Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,2-Dihydrocedrelone

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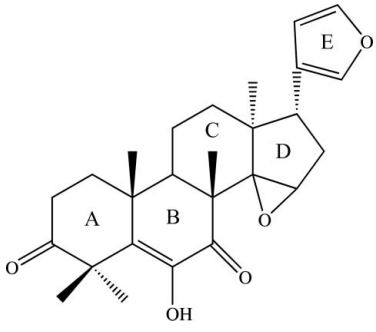
Received 22 August 2007; accepted 30 August 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.062; wR factor = 0.189; data-to-parameter ratio = 7.6.

The title compound (systematic name: 7-furan-3-yl-6-hydroxy-4,4,8,10,13-pentamethyl-1,8,9,10,11,12,13,15,16,17-decahydro-2*H*,4*H*-20-oxacyclopropa[14,15]cyclopenta[*a*]phenanthrene-3,7-dione), $C_{26}H_{32}O_5$, is a semi-synthetic derivative of cedrelone, a natural compound isolated from *Toona ciliata*. The orientation of the furan ring and the ring conformations are the same as for cedrelone itself, with the exception of ring *A*. Rings *A*, *B*, *C* and *D* adopt sofa, half-chair, twist and envelope conformations, respectively. The crystal structure involves intramolecular O—H···O and intermolecular C—H···O and O—H···O hydrogen bonds. All the hydrogen bonds in the crystal structure contribute to the formation of a macrocyclic ring motif $R_4^4(28)$.

Related literature

Many cedrelone derivatives have been reported (Hodges *et al.*, 1963). The three-dimensional structure of cedrelone itself has previously been reported (Zeumer *et al.*, 2000). For related literature, see: Bernstein *et al.* (1995); Cremer & Pople (1975); Narayanan *et al.* (1980).



Experimental

Crystal data

$C_{26}H_{32}O_5$
M_r = 424.52
Monoclinic, $P2_1$
a = 8.302 (4) Å
b = 10.150 (2) Å
c = 13.560 (5) Å
β = 95.330 (3)°
V = 1137.7 (7) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 0.68 mm^{-1}
T = 293 (2) K
0.30 × 0.23 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2591 measured reflections
2161 independent reflections
1974 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
3 standard reflections
every 200 reflections
intensity decay: 3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.189
S = 0.80
2161 reflections
286 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O3^{i}	0.82	2.41	3.139 (4)	149
C12—H12A···O15^{ii}	0.97	2.58	3.499 (5)	159
C2—H2A···O7^{iii}	0.97	2.42	3.251 (6)	144
O6—H6···O7	0.82	2.18	2.646 (4)	116

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

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).

SSD thanks the University of Madras for a University Research Fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2196).

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 doi:10.1107/S1600536807042535

supplementary materials

Acta Cryst. (2007). E**63**, o3963 [**doi:10.1107/S1600536807042535**]

1,2-Dihydrocedrelone

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Comment

A tetranortriterpenoid, cedrelone, has been previously isolated from Toona ciliata and its three dimensional structure was reported earlier (Zeumer et al., 2000). It possesses half the antifeedant activity of the most potent azadirachtin-A from Azadirachta indica. The title compound (Fig. 1), a semisynthetic derivative of cedrelone, differs from the parent molecule simply by having single bond C1—C2 rather than C1═C2. Except for ring A, the ring conformations in the title compound are the same as in cedrelone (Zeumer et al., 2000). The orientation of the furan ring is defined by the torsion angle C16—C17—C20—C22 = 179.8 (4)°; the corresponding value in cedrelone is 168.3 (4)°.

Ring A exists in a sofa conformation (Cremer & Pople, 1975) [Q_T = 0.688 (4) Å, φ_2 = −48 (3)°, q_2 = 0.677 (4) Å], and a mirror plane passes through atoms C3 and C10; in cedrelone itself, ring A adopts a distorted half-chair conformation. Ring B [Q_T =0.522 (4) Å, φ_2=32.3 (5)°,q_2 =0.435 (3) Å] adopts a half-chair conformation. Ring C adopts a twist conformation [Q_T =0.760 (3) Å, φ_2 =88.1 (3) °, q_2 =0.757 (3) Å]. The atoms C8 and C9 deviate from the mean plane of the other four atoms by 0.385 (3) Å and −0.407 (3) Å respectively. Ring D is in an envelope conformation [φ_2 = −145.1 (3)°,q_2 = 0.204 (4) Å] and ring E is planar (Nardelli, 1995). Ring pairs A/B, B/C and C/D are in quasi-trans fusion as evident from the endocyclic torsion angles of ring junction atoms. Thus, for rings A/B C4—C5—C10—C1 = −51.8 (4)° and C6—C5—C10—C9 = 9.8 (5)\5; for rings B/C C7—C8—C9—C10 = 63.5 (4)° and C14—C8—C9—C11 = −37.9 (4)°; for rings C/D C12—C13—C14—C8 = 58.5 (4)° and C17—C13—C14—C15 = −19.0 (4)°.

The packing of the molecules (Fig. 2) reveals that the crystal structure is stabilized by a network of hydrogen bonds (Table 1). The hydrogen bond C12—H12A···O15(−x + 2,y + 1/2,-z) forms an infinite chain, graph set motif C(9). A ring motif S(5) is formed by the hydrogen bond O6—H6···O7. In addition, $R_2^2(9)$ is generated through C2—H2A···O7(1 + x,y,z) and O6—H6···O3(−1 + x,y,z). All the observed hydrogen bonds in the crystal structure together form a macrocyclic ring motif $R_4^4(28)$.

Experimental

Acetic acid (5 ml) was placed in a 25 ml Erylenmeyer flask fitted with microwave and reflux condenser with guard tube. To this cedrelone (141 mg, 0.33 mmol) and activated zinc was added. The mixture was then subjected to microwave radiation. After completion of the reaction, the solution was passed through a celite bed, followed by acetic acid and was removed by vacuum distillation. The title compound was purified by short flash (under nitrogen) column chromatography.

Refinement

In the absence of significant anomalous scattering effects, 2161 Friedel equivalents were merged. The enantiomer employed in the refined model was chosen to agree with the accepted configuration of tetranortriterpenoids (Narayanan *et al.*, 1980).

Hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93, 0.96, 0.97 and 0.98 Å for Csp^2, methyl, methylene and methine, respectively; O—H = 0.82 Å. These C—H and O—H bonds were allowed to rotate freely about the C—C and C—O bonds. U_{iso}(H) = xU_{eq}(carrier atom) where x = 1.5 for methyl and O, 1.2 for all other H atoms.

Figures

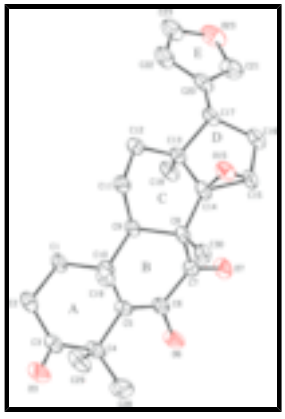

Fig. 1. Molecular structure of the title compound, with 30% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen atoms have been omitted.

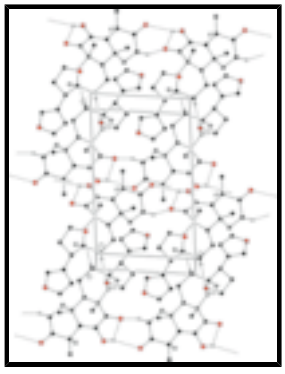

Fig. 2. A view of the packing in the crystal structure, showing the $R_4^4(28)$ ring. Dashed lines indicate hydrogen bonding. H atoms not involved in hydrogen bonding have been omitted.

7-furan-3-yl-6-hydroxy-4,4,8,10,13-pentamethyl-1,8,9,10,11,12,13,15,16,17- decahydro-2H,4*H*-20-oxacyclopropa[14,15]cyclopenta[*a*]phenanthrene- 3,7-dione

Crystal data

$C_{26}H_{32}O_5$	F_{000} = 456
M_r = 424.52	D_x = 1.239 Mg m^{-3}
Monoclinic, $P2_1$	Cu $K\alpha$ radiation λ = 1.54184 Å
a = 8.302 (4) Å	Cell parameters from 25 reflections
b = 10.150 (2) Å	θ = 15–30º
c = 13.560 (5) Å	μ = 0.68 mm^{-1}
β = 95.330 (3)º	T = 293 (2) K
V = 1137.7 (7) Å^3	Rod, colourless
Z = 2	0.30 × 0.23 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.087
Radiation source: fine-focus sealed tube	θ_{max} = 67.5º
Monochromator: graphite	θ_{min} = 3.3º
T = 293(2) K	h = 0→9
non–profiled ω/2θ scans	k = 0→12
Absorption correction: none	l = −16→16
2591 measured reflections	3 standard reflections
2161 independent reflections	every 200 reflections
1974 reflections with $I > 2\sigma(I)$	intensity decay: 3%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.062	$w = 1/[\sigma^2(F_o^2) + (0.199P)^2 + 0.4014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.189	$(\Delta/\sigma)_{max} < 0.001$
S = 0.80	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
2161 reflections	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
286 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 $F^2 > 2\text{sigma}(F^2)$ 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	1.5868 (3)	0.4276 (5)	0.4887 (2)	0.0712 (11)
O6	0.9552 (3)	0.3939 (4)	0.4596 (2)	0.0630 (9)
H6	0.8586	0.3758	0.4535	0.095*

O7	0.7759 (3)	0.2519 (4)	0.3276 (2)	0.0597 (8)
O15	0.9023 (4)	−0.0169 (3)	0.1327 (2)	0.0562 (8)
O23	0.4733 (4)	0.2782 (4)	−0.2042 (3)	0.0756 (10)
C1	1.3541 (5)	0.4167 (4)	0.2475 (3)	0.0504 (9)
H1A	1.2753	0.4857	0.2312	0.060*
H1B	1.3997	0.3917	0.1868	0.060*
C2	1.4901 (5)	0.4715 (5)	0.3212 (3)	0.0552 (10)
H2A	1.5929	0.4370	0.3043	0.066*
H2B	1.4933	0.5666	0.3145	0.066*
C3	1.4699 (5)	0.4379 (5)	0.4265 (3)	0.0543 (10)
C4	1.2979 (4)	0.4246 (5)	0.4560 (3)	0.0538 (10)
C5	1.1889 (4)	0.3489 (4)	0.3773 (2)	0.0426 (8)
C6	1.0297 (4)	0.3354 (4)	0.3850 (3)	0.0471 (9)
C7	0.9206 (4)	0.2529 (4)	0.3168 (2)	0.0444 (8)
C8	0.9996 (4)	0.1674 (4)	0.2466 (3)	0.0414 (8)
C9	1.1337 (4)	0.2540 (4)	0.2070 (2)	0.0402 (8)
H9	1.0782	0.3363	0.1871	0.048*
C10	1.2675 (4)	0.2968 (4)	0.2875 (2)	0.0392 (7)
C11	1.1805 (4)	0.1959 (5)	0.1099 (2)	0.0467 (8)
H11A	1.2008	0.1022	0.1182	0.056*
H11B	1.2792	0.2372	0.0923	0.056*
C12	1.0432 (4)	0.2181 (5)	0.0258 (3)	0.0505 (9)
H12A	1.0549	0.3052	−0.0022	0.061*
H12B	1.0538	0.1540	−0.0262	0.061*
C13	0.8738 (4)	0.2059 (4)	0.0624 (3)	0.0444 (8)
C14	0.8874 (4)	0.1230 (4)	0.1566 (3)	0.0439 (8)
C15	0.7478 (5)	0.0347 (5)	0.1551 (3)	0.0548 (10)
H15	0.7027	0.0105	0.2169	0.066*
C16	0.6376 (5)	0.0597 (5)	0.0623 (3)	0.0610 (11)
H16A	0.5500	0.1187	0.0752	0.073*
H16B	0.5924	−0.0219	0.0348	0.073*
C17	0.7501 (4)	0.1237 (4)	−0.0076 (3)	0.0493 (9)
H17	0.8114	0.0522	−0.0351	0.059*
C18	0.7977 (5)	0.3403 (4)	0.0818 (3)	0.0520 (9)
H18A	0.7887	0.3912	0.0218	0.078*
H18B	0.8646	0.3864	0.1320	0.078*
H18C	0.6921	0.3274	0.1036	0.078*
C19	1.3932 (4)	0.1871 (4)	0.3153 (3)	0.0486 (9)
H19A	1.3399	0.1124	0.3410	0.073*
H19B	1.4739	0.2196	0.3646	0.073*
H19C	1.4438	0.1613	0.2574	0.073*
C20	0.6656 (5)	0.1966 (5)	−0.0939 (3)	0.0541 (9)
C21	0.5047 (5)	0.2089 (7)	−0.1179 (3)	0.0687 (13)
H21	0.4257	0.1748	−0.0808	0.082*
C22	0.7396 (6)	0.2648 (5)	−0.1704 (4)	0.0666 (12)
H22	0.8499	0.2757	−0.1747	0.080*
C23	0.6198 (7)	0.3102 (5)	−0.2348 (4)	0.0716 (14)
H23	0.6348	0.3569	−0.2923	0.086*
C28	1.2986 (5)	0.3573 (8)	0.5571 (3)	0.0775 (16)

H28A	1.3600	0.4095	0.6063	0.116*
H28B	1.3467	0.2716	0.5541	0.116*
H28C	1.1895	0.3487	0.5743	0.116*
C29	1.2402 (6)	0.5703 (6)	0.4622 (5)	0.0755 (15)
H29A	1.2370	0.6102	0.3979	0.113*
H29B	1.3140	0.6181	0.5077	0.113*
H29C	1.1341	0.5723	0.4849	0.113*
C30	1.0586 (5)	0.0445 (4)	0.3070 (3)	0.0511 (9)
H30A	1.1104	−0.0153	0.2651	0.077*
H30B	0.9678	0.0016	0.3322	0.077*
H30C	1.1342	0.0709	0.3613	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0370 (14)	0.101 (3)	0.0708 (18)	0.0006 (17)	−0.0225 (13)	−0.0111 (19)
O6	0.0343 (13)	0.091 (2)	0.0628 (16)	−0.0005 (15)	−0.0003 (11)	−0.0290 (16)
O7	0.0232 (12)	0.082 (2)	0.0742 (17)	−0.0001 (14)	0.0049 (10)	−0.0155 (16)
O15	0.0564 (17)	0.0424 (14)	0.0668 (17)	−0.0008 (13)	−0.0096 (13)	−0.0048 (12)
O23	0.0601 (19)	0.075 (2)	0.085 (2)	0.0165 (18)	−0.0323 (16)	−0.0062 (19)
C1	0.0315 (17)	0.058 (2)	0.060 (2)	−0.0098 (17)	−0.0086 (15)	0.0033 (18)
C2	0.0330 (18)	0.058 (2)	0.071 (2)	−0.0062 (18)	−0.0121 (15)	−0.002 (2)
C3	0.0367 (19)	0.062 (2)	0.061 (2)	−0.0051 (18)	−0.0128 (15)	−0.0082 (19)
C4	0.0306 (18)	0.072 (3)	0.055 (2)	0.0025 (19)	−0.0158 (14)	−0.0133 (19)
C5	0.0311 (16)	0.0515 (19)	0.0429 (16)	0.0036 (16)	−0.0091 (12)	−0.0012 (15)
C6	0.0336 (16)	0.061 (2)	0.0448 (16)	−0.0009 (17)	−0.0049 (12)	−0.0070 (16)
C7	0.0284 (16)	0.056 (2)	0.0479 (17)	0.0004 (16)	−0.0008 (12)	0.0012 (16)
C8	0.0255 (14)	0.0461 (19)	0.0511 (17)	−0.0025 (15)	−0.0043 (12)	−0.0021 (15)
C9	0.0243 (15)	0.0468 (18)	0.0481 (16)	−0.0006 (15)	−0.0044 (12)	0.0013 (15)
C10	0.0260 (14)	0.0438 (17)	0.0461 (16)	0.0030 (14)	−0.0062 (12)	0.0007 (14)
C11	0.0301 (15)	0.061 (2)	0.0483 (17)	−0.0009 (17)	−0.0027 (13)	−0.0041 (17)
C12	0.0378 (18)	0.064 (2)	0.0475 (18)	−0.0063 (18)	−0.0073 (13)	0.0010 (17)
C13	0.0336 (17)	0.0467 (18)	0.0504 (17)	−0.0047 (15)	−0.0093 (13)	−0.0034 (16)
C14	0.0288 (16)	0.0430 (19)	0.058 (2)	−0.0015 (15)	−0.0051 (13)	−0.0040 (16)
C15	0.0432 (19)	0.059 (2)	0.060 (2)	−0.021 (2)	−0.0044 (15)	−0.0022 (19)
C16	0.046 (2)	0.065 (3)	0.069 (2)	−0.014 (2)	−0.0117 (17)	−0.009 (2)
C17	0.0391 (19)	0.051 (2)	0.0547 (19)	−0.0077 (17)	−0.0134 (14)	−0.0054 (17)
C18	0.0413 (19)	0.0432 (19)	0.067 (2)	−0.0006 (17)	−0.0164 (15)	−0.0061 (18)
C19	0.0234 (15)	0.056 (2)	0.064 (2)	0.0078 (16)	−0.0068 (13)	0.0006 (18)
C20	0.0399 (19)	0.053 (2)	0.065 (2)	0.0002 (18)	−0.0192 (16)	−0.0077 (19)
C21	0.042 (2)	0.091 (4)	0.069 (2)	0.010 (2)	−0.0145 (17)	−0.009 (3)
C22	0.056 (2)	0.062 (3)	0.076 (3)	−0.009 (2)	−0.025 (2)	0.013 (2)
C23	0.074 (3)	0.054 (2)	0.079 (3)	−0.007 (2)	−0.035 (2)	0.012 (2)
C28	0.047 (2)	0.126 (5)	0.056 (2)	0.001 (3)	−0.0131 (17)	−0.003 (3)
C29	0.049 (2)	0.075 (3)	0.099 (3)	−0.002 (2)	−0.008 (2)	−0.036 (3)
C30	0.0405 (18)	0.050 (2)	0.061 (2)	−0.0072 (18)	−0.0040 (15)	0.0051 (18)

Geometric parameters (Å, °)

O3—C3	1.230 (5)	C12—H12A	0.9700
O6—C6	1.369 (5)	C12—H12B	0.9700
O6—H6	0.8200	C13—C14	1.525 (5)
O7—C7	1.223 (4)	C13—C18	1.537 (6)
O15—C15	1.444 (6)	C13—C17	1.572 (5)
O15—C14	1.465 (5)	C14—C15	1.464 (5)
O23—C23	1.360 (7)	C15—C16	1.507 (6)
O23—C21	1.370 (6)	C15—H15	0.9800
C1—C10	1.538 (5)	C16—C17	1.535 (6)
C1—C2	1.541 (5)	C16—H16A	0.9700
C1—H1A	0.9700	C16—H16B	0.9700
C1—H1B	0.9700	C17—C20	1.502 (5)
C2—C3	1.491 (6)	C17—H17	0.9800
C2—H2A	0.9700	C18—H18A	0.9600
C2—H2B	0.9700	C18—H18B	0.9600
C3—C4	1.524 (6)	C18—H18C	0.9600
C4—C28	1.531 (7)	C19—H19A	0.9600
C4—C5	1.538 (5)	C19—H19B	0.9600
C4—C29	1.559 (8)	C19—H19C	0.9600
C5—C6	1.343 (5)	C20—C21	1.351 (6)
C5—C10	1.529 (5)	C20—C22	1.433 (7)
C6—C7	1.491 (5)	C21—H21	0.9300
C7—O7	1.223 (4)	C22—C23	1.342 (6)
C7—C8	1.486 (5)	C22—H22	0.9300
C8—C14	1.532 (5)	C23—H23	0.9300
C8—C30	1.548 (5)	C28—H28A	0.9600
C8—C9	1.553 (5)	C28—H28B	0.9600
C9—C11	1.525 (5)	C28—H28C	0.9600
C9—C10	1.546 (4)	C29—H29A	0.9600
C9—H9	0.9800	C29—H29B	0.9600
C10—C19	1.548 (5)	C29—H29C	0.9600
C11—C12	1.552 (5)	C30—H30A	0.9600
C11—H11A	0.9700	C30—H30B	0.9600
C11—H11B	0.9700	C30—H30C	0.9600
C12—C13	1.539 (5)		
C6—O6—H6	109.5	C12—C13—C17	114.4 (3)
C15—O15—C14	60.4 (3)	C15—C14—O15	59.1 (3)
C23—O23—C21	106.2 (3)	C15—C14—C13	109.2 (3)
C10—C1—C2	113.3 (3)	O15—C14—C13	110.5 (3)
C10—C1—H1A	108.9	C15—C14—C8	127.9 (3)
C2—C1—H1A	108.9	O15—C14—C8	113.8 (3)
C10—C1—H1B	108.9	C13—C14—C8	119.8 (3)
C2—C1—H1B	108.9	O15—C15—C14	60.5 (2)
H1A—C1—H1B	107.7	O15—C15—C16	111.6 (3)
C3—C2—C1	113.4 (3)	C14—C15—C16	109.1 (4)
C3—C2—H2A	108.9	O15—C15—H15	120.4

C1—C2—H2A	108.9	C14—C15—H15	120.4
C3—C2—H2B	108.9	C16—C15—H15	120.4
C1—C2—H2B	108.9	C15—C16—C17	103.3 (3)
H2A—C2—H2B	107.7	C15—C16—H16A	111.1
O3—C3—C2	121.6 (4)	C17—C16—H16A	111.1
O3—C3—C4	120.7 (4)	C15—C16—H16B	111.1
C2—C3—C4	117.6 (3)	C17—C16—H16B	111.1
C3—C4—C28	110.4 (3)	H16A—C16—H16B	109.1
C3—C4—C5	111.5 (3)	C20—C17—C16	115.0 (3)
C28—C4—C5	110.5 (4)	C20—C17—C13	116.2 (3)
C3—C4—C29	103.3 (4)	C16—C17—C13	104.6 (3)
C28—C4—C29	110.5 (5)	C20—C17—H17	106.8
C5—C4—C29	110.3 (3)	C16—C17—H17	106.8
C6—C5—C10	121.5 (3)	C13—C17—H17	106.8
C6—C5—C4	121.1 (3)	C13—C18—H18A	109.5
C10—C5—C4	117.3 (3)	C13—C18—H18B	109.5
C5—C6—O6	121.9 (3)	H18A—C18—H18B	109.5
C5—C6—C7	123.7 (3)	C13—C18—H18C	109.5
O6—C6—C7	114.3 (3)	H18A—C18—H18C	109.5
O7—C7—C8	124.6 (3)	H18B—C18—H18C	109.5
O7—C7—C6	118.5 (3)	C10—C19—H19A	109.5
C8—C7—C6	116.6 (3)	C10—C19—H19B	109.5
C7—C8—C14	114.2 (3)	H19A—C19—H19B	109.5
C7—C8—C30	105.5 (3)	C10—C19—H19C	109.5
C14—C8—C30	108.9 (3)	H19A—C19—H19C	109.5
C7—C8—C9	105.3 (3)	H19B—C19—H19C	109.5
C14—C8—C9	107.1 (3)	C21—C20—C22	105.1 (4)
C30—C8—C9	115.9 (3)	C21—C20—C17	127.8 (4)
C11—C9—C10	119.6 (3)	C22—C20—C17	127.0 (4)
C11—C9—C8	109.1 (3)	C20—C21—O23	111.0 (5)
C10—C9—C8	114.0 (3)	C20—C21—H21	124.5
C11—C9—H9	104.1	O23—C21—H21	124.5
C10—C9—H9	104.1	C23—C22—C20	107.2 (4)
C8—C9—H9	104.1	C23—C22—H22	126.4
C5—C10—C1	104.8 (3)	C20—C22—H22	126.4
C5—C10—C9	109.1 (2)	C22—C23—O23	110.4 (5)
C1—C10—C9	107.6 (3)	C22—C23—H23	124.8
C5—C10—C19	112.5 (3)	O23—C23—H23	124.8
C1—C10—C19	109.2 (3)	C4—C28—H28A	109.5
C9—C10—C19	113.2 (3)	C4—C28—H28B	109.5
C9—C11—C12	110.5 (3)	H28A—C28—H28B	109.5
C9—C11—H11A	109.6	C4—C28—H28C	109.5
C12—C11—H11A	109.6	H28A—C28—H28C	109.5
C9—C11—H11B	109.6	H28B—C28—H28C	109.5
C12—C11—H11B	109.6	C4—C29—H29A	109.5
H11A—C11—H11B	108.1	C4—C29—H29B	109.5
C13—C12—C11	112.4 (3)	H29A—C29—H29B	109.5
C13—C12—H12A	109.1	C4—C29—H29C	109.5
C11—C12—H12A	109.1	H29A—C29—H29C	109.5

C13—C12—H12B	109.1	H29B—C29—H29C	109.5
C11—C12—H12B	109.1	C8—C30—H30A	109.5
H12A—C12—H12B	107.8	C8—C30—H30B	109.5
C14—C13—C18	110.3 (3)	H30A—C30—H30B	109.5
C14—C13—C12	108.4 (3)	C8—C30—H30C	109.5
C18—C13—C12	112.7 (3)	H30A—C30—H30C	109.5
C14—C13—C17	101.9 (3)	H30B—C30—H30C	109.5
C18—C13—C17	108.6 (3)		
C10—C1—C2—C3	−23.4 (5)	C9—C11—C12—C13	−36.7 (5)
C1—C2—C3—O3	152.0 (5)	C11—C12—C13—C14	−22.8 (5)
C1—C2—C3—C4	−31.6 (6)	C11—C12—C13—C18	99.6 (4)
O3—C3—C4—C28	−17.6 (7)	C11—C12—C13—C17	−135.7 (4)
C2—C3—C4—C28	165.9 (5)	C15—O15—C14—C13	−100.7 (3)
O3—C3—C4—C5	−141.0 (5)	C15—O15—C14—C8	121.2 (4)
C2—C3—C4—C5	42.5 (6)	C18—C13—C14—C15	96.1 (4)
O3—C3—C4—C29	100.5 (5)	C12—C13—C14—C15	−140.0 (3)
C2—C3—C4—C29	−76.0 (5)	C17—C13—C14—C15	−19.0 (4)
C3—C4—C5—C6	−174.6 (4)	C18—C13—C14—O15	159.3 (3)
C28—C4—C5—C6	62.1 (5)	C12—C13—C14—O15	−76.8 (4)
C29—C4—C5—C6	−60.4 (6)	C17—C13—C14—O15	44.2 (4)
C3—C4—C5—C10	2.0 (5)	C18—C13—C14—C8	−65.3 (4)
C28—C4—C5—C10	−121.3 (4)	C12—C13—C14—C8	58.5 (4)
C29—C4—C5—C10	116.2 (4)	C17—C13—C14—C8	179.5 (3)
C10—C5—C6—O6	−172.9 (4)	C7—C8—C14—C15	−67.0 (5)
C4—C5—C6—O6	3.6 (6)	C30—C8—C14—C15	50.7 (5)
C10—C5—C6—C7	9.5 (6)	C9—C8—C14—C15	176.8 (4)
C4—C5—C6—C7	−174.0 (4)	C7—C8—C14—O15	−135.4 (4)
C5—C6—C7—O7	−176.8 (4)	C30—C8—C14—O15	−17.7 (4)
O6—C6—C7—O7	5.5 (6)	C9—C8—C14—O15	108.4 (4)
C5—C6—C7—C8	9.0 (6)	C7—C8—C14—C13	90.6 (4)
O6—C6—C7—C8	−168.8 (4)	C30—C8—C14—C13	−151.7 (3)
O7—C7—C8—C14	26.4 (5)	C9—C8—C14—C13	−25.6 (4)
C6—C7—C8—C14	−159.7 (3)	C14—O15—C15—C16	100.4 (4)
O7—C7—C8—C30	−93.2 (4)	C13—C14—C15—O15	103.0 (3)
C6—C7—C8—C30	80.7 (4)	C8—C14—C15—O15	−97.5 (4)
O7—C7—C8—C9	143.6 (4)	O15—C14—C15—C16	−104.6 (4)
C6—C7—C8—C9	−42.5 (4)	C13—C14—C15—C16	−1.6 (5)
C7—C8—C9—C11	−159.9 (3)	C8—C14—C15—C16	157.9 (4)
C14—C8—C9—C11	−37.9 (4)	O15—C15—C16—C17	−42.8 (5)
C30—C8—C9—C11	83.8 (4)	C14—C15—C16—C17	22.2 (5)
C7—C8—C9—C10	63.5 (4)	C15—C16—C17—C20	−162.2 (4)
C14—C8—C9—C10	−174.6 (3)	C15—C16—C17—C13	−33.5 (5)
C30—C8—C9—C10	−52.8 (4)	C14—C13—C17—C20	160.1 (3)
C6—C5—C10—C1	124.8 (4)	C18—C13—C17—C20	43.8 (4)
C4—C5—C10—C1	−51.8 (4)	C12—C13—C17—C20	−83.1 (5)
C6—C5—C10—C9	9.8 (5)	C14—C13—C17—C16	32.2 (4)
C4—C5—C10—C9	−166.8 (3)	C18—C13—C17—C16	−84.1 (4)
C6—C5—C10—C19	−116.7 (4)	C12—C13—C17—C16	149.0 (4)
C4—C5—C10—C19	66.7 (4)	C16—C17—C20—C21	−1.9 (7)

C2—C1—C10—C5	63.1 (4)	C13—C17—C20—C21	−124.5 (5)
C2—C1—C10—C9	179.2 (3)	C16—C17—C20—C22	−179.8 (4)
C2—C1—C10—C19	−57.6 (4)	C13—C17—C20—C22	57.6 (6)
C11—C9—C10—C5	−179.0 (3)	C22—C20—C21—O23	0.8 (6)
C8—C9—C10—C5	−47.3 (4)	C17—C20—C21—O23	−177.5 (4)
C11—C9—C10—C1	67.8 (4)	C23—O23—C21—C20	−0.1 (6)
C8—C9—C10—C1	−160.4 (3)	C21—C20—C22—C23	−1.2 (6)
C11—C9—C10—C19	−52.8 (4)	C17—C20—C22—C23	177.1 (4)
C8—C9—C10—C19	78.9 (4)	C20—C22—C23—O23	1.2 (6)
C10—C9—C11—C12	−154.6 (3)	C21—O23—C23—C22	−0.7 (6)
C8—C9—C11—C12	71.6 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O3^{i}	0.82	2.41	3.139 (4)	149
C12—H12A···O15^{ii}	0.97	2.58	3.499 (5)	159
C2—H2A···O7^{iii}	0.97	2.42	3.251 (6)	144
O6—H6···O7	0.82	2.18	2.646 (4)	116

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

Fig. 1

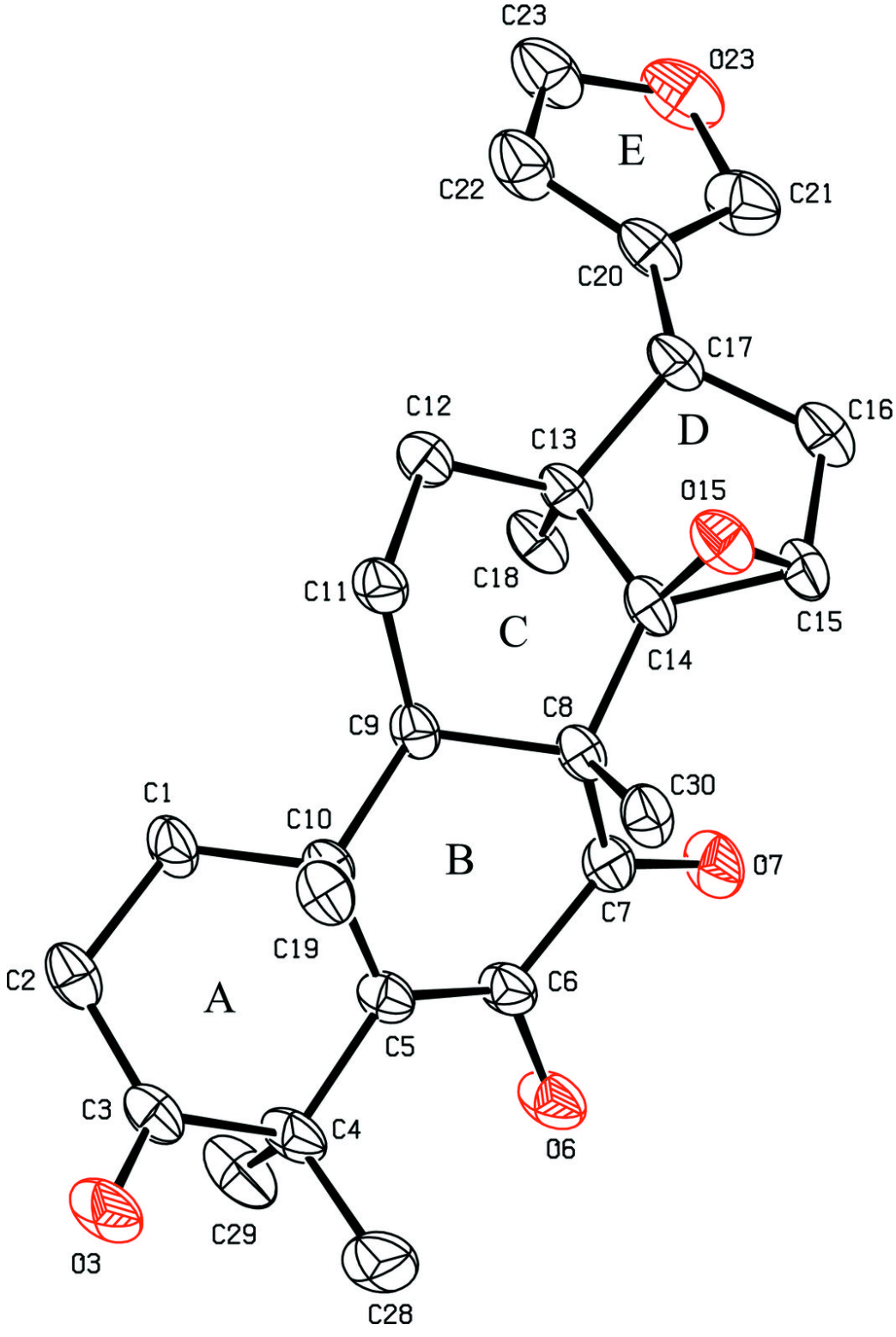
C23
O23
E
C22
C21
C20
C17
C12
C16
C13
D
O15
C18
C11
C
C15
C14
C9
C8
C30
C1
C10
B
O7
C7
C19
C2
A
C6
C5
C3
C4
O6
C29
O3
C28

Fig. 2