methane with L-erythronolactone. The major product was unequivocally established by X-ray crystallographic analysis to be the title compound, $C_{19}H_{20}O_4$. The absolute configuration was determined by the use of L-erythronolactone as the starting material. The crystal structure contains alternating O—H···O hydrogen-bonded chains of molecules lying perpendicular to the *bc* plane.

Related literature

For related literature, see: Humphlett (1967); Jackson *et al.* (1982); Petursson & Webber (1982); Petursson (2001, 2003); Collins & Ferrier (1995); Draths *et al.* (1992); Görbitz (1999).



Experimental

Crystal data

$C_{19}H_{20}O_4$
M_r = 312.37
Orthorhombic, $P2_12_12_1$
a = 6.1276 (2) Å
b = 8.8248 (3) Å
c = 30.2629 (10) Å
V = 1636.46 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 150 K
0.50 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.84, T_{max} = 0.99
7163 measured reflections
2112 independent reflections
1323 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.171
S = 0.93
2112 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O22—H26···O23^{i}	0.85	2.19	2.863 (6)	137

Symmetry code: (i) $x+1, y, z$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2137).

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supplementary materials

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2-*O*-(4,4'-Dimethylbenzhydryl)-L-erythronolactone

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Comment

Carbohydrates provide excellent starting materials for the synthesis of small chiral molecules (Collins & Ferrier, 1995). They are relatively inexpensive and provide an almost boundless pool of chiral building blocks (Draths *et al.*, 1992). *L*-Erythronolactone **1**, readily available from D-arabinose (Humphlett, 1967), is an underused carbohydrate synthon due to the difficulty in differentiating between the two secondary hydroxyl groups.

Diazodiphenylmethane has been found to be a useful protecting group in the synthesis of methyl 2,3,6-tri-*O*-methyl-[α]-*D*-glucopyranoside and kojibiose octa-acetate (Jackson *et al.*, 1982), and monoalkylations of vicinal diols have been achieved with this reagent and other diaryldiazoalkanes in the presence of catalytic amounts of tin(II) chloride with high regioselectivities (Petursson & Webber, 1982; Petursson, 2001, 2003).

The reaction of *L*-erythronolactone with diazo[bis(4-methylphenyl)]methane and a catalytic amount of tin(II) chloride in 1,2-dimethoxyethane gave a 5:1 mixture of mono-protected lactones **2** and **3** (Fig. 1). The crystal structure has firmly established that the major product is the title compound, **2** (Fig. 2). The crystal structure consists of alternating hydrogen-bonded chains of molecules lying perpendicular to the *bc* plane (Fig. 3).

Experimental

2-*O*-(4,4'-Dimethylbenzhydryl)-*L*-erythronolactone was recrystallized by vapour diffusion of hexane into ethyl acetate: m.p. 425–427 K; $[\alpha]_D^{21}$ −51.7 (*c*, 1.08 in chloroform).

Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned on the basis of the starting material.

The relatively large ratio of minimum to maximum corrections applied in the multiscan process (1:1.18) reflect changes in the illuminated volume of the crystal. Changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO*/*SCALEPACK*, Otwinowski & Minor, 1997).

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 and O—H 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

Figures

Fig. 1. The reaction leading to the title compound.

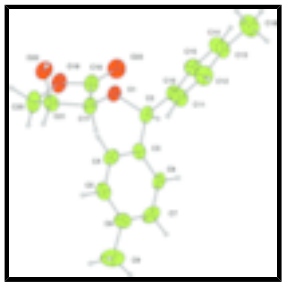

Fig. 2. The molecular structure with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

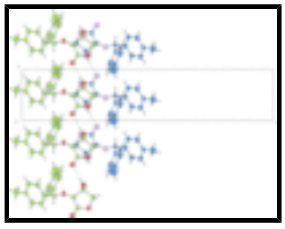

Fig. 3. The crystal structure consists of alternate layers of hydrogen-bonded chains of molecules lying perpendicular to the *bc* plane.

2-*O*-(4,4'-Dimethylbenzhydryl)-*L*-erythronolactone

Crystal data

$C_{19}H_{20}O_4$	$D_x = 1.268$ Mg m^{-3}
$M_r = 312.37$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 1832 reflections
$a = 6.1276$ (2) Å	$\theta = 5$–27º
$b = 8.8248$ (3) Å	$\mu = 0.09$ mm^{-1}
$c = 30.2629$ (10) Å	$T = 150$ K
$V = 1636.46$ (9) Å^3	Needle, colourless
$Z = 4$	0.50 × 0.10 × 0.10 mm
$F_{000} = 664$	

Data collection

Nonius KappaCCD diffractometer	1323 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.086$
$T = 150$ K	$\theta_{max} = 27.5$º
ω scans	$\theta_{min} = 5.2$º
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)	$h = -7\rightarrow7$
$T_{min} = 0.84$, $T_{max} = 0.99$	$k = -11\rightarrow11$
7163 measured reflections	$l = -37\rightarrow38$
2112 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F^2) + (0.1P)^2]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.059$	$(\Delta/\sigma)_{max} = 0.0001$
$wR(F^2) = 0.171$	$\Delta\rho_{max} = 0.39$ e Å^{-3}
$S = 0.93$	$\Delta\rho_{min} = -0.39$ e Å^{-3}
2112 reflections	Extinction correction: None
208 parameters	
Primary atom site location: structure-invariant direct methods	
Hydrogen site location: inferred from neighbouring sites	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5621 (5)	0.7497 (3)	0.16641 (6)	0.0368
C2	0.5141 (7)	0.6595 (4)	0.12785 (10)	0.0360
C3	0.6524 (7)	0.5171 (4)	0.12688 (10)	0.0328
C4	0.8599 (8)	0.5137 (4)	0.14619 (10)	0.0381
C5	0.9777 (8)	0.3806 (5)	0.14738 (11)	0.0406
C6	0.8997 (8)	0.2478 (5)	0.12948 (11)	0.0423
C7	0.6980 (8)	0.2533 (5)	0.10875 (12)	0.0461
C8	0.5765 (8)	0.3839 (4)	0.10784 (10)	0.0380
C9	1.0239 (11)	0.1014 (5)	0.13336 (14)	0.0682
C10	0.5396 (7)	0.7615 (4)	0.08805 (10)	0.0360
C11	0.7307 (8)	0.7685 (5)	0.06273 (12)	0.0447
C12	0.7456 (9)	0.8691 (5)	0.02763 (12)	0.0476
C13	0.5725 (8)	0.9631 (5)	0.01651 (11)	0.0424
C14	0.3827 (8)	0.9546 (5)	0.04116 (11)	0.0446
C15	0.3677 (8)	0.8529 (5)	0.07647 (11)	0.0422
C16	0.5911 (10)	1.0723 (6)	−0.02186 (13)	0.0653
C17	0.4910 (7)	0.6830 (4)	0.20603 (10)	0.0354
C18	0.2803 (7)	0.7510 (5)	0.22240 (11)	0.0372
O19	0.2760 (5)	0.7464 (3)	0.26658 (7)	0.0452
C20	0.4842 (8)	0.6878 (5)	0.28307 (11)	0.0488
C21	0.6449 (7)	0.7073 (5)	0.24494 (12)	0.0392
O22	0.7273 (5)	0.8564 (3)	0.24595 (8)	0.0425
O23	0.1313 (5)	0.8012 (3)	0.20124 (8)	0.0481
H21	0.3591	0.6243	0.1266	0.0485*
H41	0.9144	0.6059	0.1587	0.0493*
H51	1.1210	0.3779	0.1629	0.0504*
H71	0.6433	0.1650	0.0941	0.0580*
H81	0.4389	0.3839	0.0941	0.0458*
H91	0.9516	0.0236	0.1167	0.1067*

H92	1.0233	0.0731	0.1638	0.1061*
H93	1.1723	0.1170	0.1245	0.1064*
H111	0.8500	0.7011	0.0690	0.0541*
H121	0.8764	0.8725	0.0102	0.0612*
H141	0.2599	1.0180	0.0336	0.0573*
H151	0.2375	0.8467	0.0924	0.0525*
H161	0.4540	1.1194	−0.0262	0.1014*
H162	0.7021	1.1475	−0.0148	0.1017*
H163	0.6272	1.0160	−0.0482	0.1013*
H171	0.4638	0.5726	0.2016	0.0455*
H201	0.5259	0.7483	0.3091	0.0600*
H202	0.4712	0.5830	0.2925	0.0609*
H211	0.7611	0.6306	0.2456	0.0579*
H26	0.8375	0.8918	0.2326	0.0714*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0501 (18)	0.0382 (13)	0.0222 (11)	−0.0038 (15)	0.0037 (11)	−0.0011 (10)
C2	0.036 (2)	0.045 (2)	0.0275 (16)	−0.002 (2)	−0.0028 (17)	−0.0065 (15)
C3	0.035 (2)	0.0386 (19)	0.0252 (16)	−0.0005 (19)	−0.0009 (16)	0.0027 (14)
C4	0.041 (3)	0.039 (2)	0.0348 (18)	−0.001 (2)	−0.0005 (18)	−0.0061 (15)
C5	0.040 (3)	0.049 (2)	0.0331 (18)	0.006 (2)	−0.0016 (18)	0.0042 (16)
C6	0.056 (3)	0.038 (2)	0.0336 (18)	0.008 (2)	0.0120 (19)	0.0021 (17)
C7	0.059 (3)	0.042 (2)	0.037 (2)	−0.006 (3)	0.008 (2)	−0.0102 (17)
C8	0.046 (3)	0.044 (2)	0.0242 (17)	−0.005 (2)	−0.0048 (17)	−0.0050 (14)
C9	0.074 (4)	0.049 (3)	0.081 (3)	0.015 (3)	0.012 (3)	−0.004 (2)
C10	0.039 (3)	0.039 (2)	0.0298 (17)	0.004 (2)	0.0001 (16)	−0.0011 (15)
C11	0.045 (3)	0.052 (2)	0.0367 (19)	0.004 (3)	0.0018 (18)	0.0042 (18)
C12	0.047 (3)	0.057 (3)	0.040 (2)	−0.002 (3)	0.0105 (19)	0.0012 (19)
C13	0.051 (3)	0.046 (2)	0.0302 (17)	−0.001 (2)	−0.0081 (19)	0.0012 (16)
C14	0.051 (3)	0.042 (2)	0.0409 (19)	0.002 (3)	−0.003 (2)	0.0048 (17)
C15	0.043 (3)	0.045 (2)	0.038 (2)	0.003 (2)	0.0085 (19)	−0.0007 (17)
C16	0.075 (4)	0.068 (3)	0.053 (2)	−0.008 (3)	0.001 (3)	0.017 (2)
C17	0.040 (3)	0.043 (2)	0.0232 (16)	0.001 (2)	0.0030 (16)	−0.0016 (14)
C18	0.036 (3)	0.048 (2)	0.0275 (18)	0.000 (2)	0.0035 (16)	−0.0025 (18)
O19	0.045 (2)	0.0615 (19)	0.0292 (13)	0.001 (2)	0.0068 (11)	−0.0063 (12)
C20	0.050 (3)	0.070 (3)	0.0267 (18)	−0.006 (3)	0.0027 (19)	−0.0018 (17)
C21	0.042 (3)	0.046 (2)	0.0296 (17)	0.003 (2)	0.0038 (17)	0.0032 (15)
O22	0.0353 (17)	0.0492 (17)	0.0431 (14)	−0.0052 (15)	0.0026 (13)	−0.0066 (12)
O23	0.0347 (19)	0.0587 (19)	0.0510 (15)	0.0003 (17)	0.0023 (14)	−0.0021 (13)

Geometric parameters (Å, °)

O1—C2	1.443 (4)	C12—C13	1.388 (6)
O1—C17	1.405 (4)	C12—H121	0.960
C2—C3	1.516 (5)	C13—C14	1.384 (6)
C2—C10	1.512 (5)	C13—C16	1.513 (6)
C2—H21	1.000	C14—C15	1.398 (5)

C3—C4	1.399 (6)	C14—H141	0.965
C3—C8	1.389 (5)	C15—H151	0.934
C4—C5	1.379 (5)	C16—H161	0.946
C4—H41	0.958	C16—H162	0.974
C5—C6	1.377 (6)	C16—H163	0.965
C5—H51	0.995	C17—C18	1.507 (5)
C6—C7	1.387 (6)	C17—C21	1.524 (5)
C6—C9	1.504 (6)	C17—H171	0.997
C7—C8	1.373 (6)	C18—O19	1.338 (4)
C7—H71	0.957	C18—O23	1.200 (5)
C8—H81	0.940	O19—C20	1.465 (5)
C9—H91	0.961	C20—C21	1.527 (6)
C9—H92	0.953	C20—H201	0.985
C9—H93	0.958	C20—H202	0.971
C10—C11	1.401 (5)	C21—O22	1.409 (5)
C10—C15	1.372 (6)	C21—H211	0.982
C11—C12	1.388 (5)	O22—H26	0.847
C11—H111	0.961		
C2—O1—C17	113.3 (3)	C13—C12—H121	119.1
O1—C2—C3	111.0 (3)	C12—C13—C14	118.6 (3)
O1—C2—C10	107.1 (3)	C12—C13—C16	120.6 (4)
C3—C2—C10	114.9 (3)	C14—C13—C16	120.8 (4)
O1—C2—H21	113.3	C13—C14—C15	120.2 (4)
C3—C2—H21	105.8	C13—C14—H141	119.8
C10—C2—H21	104.7	C15—C14—H141	120.1
C2—C3—C4	121.2 (3)	C14—C15—C10	121.4 (4)
C2—C3—C8	121.4 (4)	C14—C15—H151	119.3
C4—C3—C8	117.3 (4)	C10—C15—H151	119.3
C3—C4—C5	120.3 (4)	C13—C16—H161	108.6
C3—C4—H41	117.7	C13—C16—H162	108.5
C5—C4—H41	122.0	H161—C16—H162	110.6
C4—C5—C6	122.2 (4)	C13—C16—H163	108.9
C4—C5—H51	119.7	H161—C16—H163	108.4
C6—C5—H51	118.1	H162—C16—H163	111.8
C5—C6—C7	117.3 (4)	O1—C17—C18	112.3 (3)
C5—C6—C9	121.7 (4)	O1—C17—C21	114.1 (3)
C7—C6—C9	121.1 (4)	C18—C17—C21	102.7 (3)
C6—C7—C8	121.4 (4)	O1—C17—H171	110.2
C6—C7—H71	119.6	C18—C17—H171	106.9
C8—C7—H71	119.0	C21—C17—H171	110.2
C3—C8—C7	121.4 (4)	C17—C18—O19	109.5 (3)
C3—C8—H81	119.0	C17—C18—O23	128.6 (3)
C7—C8—H81	119.7	O19—C18—O23	122.0 (4)
C6—C9—H91	109.8	C18—O19—C20	109.5 (3)
C6—C9—H92	107.4	O19—C20—C21	105.3 (3)
H91—C9—H92	108.6	O19—C20—H201	107.8
C6—C9—H93	109.6	C21—C20—H201	112.1
H91—C9—H93	113.2	O19—C20—H202	111.4
H92—C9—H93	108.1	C21—C20—H202	112.5

C2—C10—C11	123.3 (4)	H201—C20—H202	107.7
C2—C10—C15	118.3 (3)	C20—C21—C17	99.7 (4)
C11—C10—C15	118.4 (3)	C20—C21—O22	108.7 (3)
C10—C11—C12	120.1 (4)	C17—C21—O22	111.7 (3)
C10—C11—H111	120.0	C20—C21—H211	111.9
C12—C11—H111	119.8	C17—C21—H211	111.6
C11—C12—C13	121.2 (4)	O22—C21—H211	112.5
C11—C12—H121	119.7	C21—O22—H26	128.3

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H41···O23^{i}	0.96	2.53	3.461 (6)	165
O22—H26···O23^{i}	0.85	2.19	2.863 (6)	137

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

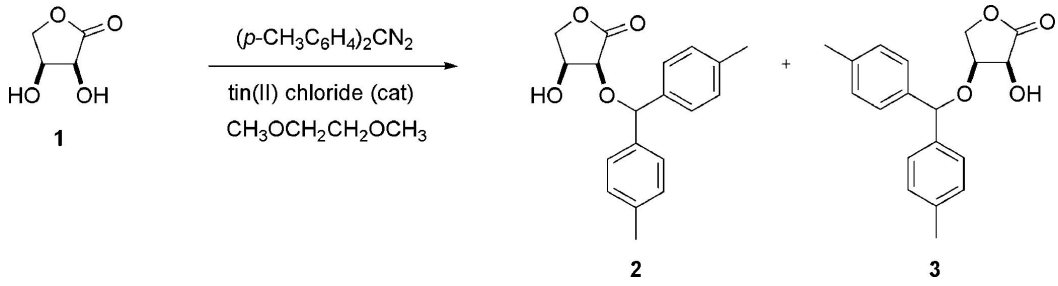
(p-CH3C6H4)2CN2
tin(II) chloride (cat)
CH3OCH2CH2OCH3
HO
OH
O
O
1
2
3
+

Fig. 2

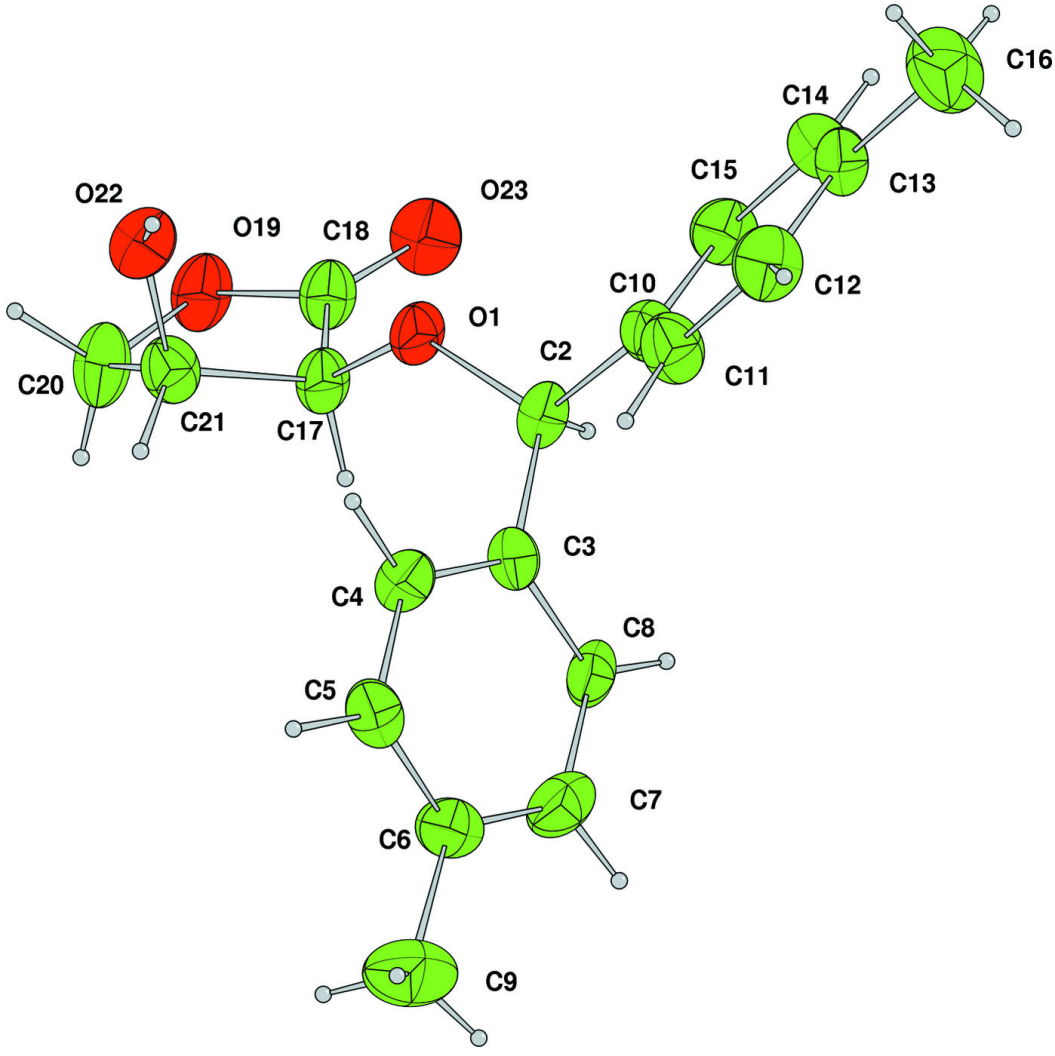
C16
C14
C15
C13
O22
O23
O19
C18
C10
C12
O1
C2
C11
C20
C21
C17
C3
C4
C8
C5
C7
C6
C9

Fig. 3