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Isocedrelone

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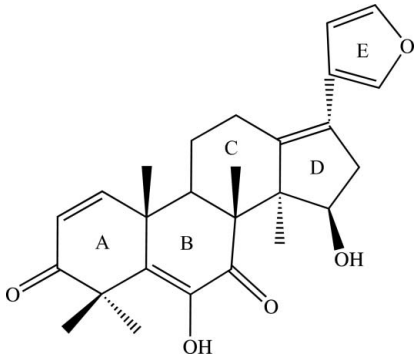
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.059; wR factor = 0.188; data-to-parameter ratio = 12.4.

The title compound (systematic name: 17-furan-3-yl-6,15-dihydroxy-4,4,8,10,14-pentamethyl-8,9,10,11,12,14,15,16-octahydro-4*H*-cyclopenta[*a*]phenanthrene-3,7-dione), $C_{26}H_{30}O_5$, is a semi-synthetic derivative of cedrelone, a tetranortriterpenoid isolated from *Toona ciliata*. Both cedrelone and the title compound show similar antifeedant activity against third instar larvae of *Spodoptera litura*. The modification of the *D* ring of the parent compound has altered the conformation of ring *C*; however, the orientation of the furan ring as well as the conformations of other rings remain the same. The three fused six-membered rings adopt a boat, a half-chair and a chair conformation and the five-membered rings *D* and *E* adopt envelope and planar conformations, respectively. A macrocyclic ring motif, $R_7^6(40)$, $S(5)$ and $S(7)$, generated by C—H···O and O—H···O hydrogen bonds, stabilizes the molecules in the crystal structure.

Related literature

Several cedrelone derivatives have been synthesized through classical chemical modifications and the crystal structures of cedrelone (Zeumer *et al.*, 2000) and a few derivatives have been reported. For related literature, see: Bernstein *et al.* (1995); Cremer & Pople (1975); Flack (1983); Flack & Bernardinelli (2000); Narayanan *et al.* (1980).



Experimental

Crystal data

$C_{26}H_{30}O_5$
M_r = 422.50
Orthorhombic, $P2_12_12_1$
a = 11.639 (4) Å
b = 13.027 (2) Å
c = 14.417 (5) Å
V = 2185.9 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.23 × 0.21 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
3810 measured reflections
3557 independent reflections
1738 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
3 standard reflections
every 120 reflections
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.188
S = 1.02
3557 reflections
287 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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	2.10	2.573 (4)	117
O15—H15A···O7	0.82	1.90	2.687 (4)	162
C11—H11A···O3^{i}	0.97	2.55	3.431 (6)	151
C11—H11B···O7^{ii}	0.97	2.56	3.458 (5)	155

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: RK2029).

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supplementary materials

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Isocedrelone

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Comment

An intact limonoid Cedrelone, has been previously isolated from *Toona ciliata* and its three dimensional structure was reported earlier (Zeumer *et al.*, 2000). It possess half the antifeedant activity of the most potent, Azadirachtin–A from *Azadirachta indica*. The title compound, a semisynthetic derivative of cedrelone differs chemically from the parent molecule by the modification of ring *D*. Abstraction of C17 proton has resulted in the shift of C13 methyl group to C14, opening up of epoxide ring between C14—C15, formation of hydroxyl at C15 and the double bond between C13═C17. This modification has altered the ring conformations and the orientation of furan ring with respect to ring *D* is C16—C17—C20—C22 = 164.1 (5)° for (I) and 168.3 (4)° for cedrelone, compared with parent compound (Zeumer *et al.*, 2000);

Ring *A* [Q_T = 0.522 (4) Å, φ_2 = −68.6 (5)°, q_2 = 0.515 (4) Å] is in a boat conformation. The atoms C1 and C10 deviate by 0.165 (4)Å and 0.734 (3)Å from the plane involving the other four atoms of the ring. Ring *B* [Q_T = 0.418 (3) Å, φ_2 = −138.4 (6)°, q_2 = 0.331 (3) Å] adopts a half–chair conformation as in cedrelone (Zeumer *et al.*, 2000). The atoms C8 and C9 deviate from the LSQ–plane of the other four atoms by 0.209 (3)Å and −0.427 (3)Å respectively. Ring *C* takes up a chair conformation [Q_T = 0.586 (4) Å, φ_2 = −4.85(8.12)°, q_2 =0.027 (4) Å] with atoms C8 and C12 deviating from the plane by 0.717 (3)Å and −0.667 (4)Å respectively from the plane of other four atoms of the ring. It adopts a twist conformation in cedrelone. Rings *D* and *E* are in an envelope [φ_2 = 70.23(1.12)°, q_2 = 0.205 (4)Å for ring *D*] and a planar conformation (Nardelli, 1995) respectively (Cremer & Pople, 1975).

Ring motifs are generated through O—H···O and C—H···O hydrogen bonds in the crystal lattice (Fig 2). Two ring motifs *S*(5) and *S*(7) are genereated through hydrogen bonds O6—H6···O7 and O15—H15A···O7 respectively. A macrocyclic ring motif R_7^6[40] (Bernstein *et al.*, 1995) is generated through hydrogen bonds C11—H11A···O3 [$2 - x, -1/2 + y, 3/2 - z$] and C19—H11B···O7 [$3/2 - x, 2 - y, -1/2 + z$].

Experimental

To a solution of Cedrelone (40 mg, 0.085 mmol) in acetone (3 ml) 400 mg of the freshly prepared *N*–bromoacetamide resin was added and placed under the microwave with stirring for 3 min. The reaction was monitored by TLC using Ethylacetate and hexane in the ratio 1:1). The resin was then filtered and was washed three times with 2 ml of acetone. The solvent was removed under reduced pressure. The crude product was chromotographed on silica gel (70–325 mesh) using an eluant ethylacetate and hexane, in the increasing order of polarity. Elution of the column using ethylacetate/hexane = 10/90 yielded compound (I).

Refinement

In the absence of suitable anomalous scatters, Friedel equivalents could not be used to determine the absolute structure. Refinement of the Flack parameter (Flack, 1983) led to inconclusive values (Flack & Bernardinelli, 2000) for this parameter

[1(3)]. Therefore, 3557 Friedel equivalents were merged before the final refinement. The enantiomer employed in the refined model was chosen to agree with the accepted configuration of tetranortriterpenoids (Narayanan *et al.*, 1980).

The C—H and CH_2, atoms were constrained to an ideal geometry (CH = 0.98, CH_2 = 0.97, OH = 0.82 Å) with U_{iso}(H) = 1.2U_{eq}(C), but where allowed to rotate freely about the C—C and C—O bonds, respectively. For CH_3 and OH, hydrogen atoms were constrained to ride on their parent atom with U_{iso}(H) = 1.5U_{eq}(parent atom).

Figures

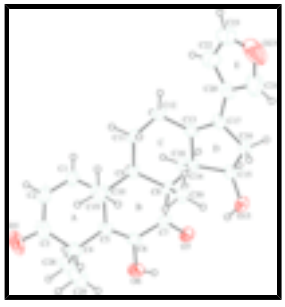

Fig. 1. Molecular structure and atomic numbering schemeof (I) with 30% probability displacement ellipsoids and atomic numbering scheme. The H atoms are presented as a spheres of arbitrary radius.

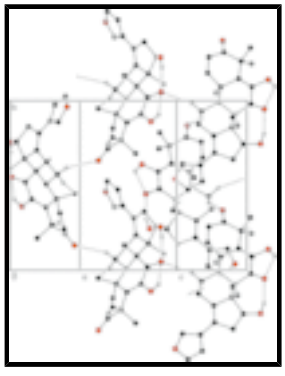

Fig. 2. A view of the crystal packing of (I) view down 'b' axis with the hydrogen bonds. Hydrogen atoms not involved in hydrogen bonds have been omitted for clarity.

17-furan-3-yl-6,15-dihydroxy-4,4,8,10,14-pentamethyl-8,9,10,11,12,14,15,16- octahydro-4*H*-cyclopenta[*a*]phenanthrene-3,7-dione

Crystal data

$C_{26}H_{30}O_5$	F_{000} = 904
M_r = 422.50	D_x = 1.284 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 11.639 (4) Å	θ = 5–11°
b = 13.027 (2) Å	μ = 0.09 mm^{-1}
c = 14.417 (5) Å	T = 293 (2) K
V = 2185.9 (11) Å^3	Prism, yellow
Z = 4	0.23 × 0.21 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.022
Radiation source: fine–focus sealed tube	θ_{max} = 30.0º
Monochromator: graphite	θ_{min} = 2.1º
T = 293(2) K	h = 0→16
ω scans	k = 0→18
Absorption correction: none	l = −1→20
3810 measured reflections	3 standard reflections
3557 independent reflections	every 120 reflections
1738 reflections with $I > 2\sigma(I)$	intensity decay: 2%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.059	$w = 1/[\sigma^2(F_o^2) + (0.0895P)^2 + 0.3096P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.188	$(\Delta/\sigma)_{max}$ = 0.042
S = 1.02	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
3557 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
287 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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}
C1	0.8295 (5)	1.1629 (3)	0.8368 (3)	0.0636 (11)
H1	0.7581	1.1466	0.8118	0.076*
C2	0.8688 (5)	1.2571 (3)	0.8284 (3)	0.0790 (14)

H2	0.8265	1.3061	0.7960	0.095*
C3	0.9800 (6)	1.2848 (4)	0.8704 (4)	0.0847 (16)
C4	1.0118 (4)	1.2324 (3)	0.9612 (3)	0.0623 (11)
C5	0.9509 (3)	1.1291 (3)	0.9719 (3)	0.0502 (9)
C6	0.9440 (3)	1.0809 (3)	1.0537 (3)	0.0537 (9)
C7	0.8920 (3)	0.9804 (3)	1.0695 (2)	0.0497 (9)
C8	0.8527 (3)	0.9158 (3)	0.9891 (2)	0.0428 (8)
C9	0.8120 (3)	0.9916 (3)	0.9116 (2)	0.0427 (8)
H9	0.7444	1.0262	0.9375	0.051*
C10	0.8975 (3)	1.0802 (3)	0.8861 (2)	0.0486 (9)
C11	0.7683 (4)	0.9354 (3)	0.8260 (3)	0.0570 (10)
H11A	0.8307	0.8966	0.7986	0.068*
H11B	0.7420	0.9851	0.7806	0.068*
C12	0.6694 (4)	0.8622 (3)	0.8501 (3)	0.0641 (11)
H12A	0.6031	0.9010	0.8710	0.077*
H12B	0.6474	0.8230	0.7957	0.077*
C13	0.7087 (3)	0.7923 (3)	0.9242 (3)	0.0499 (9)
C14	0.7492 (3)	0.8424 (3)	1.0148 (2)	0.0464 (8)
C15	0.7747 (4)	0.7430 (3)	1.0737 (3)	0.0588 (10)
H15	0.7050	0.7293	1.1098	0.071*
C16	0.7876 (4)	0.6542 (3)	1.0074 (3)	0.0621 (11)
H16A	0.8680	0.6389	0.9962	0.075*
H16B	0.7499	0.5933	1.0311	0.075*
C17	0.7301 (3)	0.6918 (3)	0.9207 (3)	0.0530 (9)
C18	0.6518 (3)	0.9029 (3)	1.0623 (3)	0.0620 (11)
H18A	0.6801	0.9331	1.1186	0.093*
H18B	0.5894	0.8573	1.0764	0.093*
H18C	0.6253	0.9561	1.0215	0.093*
C19	0.9946 (4)	1.0471 (3)	0.8182 (3)	0.0697 (13)
H19A	0.9612	1.0156	0.7643	0.105*
H19B	1.0444	0.9989	0.8485	0.105*
H19C	1.0380	1.1063	0.7998	0.105*
C20	0.7107 (4)	0.6200 (3)	0.8442 (3)	0.0631 (12)
C21	0.7550 (9)	0.5260 (4)	0.8367 (5)	0.128 (3)
H21	0.8024	0.4972	0.8817	0.153*
C22	0.6395 (6)	0.6265 (5)	0.7665 (4)	0.107 (2)
H22	0.5935	0.6827	0.7520	0.128*
C23	0.6464 (7)	0.5425 (5)	0.7167 (4)	0.116 (2)
H23	0.6059	0.5289	0.6625	0.139*
C28	0.9729 (5)	1.3119 (4)	1.0330 (4)	0.0911 (17)
H28A	1.0141	1.3749	1.0234	0.137*
H28B	0.9883	1.2865	1.0943	0.137*
H28C	0.8920	1.3241	1.0263	0.137*
C29	1.1425 (5)	1.2162 (4)	0.9664 (5)	0.1022 (19)
H29A	1.1807	1.2812	0.9603	0.153*
H29B	1.1663	1.1715	0.9171	0.153*
H29C	1.1620	1.1859	1.0250	0.153*
C30	0.9592 (3)	0.8524 (3)	0.9602 (3)	0.0559 (10)
H30A	0.9398	0.8090	0.9087	0.084*

H30B	0.9838	0.8108	1.0115	0.084*
H30C	1.0202	0.8981	0.9426	0.084*
O3	1.0399 (5)	1.3534 (3)	0.8388 (3)	0.1323 (18)
O6	0.9912 (3)	1.1237 (3)	1.1320 (2)	0.0848 (10)
H6	0.9856	1.0832	1.1753	0.127*
O7	0.8893 (3)	0.9497 (2)	1.15122 (18)	0.0684 (8)
O15	0.8667 (3)	0.7449 (3)	1.1382 (2)	0.0868 (10)
H15A	0.8798	0.8044	1.1535	0.130*
O23	0.7255 (6)	0.4771 (4)	0.7596 (4)	0.145 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.094 (3)	0.050 (2)	0.047 (2)	0.004 (2)	0.000 (2)	0.0076 (18)
C2	0.129 (4)	0.051 (2)	0.057 (2)	0.009 (3)	−0.008 (3)	0.011 (2)
C3	0.125 (5)	0.057 (3)	0.072 (3)	−0.012 (3)	0.022 (3)	0.001 (2)
C4	0.069 (3)	0.061 (2)	0.057 (2)	−0.012 (2)	0.010 (2)	−0.002 (2)
C5	0.054 (2)	0.052 (2)	0.0448 (19)	−0.0050 (18)	0.0080 (17)	−0.0054 (17)
C6	0.058 (2)	0.062 (2)	0.0413 (19)	−0.0113 (19)	−0.0026 (18)	−0.0022 (19)
C7	0.050 (2)	0.059 (2)	0.0397 (19)	0.0057 (18)	−0.0008 (17)	0.0058 (17)
C8	0.0473 (18)	0.0443 (17)	0.0366 (17)	0.0033 (16)	0.0003 (16)	0.0054 (15)
C9	0.0503 (18)	0.0411 (17)	0.0367 (17)	0.0062 (16)	−0.0015 (15)	0.0005 (15)
C10	0.064 (2)	0.0459 (18)	0.0356 (17)	0.0067 (18)	0.0051 (18)	0.0027 (16)
C11	0.083 (3)	0.051 (2)	0.0370 (17)	0.004 (2)	−0.012 (2)	0.0045 (17)
C12	0.076 (3)	0.054 (2)	0.062 (2)	−0.004 (2)	−0.031 (2)	0.001 (2)
C13	0.052 (2)	0.053 (2)	0.045 (2)	−0.0036 (17)	−0.0008 (18)	−0.0010 (17)
C14	0.0491 (19)	0.0495 (19)	0.0407 (18)	−0.0018 (17)	−0.0015 (17)	0.0022 (16)
C15	0.065 (2)	0.056 (2)	0.055 (2)	−0.006 (2)	0.003 (2)	0.014 (2)
C16	0.073 (3)	0.048 (2)	0.065 (3)	0.001 (2)	0.005 (2)	0.013 (2)
C17	0.053 (2)	0.049 (2)	0.056 (2)	−0.0058 (17)	0.010 (2)	−0.0003 (18)
C18	0.058 (2)	0.068 (2)	0.060 (2)	−0.005 (2)	0.014 (2)	−0.007 (2)
C19	0.098 (3)	0.060 (2)	0.051 (2)	−0.006 (2)	0.033 (2)	−0.004 (2)
C20	0.079 (3)	0.045 (2)	0.065 (3)	−0.006 (2)	0.020 (2)	−0.002 (2)
C21	0.237 (9)	0.064 (3)	0.083 (4)	0.021 (5)	−0.003 (5)	−0.014 (3)
C22	0.152 (6)	0.089 (4)	0.079 (4)	0.009 (4)	−0.020 (4)	−0.019 (3)
C23	0.191 (7)	0.092 (4)	0.065 (3)	−0.033 (4)	0.014 (4)	−0.042 (3)
C28	0.118 (4)	0.065 (3)	0.090 (4)	−0.024 (3)	0.019 (4)	−0.024 (3)
C29	0.080 (4)	0.091 (4)	0.135 (5)	−0.029 (3)	0.020 (4)	0.002 (4)
C30	0.050 (2)	0.055 (2)	0.063 (2)	0.0057 (19)	0.0064 (19)	0.008 (2)
O3	0.187 (5)	0.092 (3)	0.118 (3)	−0.064 (3)	0.025 (3)	0.035 (3)
O6	0.116 (3)	0.088 (2)	0.0506 (16)	−0.032 (2)	−0.0207 (19)	−0.0003 (16)
O7	0.089 (2)	0.0785 (19)	0.0375 (14)	−0.0127 (17)	−0.0066 (14)	0.0097 (13)
O15	0.106 (2)	0.076 (2)	0.078 (2)	−0.011 (2)	−0.036 (2)	0.0305 (19)
O23	0.229 (6)	0.085 (3)	0.121 (4)	−0.012 (3)	0.024 (4)	−0.022 (3)

Geometric parameters (Å, °)

C1—C2	1.316 (6)	C15—O15	1.419 (5)
C1—C10	1.514 (5)	C15—C16	1.508 (6)

C1—H1	0.9300	C15—H15	0.9800
C2—C3	1.475 (8)	C16—C17	1.500 (6)
C2—H2	0.9300	C16—H16A	0.9700
C3—O3	1.222 (6)	C16—H16B	0.9700
C3—C4	1.522 (7)	C17—C20	1.463 (6)
C4—C28	1.533 (6)	C18—H18A	0.9600
C4—C5	1.528 (6)	C18—H18B	0.9600
C4—C29	1.537 (7)	C18—H18C	0.9600
C5—C6	1.339 (5)	C19—H19A	0.9600
C5—C10	1.524 (5)	C19—H19B	0.9600
C6—O6	1.374 (5)	C19—H19C	0.9600
C6—C7	1.461 (6)	C20—C21	1.334 (8)
C7—O7	1.245 (4)	C20—C22	1.396 (7)
C7—C8	1.503 (5)	C21—O23	1.327 (8)
C8—C30	1.546 (5)	C21—H21	0.9300
C8—C9	1.565 (5)	C22—C23	1.311 (7)
C8—C14	1.583 (5)	C22—H22	0.9300
C9—C11	1.522 (5)	C23—O23	1.399 (9)
C9—C10	1.567 (5)	C23—H23	0.9300
C9—H9	0.9800	C28—H28A	0.9600
C10—C19	1.557 (5)	C28—H28B	0.9600
C11—C12	1.534 (6)	C28—H28C	0.9600
C11—H11A	0.9700	C29—H29A	0.9600
C11—H11B	0.9700	C29—H29B	0.9600
C12—C13	1.477 (5)	C29—H29C	0.9600
C12—H12A	0.9700	C30—H30A	0.9600
C12—H12B	0.9700	C30—H30B	0.9600
C13—C17	1.333 (5)	C30—H30C	0.9600
C13—C14	1.534 (5)	O6—H6	0.8200
C14—C18	1.542 (5)	O15—H15A	0.8200
C14—C15	1.576 (5)		
C2—C1—C10	121.7 (5)	C15—C14—C8	118.6 (3)
C2—C1—H1	119.1	O15—C15—C16	110.7 (4)
C10—C1—H1	119.1	O15—C15—C14	118.8 (3)
C1—C2—C3	119.7 (5)	C16—C15—C14	107.9 (3)
C1—C2—H2	120.2	O15—C15—H15	106.2
C3—C2—H2	120.2	C16—C15—H15	106.2
O3—C3—C2	121.7 (5)	C14—C15—H15	106.2
O3—C3—C4	120.7 (6)	C17—C16—C15	103.5 (3)
C2—C3—C4	117.2 (4)	C17—C16—H16A	111.1
C3—C4—C28	101.9 (4)	C15—C16—H16A	111.1
C3—C4—C5	111.6 (4)	C17—C16—H16B	111.1
C28—C4—C5	113.0 (3)	C15—C16—H16B	111.1
C3—C4—C29	110.1 (4)	H16A—C16—H16B	109.0
C28—C4—C29	110.6 (4)	C13—C17—C20	128.8 (4)
C5—C4—C29	109.5 (4)	C13—C17—C16	111.9 (4)
C6—C5—C10	119.6 (3)	C20—C17—C16	119.2 (3)
C6—C5—C4	122.0 (4)	C14—C18—H18A	109.5
C10—C5—C4	118.4 (3)	C14—C18—H18B	109.5

C5—C6—O6	120.6 (3)	H18A—C18—H18B	109.5
C5—C6—C7	125.7 (3)	C14—C18—H18C	109.5
O6—C6—C7	113.7 (3)	H18A—C18—H18C	109.5
O7—C7—C6	116.5 (4)	H18B—C18—H18C	109.5
O7—C7—C8	122.9 (4)	C10—C19—H19A	109.5
C6—C7—C8	120.5 (3)	C10—C19—H19B	109.5
C7—C8—C30	105.2 (3)	H19A—C19—H19B	109.5
C7—C8—C9	106.8 (3)	C10—C19—H19C	109.5
C30—C8—C9	112.8 (3)	H19A—C19—H19C	109.5
C7—C8—C14	112.9 (3)	H19B—C19—H19C	109.5
C30—C8—C14	110.6 (3)	C21—C20—C22	102.7 (5)
C9—C8—C14	108.5 (3)	C21—C20—C17	126.1 (5)
C11—C9—C8	112.1 (3)	C22—C20—C17	131.1 (4)
C11—C9—C10	112.1 (3)	C20—C21—O23	114.1 (7)
C8—C9—C10	116.1 (3)	C20—C21—H21	122.9
C11—C9—H9	105.1	O23—C21—H21	122.9
C8—C9—H9	105.1	C23—C22—C20	110.7 (6)
C10—C9—H9	105.1	C23—C22—H22	124.6
C5—C10—C1	107.3 (3)	C20—C22—H22	124.7
C5—C10—C19	109.3 (3)	C22—C23—O23	107.8 (6)
C1—C10—C19	106.3 (3)	C22—C23—H23	126.1
C5—C10—C9	112.2 (3)	O23—C23—H23	126.1
C1—C10—C9	107.6 (3)	C4—C28—H28A	109.5
C19—C10—C9	113.9 (3)	C4—C28—H28B	109.5
C9—C11—C12	111.5 (3)	H28A—C28—H28B	109.5
C9—C11—H11A	109.3	C4—C28—H28C	109.5
C12—C11—H11A	109.3	H28A—C28—H28C	109.5
C9—C11—H11B	109.3	H28B—C28—H28C	109.5
C12—C11—H11B	109.3	C4—C29—H29A	109.5
H11A—C11—H11B	108.0	C4—C29—H29B	109.5
C13—C12—C11	108.3 (3)	H29A—C29—H29B	109.5
C13—C12—H12A	110.0	C4—C29—H29C	109.5
C11—C12—H12A	110.0	H29A—C29—H29C	109.5
C13—C12—H12B	110.0	H29B—C29—H29C	109.5
C11—C12—H12B	110.0	C8—C30—H30A	109.5
H12A—C12—H12B	108.4	C8—C30—H30B	109.5
C17—C13—C12	129.5 (4)	H30A—C30—H30B	109.5
C17—C13—C14	113.1 (4)	C8—C30—H30C	109.5
C12—C13—C14	116.7 (3)	H30A—C30—H30C	109.5
C13—C14—C18	111.7 (3)	H30B—C30—H30C	109.5
C13—C14—C15	99.7 (3)	C6—O6—H6	109.5
C18—C14—C15	108.6 (3)	C15—O15—H15A	109.5
C13—C14—C8	107.0 (3)	C21—O23—C23	104.4 (5)
C18—C14—C8	110.8 (3)		
C10—C1—C2—C3	−2.1 (7)	C11—C9—C10—C19	−49.2 (4)
C1—C2—C3—O3	152.4 (5)	C8—C9—C10—C19	81.3 (4)
C1—C2—C3—C4	−34.0 (7)	C8—C9—C11—C12	57.4 (4)
O3—C3—C4—C28	77.8 (6)	C10—C9—C11—C12	−170.0 (3)
C2—C3—C4—C28	−95.9 (5)	C9—C11—C12—C13	−54.8 (4)

O3—C3—C4—C5	−161.4 (5)	C11—C12—C13—C17	−110.9 (5)
C2—C3—C4—C5	24.9 (6)	C11—C12—C13—C14	58.4 (5)
O3—C3—C4—C29	−39.6 (7)	C17—C13—C14—C18	−126.8 (4)
C2—C3—C4—C29	146.7 (5)	C12—C13—C14—C18	62.1 (4)
C3—C4—C5—C6	−164.1 (4)	C17—C13—C14—C15	−12.2 (4)
C28—C4—C5—C6	−50.0 (6)	C12—C13—C14—C15	176.7 (3)
C29—C4—C5—C6	73.7 (5)	C17—C13—C14—C8	111.8 (4)
C3—C4—C5—C10	16.3 (5)	C12—C13—C14—C8	−59.2 (4)
C28—C4—C5—C10	130.4 (4)	C7—C8—C14—C13	172.8 (3)
C29—C4—C5—C10	−105.9 (5)	C30—C8—C14—C13	−69.6 (4)
C10—C5—C6—O6	−179.8 (4)	C9—C8—C14—C13	54.6 (4)
C4—C5—C6—O6	0.6 (6)	C7—C8—C14—C18	50.8 (4)
C10—C5—C6—C7	2.4 (6)	C30—C8—C14—C18	168.4 (3)
C4—C5—C6—C7	−177.2 (4)	C9—C8—C14—C18	−67.3 (4)
C5—C6—C7—O7	−177.1 (4)	C7—C8—C14—C15	−75.7 (4)
O6—C6—C7—O7	4.9 (5)	C30—C8—C14—C15	41.9 (4)
C5—C6—C7—C8	7.4 (6)	C9—C8—C14—C15	166.1 (3)
O6—C6—C7—C8	−170.6 (4)	C13—C14—C15—O15	146.0 (4)
O7—C7—C8—C30	−87.2 (4)	C18—C14—C15—O15	−97.0 (4)
C6—C7—C8—C30	88.0 (4)	C8—C14—C15—O15	30.6 (5)
O7—C7—C8—C9	152.7 (4)	C13—C14—C15—C16	19.1 (4)
C6—C7—C8—C9	−32.1 (4)	C18—C14—C15—C16	136.1 (3)
O7—C7—C8—C14	33.5 (5)	C8—C14—C15—C16	−96.3 (4)
C6—C7—C8—C14	−151.3 (3)	O15—C15—C16—C17	−150.9 (3)
C7—C8—C9—C11	−179.3 (3)	C14—C15—C16—C17	−19.4 (4)
C30—C8—C9—C11	65.6 (4)	C12—C13—C17—C20	−6.3 (7)
C14—C8—C9—C11	−57.3 (4)	C14—C13—C17—C20	−175.9 (4)
C7—C8—C9—C10	50.1 (4)	C12—C13—C17—C16	170.2 (4)
C30—C8—C9—C10	−65.0 (4)	C14—C13—C17—C16	0.5 (5)
C14—C8—C9—C10	172.1 (3)	C15—C16—C17—C13	12.1 (5)
C6—C5—C10—C1	133.3 (4)	C15—C16—C17—C20	−171.1 (3)
C4—C5—C10—C1	−47.0 (5)	C13—C17—C20—C21	164.9 (6)
C6—C5—C10—C19	−111.8 (4)	C16—C17—C20—C21	−11.3 (8)
C4—C5—C10—C19	67.8 (4)	C13—C17—C20—C22	−19.7 (8)
C6—C5—C10—C9	15.4 (5)	C16—C17—C20—C22	164.1 (5)
C4—C5—C10—C9	−164.9 (3)	C22—C20—C21—O23	4.7 (8)
C2—C1—C10—C5	41.0 (5)	C17—C20—C21—O23	−178.8 (5)
C2—C1—C10—C19	−75.9 (5)	C21—C20—C22—C23	−2.0 (8)
C2—C1—C10—C9	161.8 (4)	C17—C20—C22—C23	−178.2 (5)
C11—C9—C10—C5	−174.0 (3)	C20—C22—C23—O23	−1.1 (8)
C8—C9—C10—C5	−43.4 (4)	C20—C21—O23—C23	−5.5 (9)
C11—C9—C10—C1	68.3 (4)	C22—C23—O23—C21	3.8 (8)
C8—C9—C10—C1	−161.1 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O7	0.82	2.10	2.573 (4)	117
O15—H15A···O7	0.82	1.90	2.687 (4)	162

C11—H11A···O3^{i}	0.97	2.55	3.431 (6)	151
C11—H11B···O7^{ii}	0.97	2.56	3.458 (5)	155

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

Fig. 1

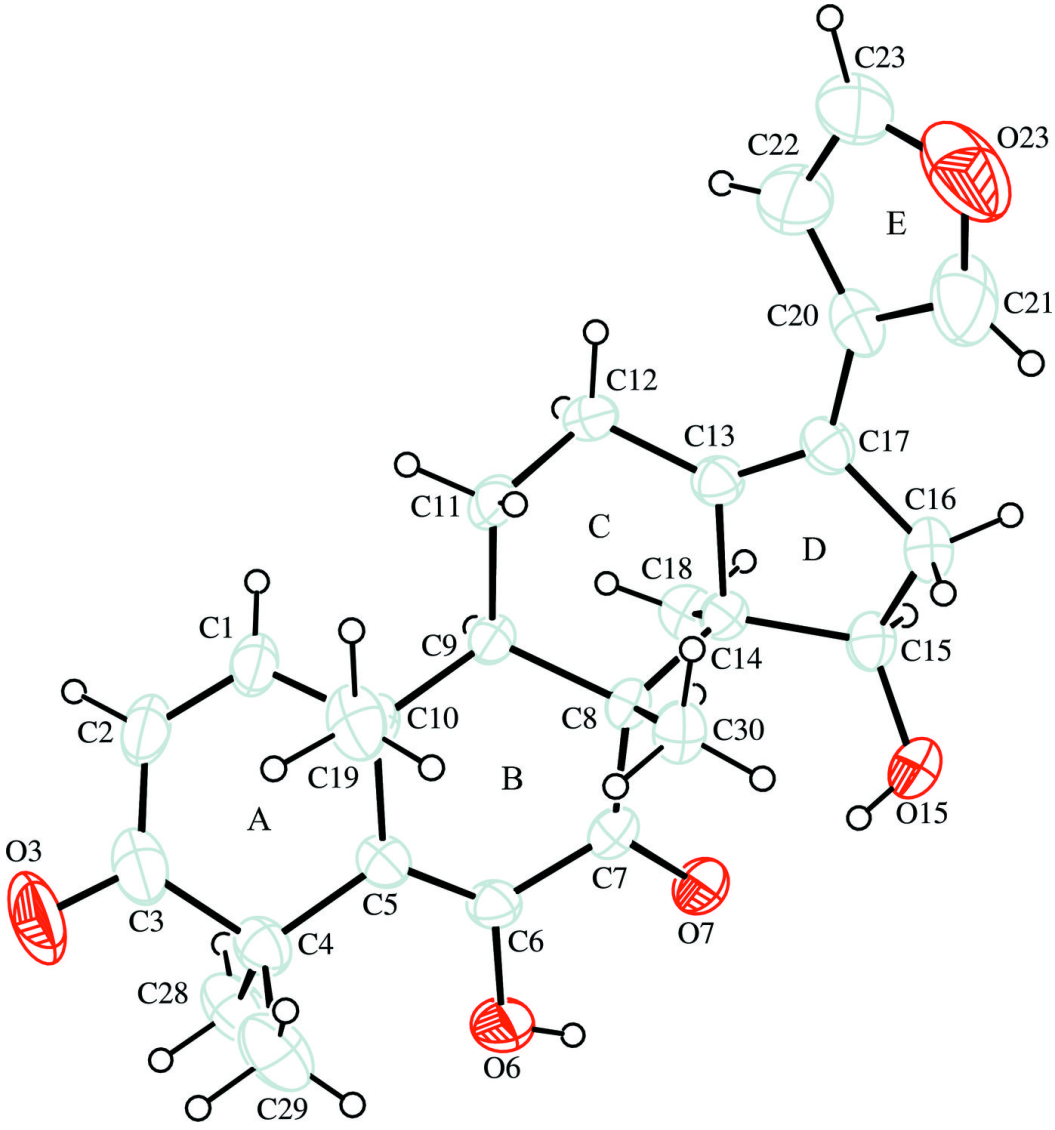
C23
O23
C22
E
C20
C21
C12
C13
C17
C11
C16
C
D
C18
C1
C9
C14
C15
C2
C10
C8
C30
C19
B
O15
A
O3
C7
C5
C3
C4
C6
O7
C28
O6
C29

Fig. 2