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Cedrelone butyrolactone

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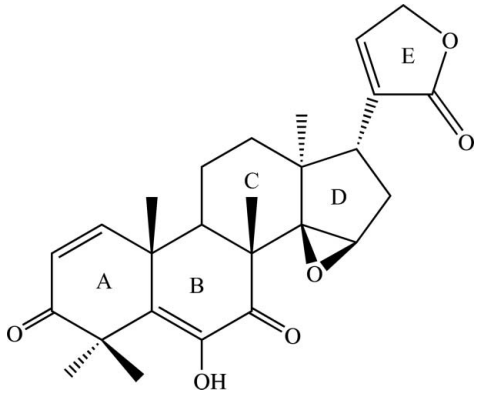
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.047; wR factor = 0.125; data-to-parameter ratio = 10.2.

The title compound (systematic name: 24-nor-13α,14β,15β,17α-chola-1,5,20-triene-14,15:21,23-diepoxy-6-hydroxy-4,4,8-trimethyl-3,7,21-trione), $C_{26}H_{30}O_6$, is a semisynthetic derivative of cedrelone, obtained *via* microwave oxidation. Modification of the parent compound results in a change in the orientation of the furan ring; however, the conformations of the other rings are not altered. The conformations adopted by rings *A*–*E* are boat, half-chair, twist, envelope and planar, respectively. Motifs $R_2^2[10]$, $S(5)$, $C(7)$ and $C(12)$ are formed through O—H···O and C—H···O hydrogen bonds in the crystal structure.

Related literature

For related literature, see: Bernstein *et al.* (1995); Cremer & Pople (1975); Geetha Gopalakrishnan *et al.* (2000, 2001); Harris *et al.* (1968); Henderson *et al.* (1968); Narayanan *et al.* (1967); Suresh *et al.* (2002); Zeumer *et al.* (2000).



Experimental

Crystal data

$C_{26}H_{30}O_6$
M_r = 438.50
Orthorhombic, $P2_12_12_1$
a = 12.6858 (9) Å
b = 13.1117 (9) Å
c = 13.3530 (10) Å
V = 2221.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.26 × 0.21 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.974, T_{max} = 0.985
14146 measured reflections
2967 independent reflections
2364 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.08
2967 reflections
290 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O7	0.82	2.18	2.624 (3)	114
O6—H6···O3^{i}	0.82	2.15	2.897 (3)	151
C2—H2···O21^{ii}	0.93	2.48	3.239 (4)	139
C28—H28*C*···O7^{iii}	0.96	2.40	3.320 (4)	162

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PARST97* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2184).

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  doi:10.1107/S1600536807039402

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supplementary materials

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Cedrelone butyrolactone

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Comment

Cedrelone (Zeumer *et al.*, 2000), one of the major limonoids extracted from Toona ciliata, shows minimum antifeedant activity; however it was reported that its photo and microwave products show enhanced and reduced activity, respectively (Suresh *et al.*, 2002).

The present study reports the crystal structure of a microwave-modified product which has an additional carbonyl group at C21 in the furan ring. This substitution has modified only the furan ring orientation with respect to ring D. The torsion angle, C16—C17—C20—C22, showing the relative orientation of the furan ring with respect to ring D, is 25.5 (4)°, compared to 168.3 (4)° in cedrelone itself.

Ring A is in a boat conformation (Cremer & Pople, 1975). The atoms C3 and C10 deviate by 0.561 (2) Å & 0.405 (2) Å from the plane involving the other atoms of the ring. Ring B takes up a half-chair conformation, the atoms C9 and C8 deviating from the least-squares plane of the other four atoms by 0.530 (2) Å and −0.183 (2) Å. Ring C adopts a twist conformation, with the atoms C11 and C13 deviating from the plane through atoms C8, C9, C12, C14 by −0.789 (3) Å and 0.697 (2) Å. Ring D is in an envelope conformation with C17 as the flap atom, lying 0.543 (4) Å from the plane of the remaining four atoms. Ring E is in a planar conformation, the maximum deviation (for atom C22) being 0.045 (4) Å. The ring fusions at the junctions A/B, B/C and C/D are quasi-*trans*, *trans* and *trans*, respectively, as seen from the endocyclic torsion angles.

The packing of the molecules in the crystal structure (Fig. 2) is achieved through a network of O—H···O and C—H···O hydrogen bonds (Table 1). An S(5) motif is generated through the O6—H6···O7 intramolecular hydrogen bond. Three chain motifs C(7),*C*(7) and C(12) are generated through hydrogen bonds O6—H6···O3 (−1/2 + *x*, 3/2 − *y*, −*z*), C28—H28C···O7 (1/2 + *x*,3/2 − *y*,-*z*) and C2—H2···O21(1/2 + *x*, 1/2 − *y*,-*z*). These in turn generate a ring motif R_2^2[10] (Bernstein *et al.*,1995).

Experimental

The microwave-oxidized product was obtained through the procedure reported in the literature (Geetha Gopalakrishnan *et al.*, 2000; 2001). Good diffraction quality crystals were obtained from ethyl acetate/hexane (1:1) by slow evaporation.

Refinement

In the absence of significant anomalous scattering, 1980 Friedel pairs were merged. The enantiomer employed in the refined model was chosen to agree with the accepted configuration of limonoids (Henderson *et al.*, 1968; Narayanan *et al.*, 1967; Harris *et al.*, 1968). Methine and methylene H atoms were constrained to an ideal geometry (C—H = 0.98 and 0.97 Å, respectively), with U_{iso}(H) = 1.2U_{eq}(parent atom). Methyl and hydroxyl H hydrogen atoms were placed in geometrically idealized positions (C—H = 0.96 Å and O—H = 0.98 Å) and were constrained to ride on their parent atom with U_{iso}(H) = 1.5U_{eq}(C,*O*).

Figures

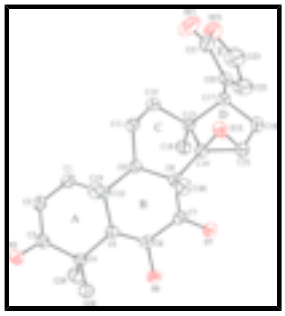


Fig. 1. Molecular structure of the title compound, with 30% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen atoms have been omitted.

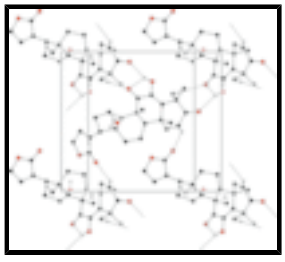


Fig. 2. A view of the crystal packing of the title compound. Dashed lines indicate hydrogen bonds. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

24-nor-13α,14β,15β,17α-chola-1,5,20-triene- 14,15:21,23-diepoxy-6-hydroxy-4,4,8-trimethyl-3,7,21-trione

Crystal data

$C_{26}H_{30}O_6$	F_{000} = 936
M_r = 438.50	D_x = 1.311 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5100 reflections
a = 12.6858 (9) Å	θ = 2.2–28.0º
b = 13.1117 (9) Å	μ = 0.09 mm^{-1}
c = 13.3530 (10) Å	T = 293 (2) K
V = 2221.0 (3) Å^3	Needle, yellow
Z = 4	0.26 × 0.21 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2967 independent reflections
Radiation source: fine-focus sealed tube	2364 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.035
T = 293(2) K	θ_{max} = 28.0º
ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→16
T_{min} = 0.974, T_{max} = 0.985	k = −7→16
14146 measured reflections	l = −17→16

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.125$

$S = 1.08$

2967 reflections

290 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

Calculated $w = 1/[\sigma^2(F_o^2) + (0.0777P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$?

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.24$ e Å^{-3}

$\Delta\rho_{min} = -0.18$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.81936 (15)	0.57248 (16)	−0.04775 (16)	0.0540 (5)
O6	0.49433 (14)	0.78724 (13)	0.06943 (16)	0.0462 (5)
H6	0.4363	0.8130	0.0794	0.069*
O7	0.34393 (15)	0.73469 (14)	0.19410 (15)	0.0484 (5)
O15	0.29074 (16)	0.48982 (17)	0.35735 (13)	0.0534 (5)
O21	0.0963 (2)	0.2091 (2)	0.1912 (3)	0.1014 (11)
O23	−0.04455 (19)	0.2598 (2)	0.1022 (2)	0.0797 (8)
C1	0.5936 (2)	0.4466 (2)	0.0257 (2)	0.0449 (6)
H1	0.5544	0.3875	0.0154	0.054*
C2	0.6681 (2)	0.4702 (2)	−0.0389 (2)	0.0474 (6)
H2	0.6833	0.4270	−0.0922	0.057*
C3	0.7267 (2)	0.5652 (2)	−0.02574 (19)	0.0405 (6)
C4	0.6621 (2)	0.65651 (19)	0.00986 (18)	0.0375 (5)
C5	0.57172 (18)	0.62388 (19)	0.07896 (17)	0.0338 (5)
C6	0.49469 (19)	0.68980 (18)	0.10440 (18)	0.0362 (5)
C7	0.40724 (19)	0.66755 (18)	0.17463 (17)	0.0353 (5)

C8	0.40818 (19)	0.56473 (19)	0.22575 (17)	0.0350 (5)
C9	0.45650 (19)	0.48718 (18)	0.15132 (18)	0.0351 (5)
H9	0.4137	0.4952	0.0907	0.042*
C10	0.56963 (18)	0.51229 (19)	0.11602 (19)	0.0360 (5)
C11	0.4311 (2)	0.3790 (2)	0.1872 (2)	0.0432 (6)
H11A	0.4449	0.3732	0.2584	0.052*
H11B	0.4753	0.3301	0.1523	0.052*
C12	0.3141 (2)	0.35638 (19)	0.1657 (2)	0.0434 (6)
H12A	0.2907	0.3012	0.2088	0.052*
H12B	0.3066	0.3341	0.0968	0.052*
C13	0.24314 (18)	0.45085 (18)	0.18341 (18)	0.0353 (5)
C14	0.2988 (2)	0.5246 (2)	0.25409 (17)	0.0372 (5)
C15	0.2220 (2)	0.5690 (2)	0.3221 (2)	0.0521 (7)
H15	0.2306	0.6392	0.3459	0.062*
C16	0.1141 (2)	0.5284 (2)	0.2979 (2)	0.0551 (8)
H16A	0.0756	0.5751	0.2550	0.066*
H16B	0.0736	0.5163	0.3584	0.066*
C17	0.1385 (2)	0.4279 (2)	0.2433 (2)	0.0422 (6)
H17	0.1554	0.3768	0.2944	0.051*
C18	0.2138 (2)	0.5065 (2)	0.0863 (2)	0.0434 (6)
H18A	0.1788	0.4600	0.0419	0.065*
H18B	0.2766	0.5318	0.0549	0.065*
H18C	0.1677	0.5625	0.1015	0.065*
C19	0.6576 (2)	0.4912 (2)	0.1933 (2)	0.0519 (7)
H19A	0.6576	0.4201	0.2105	0.078*
H19B	0.6453	0.5311	0.2524	0.078*
H19C	0.7247	0.5092	0.1650	0.078*
C20	0.0501 (2)	0.3867 (2)	0.1819 (2)	0.0471 (7)
C21	0.0405 (3)	0.2773 (3)	0.1616 (3)	0.0637 (9)
C22	−0.0315 (2)	0.4304 (3)	0.1377 (4)	0.0765 (11)
H22	−0.0468	0.4997	0.1407	0.092*
C23	−0.0956 (3)	0.3537 (3)	0.0829 (4)	0.0822 (12)
H23A	−0.1675	0.3527	0.1075	0.099*
H23B	−0.0965	0.3683	0.0117	0.099*
C28	0.6164 (2)	0.6976 (2)	−0.0908 (2)	0.0513 (7)
H28A	0.5738	0.7566	−0.0779	0.077*
H28B	0.5740	0.6457	−0.1216	0.077*
H28C	0.6733	0.7157	−0.1347	0.077*
C29	0.7320 (2)	0.7357 (2)	0.0607 (2)	0.0494 (7)
H29A	0.6896	0.7922	0.0823	0.074*
H29B	0.7843	0.7593	0.0142	0.074*
H29C	0.7661	0.7055	0.1176	0.074*
C30	0.4720 (2)	0.5847 (2)	0.3232 (2)	0.0505 (7)
H30A	0.5347	0.6223	0.3073	0.076*
H30B	0.4909	0.5208	0.3533	0.076*
H30C	0.4297	0.6234	0.3692	0.076*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0394 (10)	0.0535 (11)	0.0692 (13)	−0.0030 (9)	0.0126 (9)	−0.0034 (10)
O6	0.0425 (10)	0.0331 (9)	0.0629 (12)	0.0020 (8)	0.0026 (9)	0.0069 (9)
O7	0.0473 (10)	0.0382 (10)	0.0597 (12)	0.0082 (9)	0.0059 (9)	0.0002 (9)
O15	0.0693 (13)	0.0627 (13)	0.0282 (9)	−0.0034 (11)	0.0059 (8)	0.0048 (9)
O21	0.0861 (18)	0.0517 (15)	0.166 (3)	0.0126 (14)	−0.017 (2)	−0.0224 (19)
O23	0.0543 (13)	0.0725 (16)	0.112 (2)	−0.0035 (12)	−0.0076 (14)	−0.0265 (16)
C1	0.0428 (13)	0.0369 (14)	0.0550 (15)	−0.0009 (11)	0.0022 (12)	−0.0066 (12)
C2	0.0475 (15)	0.0449 (15)	0.0497 (15)	−0.0020 (13)	0.0094 (12)	−0.0145 (13)
C3	0.0402 (13)	0.0446 (14)	0.0368 (12)	−0.0032 (11)	0.0019 (10)	−0.0023 (11)
C4	0.0368 (12)	0.0366 (13)	0.0392 (12)	−0.0040 (11)	−0.0006 (10)	−0.0007 (10)
C5	0.0329 (11)	0.0343 (12)	0.0343 (11)	−0.0034 (10)	−0.0049 (9)	−0.0014 (10)
C6	0.0388 (12)	0.0302 (12)	0.0395 (13)	−0.0016 (10)	−0.0049 (10)	0.0024 (10)
C7	0.0384 (12)	0.0325 (12)	0.0351 (11)	0.0004 (10)	−0.0059 (10)	−0.0065 (10)
C8	0.0381 (12)	0.0346 (13)	0.0322 (11)	0.0015 (11)	−0.0013 (9)	0.0015 (10)
C9	0.0368 (12)	0.0308 (12)	0.0378 (12)	0.0030 (10)	−0.0012 (10)	0.0026 (10)
C10	0.0351 (12)	0.0325 (12)	0.0403 (12)	0.0019 (10)	−0.0011 (10)	0.0015 (10)
C11	0.0458 (14)	0.0344 (13)	0.0496 (14)	0.0072 (11)	0.0062 (12)	0.0065 (12)
C12	0.0495 (14)	0.0314 (13)	0.0494 (14)	−0.0012 (11)	0.0101 (12)	0.0022 (11)
C13	0.0407 (13)	0.0325 (12)	0.0326 (11)	0.0016 (10)	0.0062 (10)	0.0010 (10)
C14	0.0451 (13)	0.0381 (13)	0.0284 (11)	0.0029 (11)	0.0023 (10)	0.0032 (10)
C15	0.0622 (17)	0.0466 (16)	0.0474 (15)	−0.0040 (14)	0.0177 (13)	−0.0122 (13)
C16	0.0556 (17)	0.0500 (16)	0.0599 (18)	0.0012 (14)	0.0230 (14)	−0.0108 (14)
C17	0.0443 (13)	0.0378 (13)	0.0446 (14)	0.0016 (12)	0.0112 (11)	0.0029 (11)
C18	0.0472 (15)	0.0453 (15)	0.0377 (13)	−0.0047 (12)	−0.0013 (10)	0.0048 (12)
C19	0.0441 (14)	0.0576 (18)	0.0539 (16)	0.0107 (13)	−0.0051 (12)	0.0109 (14)
C20	0.0410 (13)	0.0437 (15)	0.0565 (16)	0.0018 (12)	0.0145 (13)	0.0042 (14)
C21	0.0500 (16)	0.0528 (18)	0.088 (2)	0.0009 (15)	0.0083 (17)	−0.0156 (18)
C22	0.0462 (17)	0.058 (2)	0.125 (3)	−0.0034 (16)	−0.007 (2)	0.011 (2)
C23	0.0549 (19)	0.081 (3)	0.111 (3)	−0.005 (2)	−0.012 (2)	0.001 (2)
C28	0.0538 (16)	0.0568 (18)	0.0432 (15)	−0.0037 (14)	0.0000 (12)	0.0081 (13)
C29	0.0430 (14)	0.0467 (15)	0.0585 (17)	−0.0114 (13)	0.0012 (12)	−0.0101 (14)
C30	0.0580 (16)	0.0553 (16)	0.0382 (14)	0.0027 (14)	−0.0083 (12)	−0.0035 (13)

Geometric parameters (Å, °)

O3—C3	1.216 (3)	C12—H12A	0.9700
O6—C6	1.360 (3)	C12—H12B	0.9700
O6—H6	0.8200	C13—C14	1.525 (4)
O7—C7	1.220 (3)	C13—C18	1.533 (3)
O15—C15	1.436 (4)	C13—C17	1.578 (3)
O15—C14	1.456 (3)	C14—C15	1.454 (4)
O21—C21	1.206 (4)	C15—C16	1.503 (4)
O23—C21	1.359 (4)	C15—H15	0.9800
O23—C23	1.416 (5)	C16—C17	1.538 (4)
C1—C2	1.317 (4)	C16—H16A	0.9700

C1—C10	1.514 (4)	C16—H16B	0.9700
C1—H1	0.9300	C17—C20	1.491 (4)
C2—C3	1.461 (4)	C17—H17	0.9800
C2—H2	0.9300	C18—H18A	0.9600
C3—C4	1.527 (4)	C18—H18B	0.9600
C4—C29	1.526 (3)	C18—H18C	0.9600
C4—C5	1.532 (3)	C19—H19A	0.9600
C4—C28	1.559 (4)	C19—H19B	0.9600
C5—C6	1.348 (3)	C19—H19C	0.9600
C5—C10	1.545 (3)	C20—C22	1.323 (4)
C6—C7	1.482 (3)	C20—C21	1.465 (4)
C7—O7	1.220 (3)	C22—C23	1.486 (5)
C7—C8	1.511 (3)	C22—H22	0.9300
C8—C14	1.531 (4)	C23—H23A	0.9700
C8—C9	1.548 (3)	C23—H23B	0.9700
C8—C30	1.555 (3)	C28—H28A	0.9600
C9—C11	1.531 (3)	C28—H28B	0.9600
C9—C10	1.546 (3)	C28—H28C	0.9600
C9—H9	0.9800	C29—H29A	0.9600
C10—C19	1.545 (4)	C29—H29B	0.9600
C11—C12	1.540 (4)	C29—H29C	0.9600
C11—H11A	0.9700	C30—H30A	0.9600
C11—H11B	0.9700	C30—H30B	0.9600
C12—C13	1.549 (3)	C30—H30C	0.9600
C6—O6—H6	109.5	C15—C14—C8	128.6 (2)
C15—O15—C14	60.35 (17)	O15—C14—C8	113.9 (2)
C21—O23—C23	108.8 (3)	C13—C14—C8	119.0 (2)
C2—C1—C10	122.2 (2)	O15—C15—C14	60.51 (17)
C2—C1—H1	118.9	O15—C15—C16	111.5 (3)
C10—C1—H1	118.9	C14—C15—C16	109.5 (2)
C1—C2—C3	119.1 (2)	O15—C15—H15	120.3
C1—C2—H2	120.4	C14—C15—H15	120.3
C3—C2—H2	120.4	C16—C15—H15	120.3
O3—C3—C2	122.0 (3)	C15—C16—C17	102.8 (2)
O3—C3—C4	122.2 (2)	C15—C16—H16A	111.2
C2—C3—C4	115.7 (2)	C17—C16—H16A	111.2
C29—C4—C3	111.1 (2)	C15—C16—H16B	111.2
C29—C4—C5	110.9 (2)	C17—C16—H16B	111.2
C3—C4—C5	111.7 (2)	H16A—C16—H16B	109.1
C29—C4—C28	111.4 (2)	C20—C17—C16	114.9 (2)
C3—C4—C28	101.7 (2)	C20—C17—C13	115.0 (2)
C5—C4—C28	109.7 (2)	C16—C17—C13	104.3 (2)
C6—C5—C4	121.0 (2)	C20—C17—H17	107.4
C6—C5—C10	120.9 (2)	C16—C17—H17	107.4
C4—C5—C10	118.1 (2)	C13—C17—H17	107.4
C5—C6—O6	121.2 (2)	C13—C18—H18A	109.5
C5—C6—C7	125.2 (2)	C13—C18—H18B	109.5
O6—C6—C7	113.6 (2)	H18A—C18—H18B	109.5
O7—C7—C6	119.1 (2)	C13—C18—H18C	109.5

O7—C7—C8	123.6 (2)	H18A—C18—H18C	109.5
C6—C7—C8	117.1 (2)	H18B—C18—H18C	109.5
C7—C8—C14	114.3 (2)	C10—C19—H19A	109.5
C7—C8—C9	107.40 (19)	C10—C19—H19B	109.5
C14—C8—C9	106.9 (2)	H19A—C19—H19B	109.5
C7—C8—C30	103.4 (2)	C10—C19—H19C	109.5
C14—C8—C30	108.8 (2)	H19A—C19—H19C	109.5
C9—C8—C30	116.2 (2)	H19B—C19—H19C	109.5
C11—C9—C10	119.2 (2)	C22—C20—C21	106.1 (3)
C11—C9—C8	108.9 (2)	C22—C20—C17	132.6 (3)
C10—C9—C8	115.0 (2)	C21—C20—C17	121.3 (3)
C11—C9—H9	103.9	O21—C21—O23	122.1 (3)
C10—C9—H9	103.9	O21—C21—C20	128.1 (3)
C8—C9—H9	103.9	O23—C21—C20	109.8 (3)
C1—C10—C5	106.3 (2)	C20—C22—C23	110.7 (3)
C1—C10—C19	106.6 (2)	C20—C22—H22	124.6
C5—C10—C19	111.8 (2)	C23—C22—H22	124.6
C1—C10—C9	107.9 (2)	O23—C23—C22	104.4 (3)
C5—C10—C9	108.38 (19)	O23—C23—H23A	110.9
C19—C10—C9	115.4 (2)	C22—C23—H23A	110.9
C9—C11—C12	108.8 (2)	O23—C23—H23B	110.9
C9—C11—H11A	109.9	C22—C23—H23B	110.9
C12—C11—H11A	109.9	H23A—C23—H23B	108.9
C9—C11—H11B	109.9	C4—C28—H28A	109.5
C12—C11—H11B	109.9	C4—C28—H28B	109.5
H11A—C11—H11B	108.3	H28A—C28—H28B	109.5
C11—C12—C13	112.2 (2)	C4—C28—H28C	109.5
C11—C12—H12A	109.2	H28A—C28—H28C	109.5
C13—C12—H12A	109.2	H28B—C28—H28C	109.5
C11—C12—H12B	109.2	C4—C29—H29A	109.5
C13—C12—H12B	109.2	C4—C29—H29B	109.5
H12A—C12—H12B	107.9	H29A—C29—H29B	109.5
C14—C13—C18	109.5 (2)	C4—C29—H29C	109.5
C14—C13—C12	109.4 (2)	H29A—C29—H29C	109.5
C18—C13—C12	113.1 (2)	H29B—C29—H29C	109.5
C14—C13—C17	101.3 (2)	C8—C30—H30A	109.5
C18—C13—C17	108.3 (2)	C8—C30—H30B	109.5
C12—C13—C17	114.4 (2)	H30A—C30—H30B	109.5
C15—C14—O15	59.14 (17)	C8—C30—H30C	109.5
C15—C14—C13	109.3 (2)	H30A—C30—H30C	109.5
O15—C14—C13	110.8 (2)	H30B—C30—H30C	109.5
C10—C1—C2—C3	−2.8 (4)	C11—C12—C13—C14	−22.2 (3)
C1—C2—C3—O3	145.9 (3)	C11—C12—C13—C18	100.2 (3)
C1—C2—C3—C4	−38.6 (4)	C11—C12—C13—C17	−135.1 (2)
O3—C3—C4—C29	−27.5 (3)	C15—O15—C14—C13	−100.6 (2)
C2—C3—C4—C29	157.0 (2)	C15—O15—C14—C8	122.0 (3)
O3—C3—C4—C5	−151.9 (2)	C18—C13—C14—C15	94.1 (2)
C2—C3—C4—C5	32.5 (3)	C12—C13—C14—C15	−141.4 (2)
O3—C3—C4—C28	91.1 (3)	C17—C13—C14—C15	−20.2 (3)

C2—C3—C4—C28	−84.4 (3)	C18—C13—C14—O15	157.5 (2)
C29—C4—C5—C6	67.1 (3)	C12—C13—C14—O15	−78.0 (3)
C3—C4—C5—C6	−168.3 (2)	C17—C13—C14—O15	43.2 (2)
C28—C4—C5—C6	−56.3 (3)	C18—C13—C14—C8	−67.6 (3)
C29—C4—C5—C10	−114.7 (2)	C12—C13—C14—C8	56.9 (3)
C3—C4—C5—C10	9.9 (3)	C17—C13—C14—C8	178.1 (2)
C28—C4—C5—C10	121.8 (2)	C7—C8—C14—C15	−61.7 (3)
C4—C5—C6—O6	0.2 (4)	C9—C8—C14—C15	179.5 (3)
C10—C5—C6—O6	−177.9 (2)	C30—C8—C14—C15	53.2 (3)
C4—C5—C6—C7	−176.6 (2)	C7—C8—C14—O15	−130.4 (2)
C10—C5—C6—C7	5.2 (4)	C9—C8—C14—O15	110.9 (2)
C5—C6—C7—O7	177.8 (2)	C30—C8—C14—O15	−15.4 (3)
O6—C6—C7—O7	0.7 (3)	C7—C8—C14—C13	96.0 (3)
C5—C6—C7—C8	3.5 (3)	C9—C8—C14—C13	−22.7 (3)
O6—C6—C7—C8	−173.6 (2)	C30—C8—C14—C13	−149.0 (2)
O7—C7—C8—C14	34.2 (3)	C14—O15—C15—C16	100.9 (2)
C6—C7—C8—C14	−151.8 (2)	C13—C14—C15—O15	103.3 (2)
O7—C7—C8—C9	152.6 (2)	C8—C14—C15—O15	−97.3 (3)
C6—C7—C8—C9	−33.3 (3)	O15—C14—C15—C16	−104.3 (3)
O7—C7—C8—C30	−83.9 (3)	C13—C14—C15—C16	−1.0 (3)
C6—C7—C8—C30	90.1 (2)	C8—C14—C15—C16	158.4 (3)
C7—C8—C9—C11	−164.3 (2)	O15—C15—C16—C17	−42.6 (3)
C14—C8—C9—C11	−41.2 (3)	C14—C15—C16—C17	22.5 (3)
C30—C8—C9—C11	80.5 (3)	C15—C16—C17—C20	−161.3 (2)
C7—C8—C9—C10	58.9 (3)	C15—C16—C17—C13	−34.5 (3)
C14—C8—C9—C10	−178.05 (19)	C14—C13—C17—C20	160.2 (2)
C30—C8—C9—C10	−56.3 (3)	C18—C13—C17—C20	45.0 (3)
C2—C1—C10—C5	43.0 (3)	C12—C13—C17—C20	−82.2 (3)
C2—C1—C10—C19	−76.4 (3)	C14—C13—C17—C16	33.5 (3)
C2—C1—C10—C9	159.1 (3)	C18—C13—C17—C16	−81.7 (3)
C6—C5—C10—C1	133.6 (2)	C12—C13—C17—C16	151.1 (2)
C4—C5—C10—C1	−44.6 (3)	C16—C17—C20—C22	25.6 (5)
C6—C5—C10—C19	−110.5 (3)	C13—C17—C20—C22	−95.5 (4)
C4—C5—C10—C19	71.3 (3)	C16—C17—C20—C21	−153.1 (3)
C6—C5—C10—C9	17.7 (3)	C13—C17—C20—C21	85.8 (4)
C4—C5—C10—C9	−160.42 (19)	C23—O23—C21—O21	178.1 (4)
C11—C9—C10—C1	62.3 (3)	C23—O23—C21—C20	−1.4 (4)
C8—C9—C10—C1	−165.6 (2)	C22—C20—C21—O21	−176.9 (4)
C11—C9—C10—C5	177.0 (2)	C17—C20—C21—O21	2.1 (6)
C8—C9—C10—C5	−50.8 (3)	C22—C20—C21—O23	2.5 (4)
C11—C9—C10—C19	−56.8 (3)	C17—C20—C21—O23	−178.4 (2)
C8—C9—C10—C19	75.4 (3)	C21—C20—C22—C23	−2.6 (4)
C10—C9—C11—C12	−150.8 (2)	C17—C20—C22—C23	178.5 (3)
C8—C9—C11—C12	74.4 (3)	C21—O23—C23—C22	−0.1 (4)
C9—C11—C12—C13	−37.8 (3)	C20—C22—C23—O23	1.8 (5)

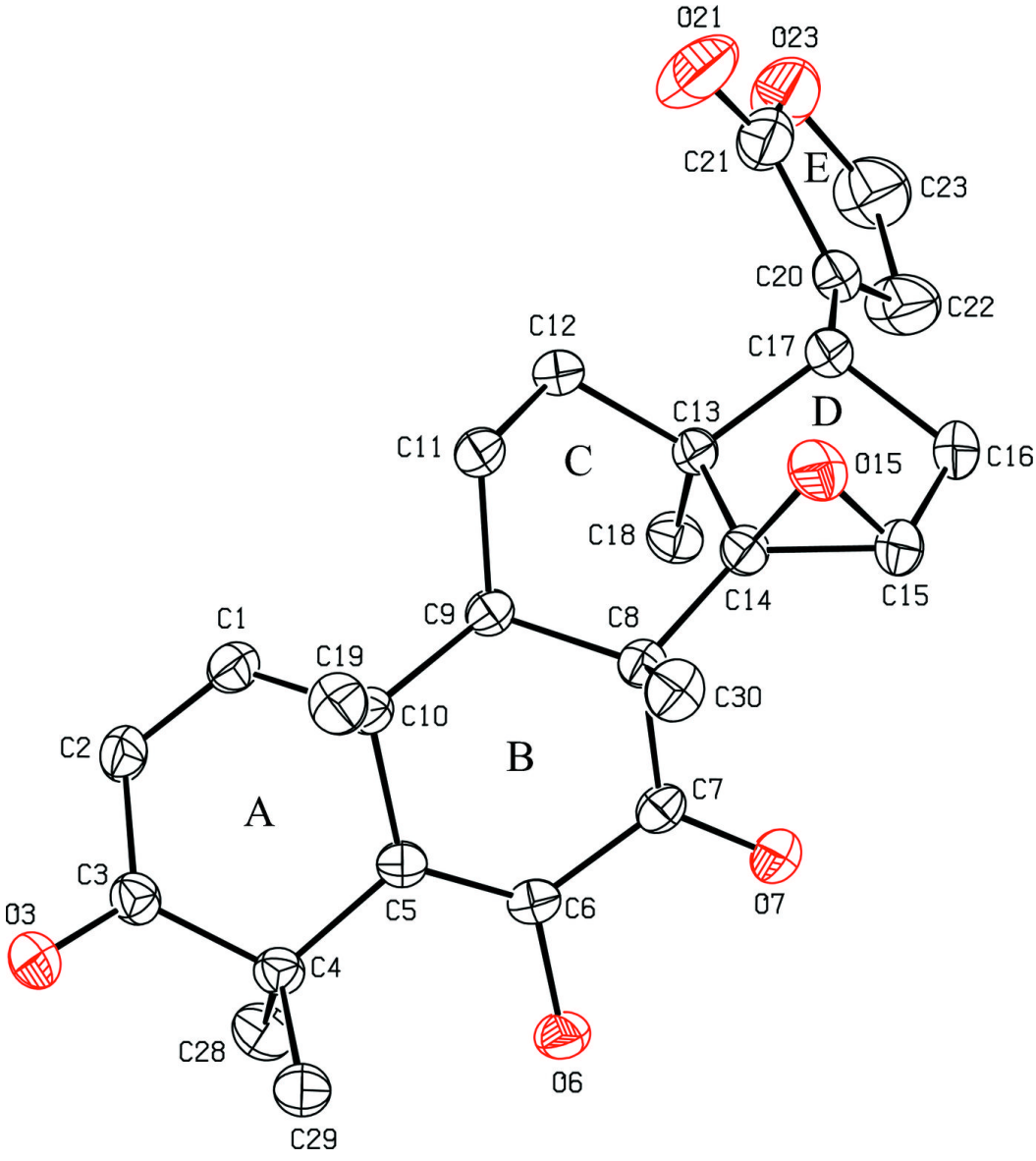
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O6—H6···O7	0.82	2.18	2.624 (3)	114
O6—H6···O3^{i}	0.82	2.15	2.897 (3)	151
C2—H2···O21^{ii}	0.93	2.48	3.239 (4)	139
C28—H28C···O7^{iii}	0.96	2.40	3.320 (4)	162

Symmetry codes: (i) *x*−1/2, −*y*+3/2, −*z*; (ii) *x*+1/2, −*y*+1/2, −*z*; (iii) *x*+1/2, −*y*+3/2, −*z*.

Fig. 1


O21
O23
C21
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C14
C15
C1
C9
C8
C19
C30
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O3
C5
C6
O7
C4
C28
O6
C29

Fig. 2