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Desacetyl epicaryoptin

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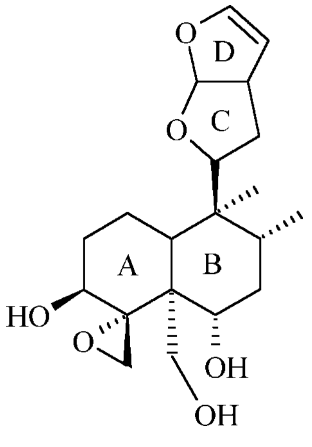
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.051; wR factor = 0.148; data-to-parameter ratio = 12.4.

The title compound, $C_{20}H_{30}O_6$, is a semisynthetic derivative of a diterpene, 3-epicaryoptin, isolated from *Clerodendron calamitosum*. The two fused cyclohexane rings adopt chair conformations and the two fused five-membered rings adopt an envelope and a planar conformation. The O atoms of the hydroxy groups participate in hydrogen bonding and $R_2^1(6)$ and $R_2^2(4)$ ring motifs are formed in the crystal structure.

Related literature

For related literature, see: Bernstein *et al.* (1995); Cremer & Pople (1975); De la Torre *et al.* (1994); Klyne & Prelog (1960); Rodriguez *et al.* (1994); Rogers *et al.* (1979).



Experimental

Crystal data

$C_{20}H_{30}O_6$
M_r = 366.44
Orthorhombic, $P2_12_12_1$
a = 6.643 (5) Å
b = 7.997 (3) Å
c = 33.230 (8) Å
V = 1765.3 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.27 × 0.25 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min}$ = 0.973, $T_{\max}$ = 0.979
3065 measured reflections
2972 independent reflections
1723 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections every 120 reflections intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.148
S = 1.02
2972 reflections
240 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.43 e Å^{-3}
$\Delta\rho_{\min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O4$^{\text{i}}$	0.82	2.51	3.201 (4)	142
O3—H3A···O6$^{\text{i}}$	0.82	2.14	2.813 (3)	139
O6—H6A···O18$^{\text{ii}}$	0.82	1.98	2.786 (4)	166
O18—H18···O6$^{\text{iii}}$	0.82	2.17	2.786 (4)	132

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

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: HG2262).

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supplementary materials

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Comment

The fused *cyclo* hexane rings A and B adopt chair conformations, as evident from the ring puckering parameters (Cremer & Pople, 1975) [Q_T =0.604 (3) Å, φ_2 =-131.7(2.5)°, q_2 =0.091 (3)Å for ring A; Q_T =0.544 (3) Å, φ_2 =106.2(3.7)°, q_2 =0.054 (3)Å for ring B]. Ring C adopts an envelope conformation [φ_2=7.5 (5)°, q_2 =0.385 (3) Å] with apex at C11 which lies 0.581 (3)Å from the plane of the remaining four atoms. Ring D exists in a planar conformation with a maximum deviation −0.025 (3)Å for atom C16 from the plane formed by other atoms. Rings A/B and C/D are *trans* and *cis* fused as seen from the endocyclic dihedral angles of the ring junction atoms. The orientation of hydroxyl group at C3, C7 and C18 are in +*ac* (C1—C2—C3—O3 = 173.8 (4) °), +*sc*(C4—C5—C6—O6 = 70.8 (3)°) and +*sc*(C6—C5—C18—O18 = 78.1 (4) °) respectively (Klyne & Prelog, 1960). The intact epoxide ring at C4 is in *ap* conformation with respect to ring A (C2—C3—C4—O4 is 159.5 (3)). The orientation of furofuran at C9 is in -*sc* conformation with respect to ring B (C8—C9—C11—C12 is −77.1 (4)))°.

The molecules (Fig.2) in the crystal lattice are linked by intermolecular O—H···O hydrogen bonds (Table 2). The hydroxyl oxygen O3 acts as donor for the epoxide oxygen O4 and the hydroxyl group O6 forming a bifurcated hydrogen bond which generates a ring motif $R_2^1(6)$ along 'c' axis. The tandem hydrogen bond configuration formed by the hydroxyl oxygen atoms, O6 and O18, act as hydrogen bond donors and acceptors, generating a ring motif $R_2^2(4)$ parallel to 'b' axis (Bernstein *et al.*, 1995).

Experimental

3-Epicaryoptin (50 mg) in MeOH (15 ml) and water (20 ml) was treated with KOH (70 mg) and refluxed on a water bath for 20 min. The crystalline product was filtered and was subjected to column chromatography resulting in compound (I). Rhombohedral shaped crystals were obtained from a mixture of three solvents *viz.*, carbon tetrachloride, chloroform and methanol in the ratio 2:1:1 at room temperature (293 K).

Refinement

In the absence of suitable anomalous scatters, Friedel equivalents could not be used to determine the absolute structure. Therefore, 1990 Friedel equivalents were merged before the final refinement. The enantiomer employed in the refined model was chosen to agree with the accepted configuration of diterpenes (Rogers *et al.*, 1979; Rodriguez *et al.*, 1994; De la Torre *et al.*, 1994). The C—H and CH_2 atoms were constrained to an ideal geometry (C—H = 0.98, CH_2 = 0.97, O—H = 0.82 A°) with U_{iso}(H) = 1.2U_{eq}(parent atom), but where allowed to rotate freely about the C—C and C—O bonds, respectively. The remaining CH_3 and O—H hydrogen atoms were placed in geometrically idelaized positions (C—H = 0.97–0.98 Å) and constrained to ride on their parent atom with U_{iso}(H) = 1.5 U_{eq}(C).

Figures

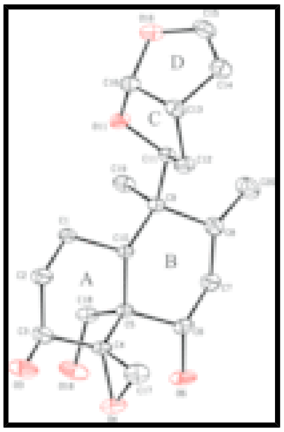

Fig. 1. Molecular structure of the compound with 30% probability displacement ellipsoids and atomic numbering scheme.

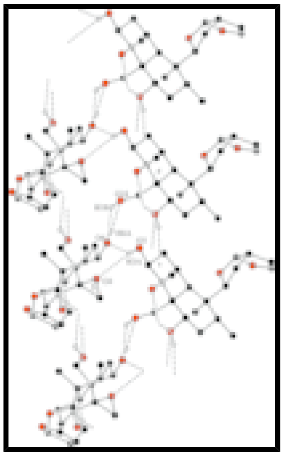

Fig. 2. A view of the crystal packing of (I), showing ring motifs $R_2^1(6)$ and $R_2^2(4)$. The molecules labeled with (*, $, #) correspond to symmetry positions (x, y, z), ($x - 1/2, -y + 3/2, -z + 2$) ($x, y - 1/2, z$) respectively.

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Crystal data

$C_{20}H_{30}O_6$

$M_r = 366.44$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.643$ (5) Å

$b = 7.997$ (3) Å

$c = 33.230$ (8) Å

$V = 1765.3$ (15) Å^3

$Z = 4$

$F_{000} = 792$

$D_x = 1.379$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 1.2$–30.0°

$\mu = 0.10$ mm^{-1}

$T = 293$ (2) K

Rhombohedral, colourless

0.27 × 0.25 × 0.21 mm

Data collection

Enraf–Nonius CAD-4 diffractometer

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

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.973$, $T_{max} = 0.979$

3065 measured reflections

2972 independent reflections

1723 reflections with $I > 2\sigma(I)$

$\theta_{max} = 30.0°$

$\theta_{min} = 1.2°$

$h = 0\rightarrow9$

$k = 0\rightarrow11$

$l = -1\rightarrow46$

3 standard reflections

every 120 reflections

intensity decay: 2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.148$

$S = 1.03$

2972 reflections

240 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

$w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.43$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ 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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.2940 (6)	0.5989 (4)	0.86301 (9)	0.0307 (8)
H1A	0.3183	0.6136	0.8344	0.037*
H1B	0.1807	0.5234	0.8660	0.037*
C2	0.2405 (7)	0.7668 (5)	0.88147 (10)	0.0418 (10)
H2A	0.3436	0.8475	0.8744	0.050*
H2B	0.1143	0.8052	0.8701	0.050*

C3	0.2208 (7)	0.7607 (4)	0.92697 (10)	0.0394 (9)
H3	0.1036	0.6932	0.9345	0.047*
C4	0.4105 (6)	0.6819 (4)	0.94354 (8)	0.0300 (8)
C5	0.4480 (5)	0.5003 (4)	0.92903 (8)	0.0242 (7)
C6	0.6390 (6)	0.4256 (4)	0.94735 (9)	0.0317 (8)
H6	0.7477	0.5077	0.9447	0.038*
C7	0.7023 (6)	0.2675 (4)	0.92603 (10)	0.0358 (8)
H7A	0.5965	0.1848	0.9289	0.043*
H7B	0.8221	0.2237	0.9389	0.043*
C8	0.7456 (6)	0.2928 (4)	0.88144 (10)	0.0339 (8)
H8	0.8531	0.3764	0.8795	0.041*
C9	0.5601 (5)	0.3649 (4)	0.85866 (8)	0.0247 (7)
C10	0.4800 (5)	0.5200 (4)	0.88236 (8)	0.0225 (7)
H10	0.5859	0.6045	0.8796	0.027*
C11	0.6397 (5)	0.4177 (4)	0.81656 (9)	0.0277 (7)
H11	0.7246	0.3262	0.8067	0.033*
C12	0.7590 (6)	0.5775 (4)	0.81173 (9)	0.0347 (8)
H12A	0.8959	0.5641	0.8214	0.042*
H12B	0.6958	0.6695	0.8259	0.042*
C13	0.7541 (5)	0.6050 (4)	0.76640 (9)	0.0322 (8)
H13	0.7524	0.7240	0.7593	0.039*
C14	0.9089 (7)	0.5105 (6)	0.74327 (11)	0.0449 (10)
H14	1.0469	0.5297	0.7442	0.054*
C15	0.8209 (7)	0.3971 (6)	0.72157 (11)	0.0465 (11)
H15	0.8930	0.3242	0.7052	0.056*
C16	0.5590 (6)	0.5156 (5)	0.75390 (9)	0.0325 (8)
H16	0.4629	0.5952	0.7424	0.039*
C17	0.5750 (7)	0.7929 (5)	0.95490 (11)	0.0439 (10)
H17A	0.7114	0.7510	0.9523	0.053*
H17B	0.5592	0.9115	0.9496	0.053*
C18	0.2661 (6)	0.3910 (4)	0.94037 (9)	0.0344 (8)
H18A	0.1555	0.4148	0.9222	0.041*
H18B	0.3017	0.2741	0.9374	0.041*
C19	0.3977 (6)	0.2313 (4)	0.85247 (10)	0.0340 (8)
H19A	0.3441	0.1983	0.8781	0.051*
H19B	0.4556	0.1358	0.8393	0.051*
H19C	0.2915	0.2760	0.8361	0.051*
C20	0.8302 (8)	0.1290 (5)	0.86471 (13)	0.0572 (12)
H20A	0.8594	0.1422	0.8366	0.086*
H20B	0.7329	0.0414	0.8682	0.086*
H20C	0.9515	0.1005	0.8789	0.086*
O3	0.1930 (6)	0.9273 (3)	0.93974 (8)	0.0604 (10)
H3A	0.1624	0.9283	0.9636	0.091*
O4	0.4475 (5)	0.7183 (3)	0.98515 (7)	0.0432 (7)
O6	0.6073 (5)	0.3942 (3)	0.98937 (6)	0.0491 (8)
H6A	0.6489	0.3006	0.9950	0.074*
O11	0.4785 (4)	0.4392 (3)	0.78776 (6)	0.0337 (6)
O16	0.6173 (4)	0.3914 (3)	0.72424 (7)	0.0431 (7)
O18	0.2036 (5)	0.4212 (3)	0.98069 (7)	0.0519 (8)

H18	0.1107	0.3580	0.9864	0.078*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
c1	0.0370 (19)	0.0368 (17)	0.0183 (14)	0.0102 (18)	0.0001 (14)	−0.0005 (14)
c2	0.062 (3)	0.040 (2)	0.0230 (15)	0.025 (2)	−0.0049 (17)	0.0028 (14)
c3	0.053 (2)	0.0356 (18)	0.0295 (17)	0.013 (2)	0.0040 (18)	−0.0012 (15)
c4	0.049 (2)	0.0262 (15)	0.0148 (13)	0.0020 (17)	0.0010 (15)	0.0007 (12)
c5	0.0351 (18)	0.0207 (13)	0.0169 (12)	0.0012 (16)	−0.0008 (13)	0.0011 (11)
c6	0.043 (2)	0.0298 (17)	0.0227 (15)	0.0002 (18)	−0.0055 (15)	0.0030 (13)
c7	0.047 (2)	0.0330 (17)	0.0274 (16)	0.0126 (19)	−0.0032 (17)	0.0050 (14)
c8	0.038 (2)	0.0328 (17)	0.0305 (16)	0.0051 (18)	0.0044 (16)	0.0039 (14)
c9	0.0312 (17)	0.0234 (14)	0.0196 (13)	−0.0018 (15)	0.0024 (14)	0.0012 (12)
c10	0.0292 (17)	0.0232 (15)	0.0150 (11)	0.0001 (14)	−0.0001 (12)	0.0011 (11)
c11	0.0305 (19)	0.0312 (17)	0.0214 (14)	0.0001 (17)	0.0043 (13)	−0.0014 (13)
c12	0.039 (2)	0.0412 (19)	0.0242 (15)	−0.009 (2)	0.0024 (15)	−0.0012 (14)
c13	0.040 (2)	0.0289 (16)	0.0275 (15)	−0.0004 (18)	0.0092 (15)	0.0046 (14)
c14	0.043 (2)	0.057 (3)	0.0343 (17)	0.000 (2)	0.0099 (18)	0.0025 (19)
c15	0.054 (3)	0.055 (3)	0.0300 (18)	0.009 (2)	0.0130 (18)	−0.0013 (19)
c16	0.040 (2)	0.0360 (19)	0.0214 (14)	−0.0018 (19)	0.0030 (16)	0.0033 (14)
c17	0.061 (3)	0.0295 (18)	0.041 (2)	−0.010 (2)	0.000 (2)	−0.0037 (15)
c18	0.048 (2)	0.0335 (17)	0.0217 (14)	−0.004 (2)	0.0034 (16)	−0.0009 (14)
c19	0.045 (2)	0.0267 (16)	0.0302 (16)	−0.0094 (17)	0.0087 (16)	−0.0051 (13)
c20	0.072 (3)	0.055 (3)	0.045 (2)	0.030 (3)	0.006 (2)	0.001 (2)
o3	0.110 (3)	0.0377 (14)	0.0333 (13)	0.0339 (18)	0.0031 (17)	−0.0041 (12)
o4	0.073 (2)	0.0354 (14)	0.0212 (11)	−0.0002 (16)	−0.0067 (13)	−0.0045 (10)
o6	0.088 (2)	0.0416 (15)	0.0182 (11)	0.0190 (18)	−0.0095 (13)	0.0028 (11)
o11	0.0348 (14)	0.0452 (14)	0.0210 (10)	−0.0094 (13)	−0.0006 (10)	0.0038 (10)
o16	0.0570 (18)	0.0453 (16)	0.0269 (12)	−0.0074 (15)	0.0028 (12)	−0.0075 (12)
o18	0.080 (2)	0.0420 (16)	0.0335 (13)	−0.0132 (17)	0.0272 (14)	0.0000 (12)

Geometric parameters (Å, °)

C1—C2	1.519 (5)	C11—C12	1.512 (5)
C1—C10	1.529 (5)	C11—H11	0.9800
C1—H1A	0.9700	C12—C13	1.523 (4)
C1—H1B	0.9700	C12—H12A	0.9700
C2—C3	1.518 (5)	C12—H12B	0.9700
C2—H2A	0.9700	C13—C14	1.490 (5)
C2—H2B	0.9700	C13—C16	1.538 (5)
C3—O3	1.410 (4)	C13—H13	0.9800
C3—C4	1.513 (6)	C14—C15	1.298 (6)
C3—H3	0.9800	C14—H14	0.9300
C4—O4	1.434 (4)	C15—O16	1.356 (5)
C4—C17	1.458 (5)	C15—H15	0.9300
C4—C5	1.550 (5)	C16—O11	1.387 (4)
C5—C6	1.529 (5)	C16—O16	1.452 (4)
C5—C18	1.538 (5)	C16—H16	0.9800

C5—C10	1.573 (4)	C17—O4	1.444 (5)
C6—O6	1.434 (4)	C17—H17A	0.9700
C6—C7	1.509 (5)	C17—H17B	0.9700
C6—H6	0.9800	C18—O18	1.423 (4)
C7—C8	1.523 (4)	C18—H18A	0.9700
C7—H7A	0.9700	C18—H18B	0.9700
C7—H7B	0.9700	C19—H19A	0.9600
C8—C20	1.530 (5)	C19—H19B	0.9600
C8—C9	1.557 (5)	C19—H19C	0.9600
C8—H8	0.9800	C20—H20A	0.9600
C9—C19	1.532 (5)	C20—H20B	0.9600
C9—C11	1.554 (4)	C20—H20C	0.9600
C9—C10	1.562 (4)	O3—H3A	0.8200
C10—H10	0.9800	O6—H6A	0.8200
C11—O11	1.447 (4)	O18—H18	0.8200

C2—C1—C10	112.6 (3)	O11—C11—C12	102.5 (3)
C2—C1—H1A	109.1	O11—C11—C9	112.1 (3)
C10—C1—H1A	109.1	C12—C11—C9	120.2 (3)
C2—C1—H1B	109.1	O11—C11—H11	107.1
C10—C1—H1B	109.1	C12—C11—H11	107.1
H1A—C1—H1B	107.8	C9—C11—H11	107.1
C3—C2—C1	113.2 (3)	C11—C12—C13	102.5 (3)
C3—C2—H2A	108.9	C11—C12—H12A	111.3
C1—C2—H2A	108.9	C13—C12—H12A	111.3
C3—C2—H2B	108.9	C11—C12—H12B	111.3
C1—C2—H2B	108.9	C13—C12—H12B	111.3
H2A—C2—H2B	107.8	H12A—C12—H12B	109.2
O3—C3—C4	113.1 (3)	C14—C13—C12	115.0 (3)
O3—C3—C2	106.3 (3)	C14—C13—C16	101.9 (3)
C4—C3—C2	107.7 (3)	C12—C13—C16	102.6 (3)
O3—C3—H3	109.9	C14—C13—H13	112.2
C4—C3—H3	109.9	C12—C13—H13	112.2
C2—C3—H3	109.9	C16—C13—H13	112.2
O4—C4—C17	59.9 (2)	C15—C14—C13	109.3 (4)
O4—C4—C3	114.1 (3)	C15—C14—H14	125.4
C17—C4—C3	117.7 (3)	C13—C14—H14	125.4
O4—C4—C5	117.6 (3)	C14—C15—O16	115.9 (4)
C17—C4—C5	122.1 (3)	C14—C15—H15	122.1
C3—C4—C5	114.3 (3)	O16—C15—H15	122.1
C6—C5—C18	109.4 (3)	O11—C16—O16	110.6 (3)
C6—C5—C4	112.0 (3)	O11—C16—C13	108.1 (3)
C18—C5—C4	109.3 (3)	O16—C16—C13	106.0 (3)
C6—C5—C10	108.6 (3)	O11—C16—H16	110.6
C18—C5—C10	113.9 (3)	O16—C16—H16	110.6
C4—C5—C10	103.6 (2)	C13—C16—H16	110.6
O6—C6—C7	110.6 (3)	O4—C17—C4	59.3 (2)
O6—C6—C5	109.5 (3)	O4—C17—H17A	117.8
C7—C6—C5	111.8 (3)	C4—C17—H17A	117.8
O6—C6—H6	108.3	O4—C17—H17B	117.8

C7—C6—H6	108.3	C4—C17—H17B	117.8
C5—C6—H6	108.3	H17A—C17—H17B	115.0
C6—C7—C8	113.5 (3)	O18—C18—C5	111.3 (3)
C6—C7—H7A	108.9	O18—C18—H18A	109.4
C8—C7—H7A	108.9	C5—C18—H18A	109.4
C6—C7—H7B	108.9	O18—C18—H18B	109.4
C8—C7—H7B	108.9	C5—C18—H18B	109.4
H7A—C7—H7B	107.7	H18A—C18—H18B	108.0
C7—C8—C20	108.0 (3)	C9—C19—H19A	109.5
C7—C8—C9	111.9 (3)	C9—C19—H19B	109.5
C20—C8—C9	115.6 (3)	H19A—C19—H19B	109.5
C7—C8—H8	107.0	C9—C19—H19C	109.5
C20—C8—H8	107.0	H19A—C19—H19C	109.5
C9—C8—H8	107.0	H19B—C19—H19C	109.5
C19—C9—C11	108.0 (2)	C8—C20—H20A	109.5
C19—C9—C8	111.4 (3)	C8—C20—H20B	109.5
C11—C9—C8	105.6 (3)	H20A—C20—H20B	109.5
C19—C9—C10	112.4 (3)	C8—C20—H20C	109.5
C11—C9—C10	110.7 (2)	H20A—C20—H20C	109.5
C8—C9—C10	108.6 (2)	H20B—C20—H20C	109.5
C1—C10—C9	113.0 (2)	C3—O3—H3A	109.5
C1—C10—C5	110.3 (3)	C4—O4—C17	60.9 (2)
C9—C10—C5	117.6 (2)	C6—O6—H6A	109.5
C1—C10—H10	104.9	C16—O11—C11	107.7 (3)
C9—C10—H10	104.9	C15—O16—C16	106.7 (3)
C5—C10—H10	104.9	C18—O18—H18	109.5
C10—C1—C2—C3	−52.8 (5)	C11—C9—C10—C5	−164.0 (3)
C1—C2—C3—O3	173.8 (4)	C8—C9—C10—C5	−48.5 (4)
C1—C2—C3—C4	52.2 (5)	C6—C5—C10—C1	−178.5 (3)
O3—C3—C4—O4	42.3 (4)	C18—C5—C10—C1	59.4 (4)
C2—C3—C4—O4	159.5 (3)	C4—C5—C10—C1	−59.2 (3)
O3—C3—C4—C17	−25.0 (4)	C6—C5—C10—C9	50.0 (4)
C2—C3—C4—C17	92.2 (4)	C18—C5—C10—C9	−72.2 (4)
O3—C3—C4—C5	−178.4 (3)	C4—C5—C10—C9	169.2 (3)
C2—C3—C4—C5	−61.2 (4)	C19—C9—C11—O11	43.2 (3)
O4—C4—C5—C6	−41.0 (4)	C8—C9—C11—O11	162.4 (3)
C17—C4—C5—C6	29.1 (4)	C10—C9—C11—O11	−80.3 (3)
C3—C4—C5—C6	−178.8 (3)	C19—C9—C11—C12	163.7 (3)
O4—C4—C5—C18	80.4 (4)	C8—C9—C11—C12	−77.1 (4)
C17—C4—C5—C18	150.5 (3)	C10—C9—C11—C12	40.2 (4)
C3—C4—C5—C18	−57.4 (3)	O11—C11—C12—C13	−39.6 (3)
O4—C4—C5—C10	−157.9 (3)	C9—C11—C12—C13	−164.7 (3)
C17—C4—C5—C10	−87.8 (3)	C11—C12—C13—C14	−83.3 (4)
C3—C4—C5—C10	64.3 (4)	C11—C12—C13—C16	26.4 (3)
C18—C5—C6—O6	−50.6 (3)	C12—C13—C14—C15	111.9 (4)
C4—C5—C6—O6	70.8 (3)	C16—C13—C14—C15	1.8 (4)
C10—C5—C6—O6	−175.4 (3)	C13—C14—C15—O16	0.8 (5)
C18—C5—C6—C7	72.3 (3)	C14—C13—C16—O11	115.3 (3)
C4—C5—C6—C7	−166.3 (3)	C12—C13—C16—O11	−4.0 (4)

C10—C5—C6—C7	−52.5 (4)	C14—C13—C16—O16	−3.4 (3)
O6—C6—C7—C8	−178.0 (3)	C12—C13—C16—O16	−122.7 (3)
C5—C6—C7—C8	59.6 (4)	C3—C4—C17—O4	103.3 (3)
C6—C7—C8—C20	173.7 (4)	C5—C4—C17—O4	−105.5 (3)
C6—C7—C8—C9	−58.0 (4)	C6—C5—C18—O18	77.7 (4)
C7—C8—C9—C19	−74.5 (4)	C4—C5—C18—O18	−45.3 (4)
C20—C8—C9—C19	49.6 (4)	C10—C5—C18—O18	−160.6 (3)
C7—C8—C9—C11	168.6 (3)	C3—C4—O4—C17	−109.2 (3)
C20—C8—C9—C11	−67.3 (4)	C5—C4—O4—C17	112.9 (4)
C7—C8—C9—C10	49.8 (4)	O16—C16—O11—C11	94.1 (3)
C20—C8—C9—C10	173.9 (3)	C13—C16—O11—C11	−21.6 (4)
C2—C1—C10—C9	−169.3 (3)	C12—C11—O11—C16	38.7 (3)
C2—C1—C10—C5	56.8 (4)	C9—C11—O11—C16	169.0 (3)
C19—C9—C10—C1	−55.1 (3)	C14—C15—O16—C16	−3.1 (5)
C11—C9—C10—C1	65.7 (4)	O11—C16—O16—C15	−113.0 (3)
C8—C9—C10—C1	−178.8 (3)	C13—C16—O16—C15	4.0 (4)
C19—C9—C10—C5	75.2 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O4^{i}	0.82	2.51	3.201 (4)	142
O3—H3A···O6^{i}	0.82	2.14	2.813 (3)	139
O6—H6A···O18^{ii}	0.82	1.98	2.786 (4)	166
O18—H18···O6^{iii}	0.82	2.17	2.786 (4)	132

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

Fig. 1

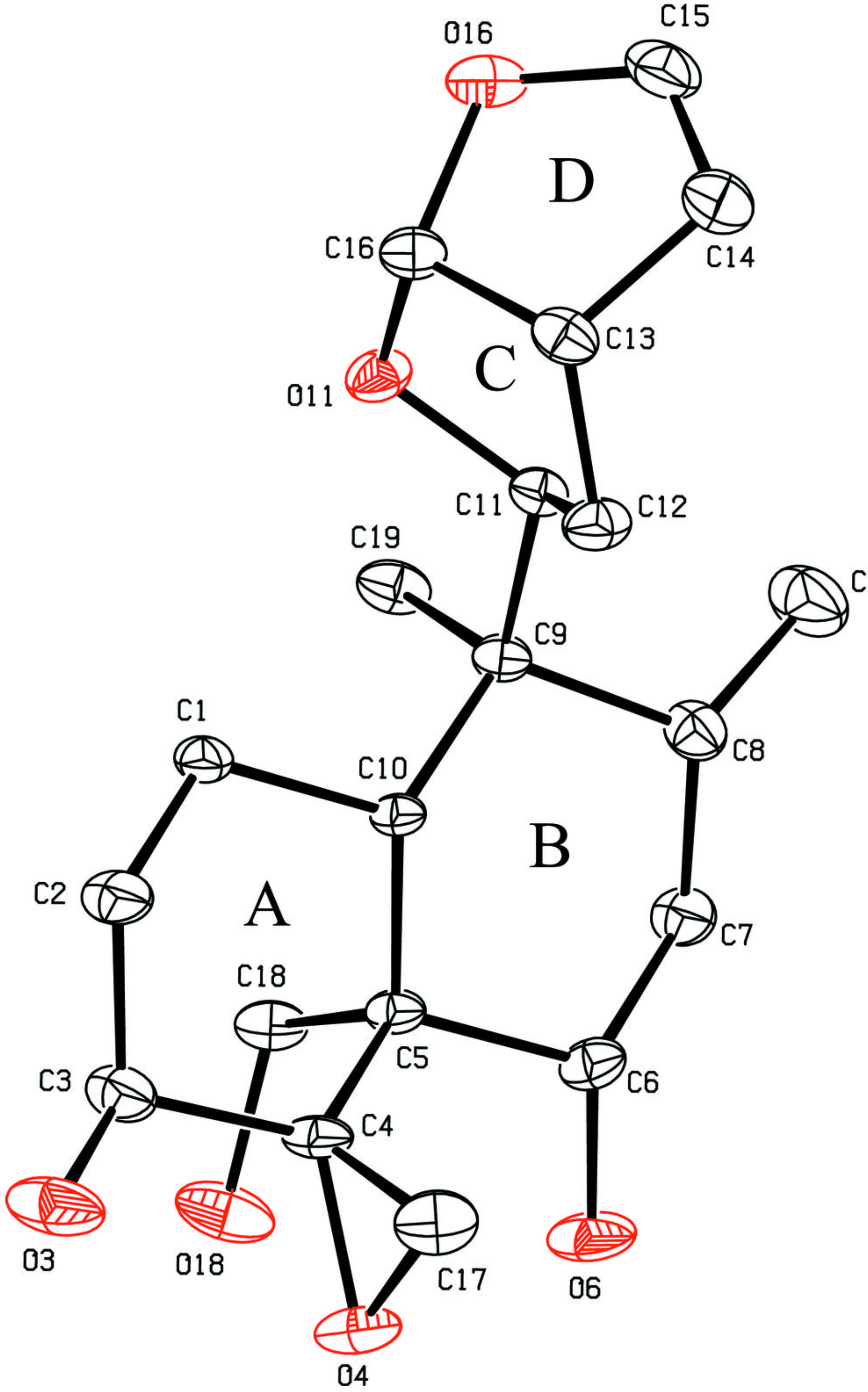
O16
C15
D
C16
C14
C13
C
O11
C11
C12
C19
C20
C9
C1
C8
C10
B
C2
A
C7
C18
C5
C6
C3
C4
O3
O18
C17
O6
O4

Fig. 2