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(2*R**,3*R**,4a*S**,6a*R**,11a*S**,11b*S**)-Methyl 2-acetoxy-11b-hydroxy-3,7-dimethyl-1,2,3,4,4a,5,6,6a,7,11,11a,11b-dodecahydrophenanthro[3,2-*b*]furan-3-carboxylate

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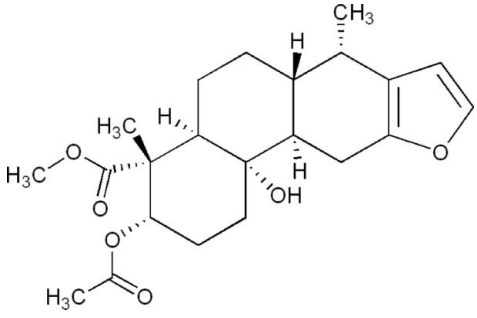
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.094; data-to-parameter ratio = 8.4.

In the title compound, $C_{22}H_{30}O_6$, the conformation of the molecule is dictated by an intramolecular C—H···O contact. The crystal structure is stabilized *via* intermolecular C—H···O, O—H···O and C—H···π contacts.

Related literature

For related literature see: Ruggiero *et al.* (1997); Chopra *et al.* (1992); Pullaih (2006); Kirtikar & Basu (1993); Parrota (2000); Boeyens (1978); Cremer & Pople (1975).



Experimental

Crystal data

$C_{22}H_{30}O_6$
M_r = 390.46
Orthorhombic, $P2_12_12_1$
a = 12.2339 (14) Å
b = 12.8744 (15) Å
c = 12.8783 (15) Å
V = 2028.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.25 × 0.21 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.918, T_{max} = 0.987
15340 measured reflections
2160 independent reflections
1875 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.094
S = 1.08
2160 reflections
258 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

Cg is the centroid of the furan ring.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O6^{i}	0.82	2.11	2.929 (3)	173
C11—H11*B*···O5	0.97	2.47	3.092 (3)	122
C15—H15*B*···O4^{ii}	0.97	2.56	3.460 (3)	154
C22—H22*A*···*Cg*^{iii}	0.96	2.70	3.54 (3)	147

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2076).

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supplementary materials

Acta Cryst. (2008). E**64**, o9 [**doi:10.1107/S1600536807061934**]

(2*R**,3*R**,4a*S**,6a*R**,11a*S**,11b*S**)-Methyl 2-acetoxy-11b-hydroxy-3,7-dimethyl-1,2,3,4,4a,5,6,6a,7,11,11a,11b-dodecahydrophenanthro[3,2-*b*]furan-3-carboxylate

S. J. Prathap, L. N. Rajanna, C. Sheetal, Y. N. Seetharam and T. N. G. Row

Comment

The title compound was isolated from the mature seed kernels of Caesalpinia decapetala (Roth.) Alston., which belongs to the family Fabaceae (Caesalpinioideae), a thorny woody climbing shrub, native to tropical and subtropical Asia and distributed in India, China, Sri Lanka, Malaysia, Korea, Vietnam and Japan(Parrota, 2000). In India, the plant is popularly known as Mysore thorn and locally as "Kurudu gajjuga". Its leaves and seeds are known to have anthelmintic, antipyretic, astringent, purgative, emmenagogue, febrifuge, and analgesic properties, and are thus used in the indegenous system of medicine for the treatment of dysentery and malarial fever (Kirtikar & Basu, 1993; Pullaih, 2006). The stem bark of the plant is widely used in the tanning industry and as a laxative (Chopra *et al.*, 1992).

The title compound is a tetracyclic molecule, consisting of a furan ring fused to a *syn*,anti,anti-perhydrophenanthrene system. The molecular conformation of the compound(I) leads to the formation of a C—H···O intra molecular hydrogen bond. The puckering parameters (Cremer & Pople,1975) for the cyclohexane ring A [q_2 = 0.0612 (3) Å, q_3 = 0.540 (3) Å, φ_2 = 96 (2)°, Q_T = 0.543 (3) Å and θ_2 = 6.46 (2)°] describe a distorted chair conformation·The total puckering amplitude Q_T is only slightly smaller than that for ideal chair (0.63 Å). φ_2 is close to 90°, which corresponds to a twist-boat conformation. Because of the 1,3 diaxial interactions, the cyclohexane ring A is distorted from an ideal chair conformation. This is most evident in the twisting of the six-membered ring at C17, which allows the C12—C17—C16 angle to increse to 113.01°. As evident from its puckering parameters [q_2 = 0.023 (3) Å, q_3 = 0.568 (2) Å, φ_2 = −82.4 (3)°, Q_T = 0.568 (2) Å and θ_2 = 2.34 (3)°], the conformation of the cyclohexane ring B can also be best described as chair, distorted in the same manner as ring A due to 1,3 diaxial interaction. On account of its fusion with the furan ring, ring C has the expected half chair conformation of a cyclohexene ring [q_2 = 0.328 (3) Å, q_3 = 0.269 (3) Å, φ_2 = −124.68 (1)°, Q_T = 0.424 (2) Å and θ_2 = 50.63 (3)°]·(Boeyens, 1978)

The crystal structure of (I)is generated by intermolecular O—H···O and C—H···O contacts forming a zig zag pattern parallel to the *b* axis. An intermolecular C—H···π interaction between H22A and the furan ring further stabilizes the packing.

Experimental

Mature seed kernels of Caesalpinia decapetala were collected from Bhalki, Bidar District, Karnataka. A specimen is deposited in the herbarium Department of Botany, Gulbarga University, Gulbarga, Karnataka, India. with voucher specimen No·HGUG-209. Seeds were finely ground (particle size 2 mm) and extracted with soxhlet extractor with n-hexane for 20 h and maintaining the Temperature at 333 K Oil recovered was weighed (29/100 g ms) and stored in air tight container for further analysis. The oil obtained was taken in glass test tube covered with aluminium foil and kept in refrigerator, after 15 days of storage granular particles were setteed at the bottom of the test tube. These particles were separated and washed with n-hexane and then it was repeatedly washed with petroleum ether and dried at the room temperature, these fine powdered

particles were re-dissolved in double distilled alcohol and kept for 4–8 days for crystallization. After 24 h formation of pointed colorless crystals were formed at the bottom of the container.

Refinement

All hydrogen atoms were initially located in a difference Fourier map. The methine (CH) and methylene (CH_2) H atoms were then placed in geometrically idealized positions and allowed to ride on their parent atoms with C—H distances in the range 0.97–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The CH_3 and OH hydrogen atoms were constrained to an ideal geometry with C—H distances as 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, and O—H distances fixed at 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$. During refinement, each methyl and hydroxyl group was however allowed to rotate freely about its C—C and C—O bond respectively.

The absolute configuration could not be determined from the diffraction data, and the configuration shown is arbitary.

Figures

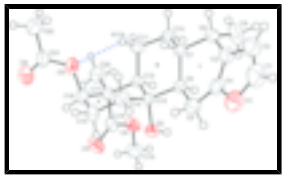

Fig. 1. *ORTEP* diagram with 50% probability ellipsoids. The dotted lines show the intramolecular C—H···O contact.

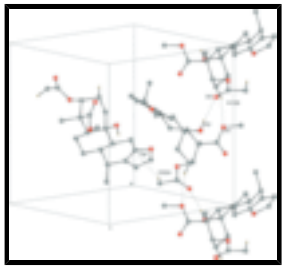

Fig. 2. Packing diagram of (I). The dotted lines indicate intermolecular contacts.

(2*R*,3*R*,4aS,6aR,11aS,11bS)-Methyl 2-acetoxy-11*b*-hydroxy-3,7-dimethyl-1,2,3,4,4a,5,6,6a,7,11,11*a*,11*b*- dodecahydrophenanthro[3,2-*b*]furan-3-carboxylate

Crystal data

$C_{22}H_{30}O_6$	$F_{000} = 840$
$M_r = 390.46$	$D_x = 1.279$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 729 reflections
$a = 12.2339$ (14) Å	$\theta = 2.2$–$22.9°$
$b = 12.8744$ (15) Å	$\mu = 0.09$ mm^{-1}
$c = 12.8783$ (15) Å	$T = 293$ (2) K
$V = 2028.4$ (4) Å^3	Block, colorless
$Z = 4$	0.25 × 0.21 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2160 independent reflections
Radiation source: fine-focus sealed tube	1875 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.037$
$T = 293(2)$ K	$\theta_{max} = 25.5^{o}$
φ and ω scans	$\theta_{min} = 2.2^{o}$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -14 \rightarrow 13$
$T_{min} = 0.918$, $T_{max} = 0.987$	$k = -15 \rightarrow 15$
15340 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.3619P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.094$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.22$ e Å^{-3}
2160 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
258 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Flack parameter: −0.9 (15)

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}
C9	0.3474 (3)	0.7674 (2)	0.3974 (3)	0.0652 (10)
H9A	0.4108	0.8105	0.4059	0.098*
H9B	0.2929	0.7869	0.4474	0.098*
H9C	0.3186	0.7761	0.3287	0.098*

C22	−0.0003 (3)	0.2087 (3)	0.5937 (3)	0.0731 (11)
H22A	−0.0429	0.2543	0.6366	0.110*
H22B	0.0757	0.2260	0.6001	0.110*
H22C	−0.0116	0.1382	0.6154	0.110*
C19	−0.1573 (3)	0.5306 (3)	0.0682 (2)	0.0616 (9)
H19A	−0.1078	0.5062	0.0158	0.092*
H19B	−0.1765	0.6015	0.0543	0.092*
H19C	−0.2220	0.4885	0.0679	0.092*
C20	−0.1288 (2)	0.4491 (2)	0.3766 (2)	0.0503 (7)
H20A	−0.1461	0.5216	0.3711	0.075*
H20B	−0.1114	0.4326	0.4474	0.075*
H20C	−0.1905	0.4086	0.3547	0.075*
C21	−0.0350 (3)	0.2205 (2)	0.4829 (2)	0.0517 (8)
O6	−0.1078 (2)	0.1728 (2)	0.4426 (2)	0.0828 (8)
O5	0.02457 (17)	0.29224 (15)	0.43436 (15)	0.0500 (5)
O2	0.14299 (15)	0.46942 (13)	0.15370 (12)	0.0366 (4)
H2	0.1343	0.5287	0.1319	0.055*
C12	0.0648 (2)	0.50420 (18)	0.32357 (18)	0.0301 (5)
H12	0.0399	0.5705	0.2945	0.036*
C6	0.2613 (2)	0.5535 (2)	0.27923 (19)	0.0337 (6)
H6	0.2344	0.6191	0.2505	0.040*
O4	−0.10551 (16)	0.52378 (14)	0.16832 (15)	0.0461 (5)
O1	0.56112 (16)	0.55596 (18)	0.21108 (18)	0.0565 (6)
C13	0.1683 (2)	0.47287 (19)	0.26309 (18)	0.0297 (5)
C11	0.0915 (2)	0.5238 (2)	0.43841 (19)	0.0369 (6)
H11A	0.0262	0.5473	0.4741	0.044*
H11B	0.1151	0.4594	0.4705	0.044*
C17	−0.0302 (2)	0.4242 (2)	0.3072 (2)	0.0362 (6)
C14	0.2016 (2)	0.3619 (2)	0.2903 (2)	0.0375 (6)
H14A	0.2660	0.3431	0.2507	0.045*
H14B	0.2202	0.3584	0.3635	0.045*
C7	0.2860 (2)	0.5744 (2)	0.39496 (19)	0.0324 (6)
H7	0.3096	0.5083	0.4253	0.039*
C10	0.1810 (2)	0.6052 (2)	0.4498 (2)	0.0356 (6)
H10A	0.1552	0.6706	0.4216	0.043*
H10B	0.1962	0.6156	0.5230	0.043*
O3	−0.08866 (18)	0.35433 (16)	0.14050 (16)	0.0563 (6)
C8	0.3792 (2)	0.6534 (2)	0.4140 (2)	0.0407 (7)
H8	0.4027	0.6459	0.4863	0.049*
C16	0.0060 (2)	0.3107 (2)	0.3240 (2)	0.0413 (7)
H16	−0.0520	0.2642	0.2996	0.050*
C15	0.1106 (2)	0.2847 (2)	0.2674 (2)	0.0437 (7)
H15A	0.0966	0.2839	0.1933	0.052*
H15B	0.1344	0.2157	0.2876	0.052*
C5	0.3634 (2)	0.5242 (3)	0.2148 (2)	0.0548 (8)
H5A	0.3707	0.4492	0.2127	0.066*
H5B	0.3544	0.5489	0.1442	0.066*
C3	0.4741 (2)	0.6255 (2)	0.3464 (2)	0.0404 (6)
C18	−0.0742 (2)	0.4278 (2)	0.1953 (2)	0.0384 (6)

C4	0.4630 (2)	0.5699 (2)	0.2598 (2)	0.0428 (7)
C1	0.6359 (3)	0.6066 (3)	0.2721 (3)	0.0648 (10)
H1	0.7104	0.6099	0.2582	0.078*
C2	0.5880 (3)	0.6503 (3)	0.3537 (3)	0.0569 (8)
H2A	0.6218	0.6892	0.4054	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C9	0.056 (2)	0.0416 (17)	0.098 (3)	−0.0105 (15)	0.007 (2)	−0.0149 (17)
C22	0.106 (3)	0.061 (2)	0.052 (2)	−0.014 (2)	0.017 (2)	0.0037 (17)
C19	0.072 (2)	0.0600 (19)	0.0530 (19)	0.0043 (18)	−0.0258 (18)	0.0001 (16)
C20	0.0325 (15)	0.0645 (19)	0.0538 (17)	−0.0025 (14)	0.0042 (14)	−0.0057 (16)
C21	0.064 (2)	0.0372 (15)	0.0538 (18)	−0.0114 (16)	0.0164 (17)	−0.0016 (13)
O6	0.104 (2)	0.0746 (16)	0.0696 (16)	−0.0517 (16)	0.0108 (16)	−0.0020 (13)
O5	0.0554 (12)	0.0465 (11)	0.0480 (12)	−0.0137 (10)	−0.0006 (11)	0.0090 (9)
O2	0.0452 (11)	0.0382 (9)	0.0266 (9)	−0.0011 (9)	−0.0021 (8)	−0.0024 (7)
C12	0.0298 (13)	0.0295 (12)	0.0312 (12)	0.0017 (10)	0.0000 (10)	−0.0010 (10)
C6	0.0348 (13)	0.0363 (13)	0.0301 (13)	−0.0047 (11)	−0.0004 (11)	0.0009 (11)
O4	0.0492 (11)	0.0427 (10)	0.0463 (11)	0.0064 (9)	−0.0158 (10)	−0.0059 (9)
O1	0.0363 (11)	0.0690 (14)	0.0642 (13)	−0.0013 (10)	0.0135 (10)	0.0010 (12)
C13	0.0301 (12)	0.0341 (12)	0.0249 (12)	−0.0001 (11)	−0.0029 (10)	−0.0009 (10)
C11	0.0349 (14)	0.0439 (14)	0.0319 (13)	0.0005 (12)	0.0018 (12)	−0.0028 (12)
C17	0.0298 (13)	0.0397 (14)	0.0390 (14)	−0.0019 (12)	−0.0009 (12)	−0.0026 (12)
C14	0.0338 (14)	0.0401 (14)	0.0385 (15)	0.0058 (12)	0.0037 (12)	0.0019 (12)
C7	0.0333 (13)	0.0348 (13)	0.0291 (13)	−0.0003 (11)	−0.0022 (11)	0.0024 (11)
C10	0.0442 (15)	0.0370 (13)	0.0257 (13)	0.0005 (12)	−0.0026 (12)	−0.0029 (11)
O3	0.0641 (14)	0.0463 (11)	0.0586 (13)	−0.0059 (11)	−0.0183 (12)	−0.0121 (10)
C8	0.0375 (15)	0.0475 (16)	0.0370 (15)	−0.0064 (13)	−0.0054 (13)	−0.0027 (12)
C16	0.0400 (15)	0.0395 (14)	0.0445 (16)	−0.0069 (12)	−0.0020 (13)	0.0027 (12)
C15	0.0496 (16)	0.0302 (13)	0.0512 (17)	0.0026 (13)	0.0000 (15)	0.0032 (12)
C5	0.0432 (17)	0.077 (2)	0.0444 (16)	−0.0173 (16)	0.0143 (14)	−0.0112 (16)
C3	0.0329 (14)	0.0425 (14)	0.0458 (17)	−0.0036 (12)	−0.0079 (13)	0.0083 (13)
C18	0.0284 (13)	0.0395 (14)	0.0473 (16)	−0.0052 (11)	−0.0039 (12)	−0.0035 (13)
C4	0.0335 (15)	0.0516 (16)	0.0434 (16)	−0.0037 (13)	0.0065 (13)	0.0046 (13)
C1	0.0292 (16)	0.083 (2)	0.082 (3)	−0.0071 (16)	−0.0034 (18)	0.012 (2)
C2	0.0401 (17)	0.069 (2)	0.061 (2)	−0.0079 (16)	−0.0107 (17)	0.0060 (17)

Geometric parameters (Å, °)

C9—C8	1.532 (4)	O1—C1	1.370 (4)
C9—H9A	0.9600	C13—C14	1.526 (3)
C9—H9B	0.9600	C11—C10	1.523 (4)
C9—H9C	0.9600	C11—H11A	0.9700
C22—C21	1.496 (5)	C11—H11B	0.9700
C22—H22A	0.9600	C17—C18	1.539 (4)
C22—H22B	0.9600	C17—C16	1.543 (4)
C22—H22C	0.9600	C14—C15	1.521 (4)
C19—O4	1.439 (3)	C14—H14A	0.9700

C19—H19A	0.9600	C14—H14B	0.9700
C19—H19B	0.9600	C7—C10	1.519 (4)
C19—H19C	0.9600	C7—C8	1.547 (3)
C20—C17	1.536 (4)	C7—H7	0.9800
C20—H20A	0.9600	C10—H10A	0.9700
C20—H20B	0.9600	C10—H10B	0.9700
C20—H20C	0.9600	O3—C18	1.193 (3)
C21—O6	1.200 (4)	C8—C3	1.495 (4)
C21—O5	1.333 (3)	C8—H8	0.9800
O5—C16	1.459 (3)	C16—C15	1.509 (4)
O2—C13	1.443 (3)	C16—H16	0.9800
O2—H2	0.8200	C15—H15A	0.9700
C12—C11	1.535 (3)	C15—H15B	0.9700
C12—C13	1.541 (3)	C5—C4	1.472 (4)
C12—C17	1.567 (3)	C5—H5A	0.9700
C12—H12	0.9800	C5—H5B	0.9700
C6—C7	1.544 (3)	C3—C4	1.332 (4)
C6—C5	1.545 (4)	C3—C2	1.433 (4)
C6—C13	1.553 (3)	C1—C2	1.328 (5)
C6—H6	0.9800	C1—H1	0.9300
O4—C18	1.340 (3)	C2—H2A	0.9300
O1—C4	1.367 (3)		
C8—C9—H9A	109.5	C18—C17—C12	111.5 (2)
C8—C9—H9B	109.5	C16—C17—C12	113.0 (2)
H9A—C9—H9B	109.5	C15—C14—C13	111.9 (2)
C8—C9—H9C	109.5	C15—C14—H14A	109.2
H9A—C9—H9C	109.5	C13—C14—H14A	109.2
H9B—C9—H9C	109.5	C15—C14—H14B	109.2
C21—C22—H22A	109.5	C13—C14—H14B	109.2
C21—C22—H22B	109.5	H14A—C14—H14B	107.9
H22A—C22—H22B	109.5	C10—C7—C6	109.2 (2)
C21—C22—H22C	109.5	C10—C7—C8	112.2 (2)
H22A—C22—H22C	109.5	C6—C7—C8	114.3 (2)
H22B—C22—H22C	109.5	C10—C7—H7	106.9
O4—C19—H19A	109.5	C6—C7—H7	106.9
O4—C19—H19B	109.5	C8—C7—H7	106.9
H19A—C19—H19B	109.5	C7—C10—C11	112.6 (2)
O4—C19—H19C	109.5	C7—C10—H10A	109.1
H19A—C19—H19C	109.5	C11—C10—H10A	109.1
H19B—C19—H19C	109.5	C7—C10—H10B	109.1
C17—C20—H20A	109.5	C11—C10—H10B	109.1
C17—C20—H20B	109.5	H10A—C10—H10B	107.8
H20A—C20—H20B	109.5	C3—C8—C9	110.3 (2)
C17—C20—H20C	109.5	C3—C8—C7	108.8 (2)
H20A—C20—H20C	109.5	C9—C8—C7	114.9 (2)
H20B—C20—H20C	109.5	C3—C8—H8	107.5
O6—C21—O5	123.9 (3)	C9—C8—H8	107.5
O6—C21—C22	124.9 (3)	C7—C8—H8	107.5
O5—C21—C22	111.3 (3)	O5—C16—C15	107.6 (2)

C21—O5—C16	119.0 (2)	O5—C16—C17	109.6 (2)
C13—O2—H2	109.5	C15—C16—C17	112.7 (2)
C11—C12—C13	110.8 (2)	O5—C16—H16	109.0
C11—C12—C17	113.3 (2)	C15—C16—H16	109.0
C13—C12—C17	111.69 (19)	C17—C16—H16	109.0
C11—C12—H12	106.9	C16—C15—C14	112.5 (2)
C13—C12—H12	106.9	C16—C15—H15A	109.1
C17—C12—H12	106.9	C14—C15—H15A	109.1
C7—C6—C5	113.7 (2)	C16—C15—H15B	109.1
C7—C6—C13	112.88 (19)	C14—C15—H15B	109.1
C5—C6—C13	110.9 (2)	H15A—C15—H15B	107.8
C7—C6—H6	106.2	C4—C5—C6	111.1 (2)
C5—C6—H6	106.2	C4—C5—H5A	109.4
C13—C6—H6	106.2	C6—C5—H5A	109.4
C18—O4—C19	114.5 (2)	C4—C5—H5B	109.4
C4—O1—C1	105.1 (2)	C6—C5—H5B	109.4
O2—C13—C14	104.64 (19)	H5A—C5—H5B	108.0
O2—C13—C12	108.96 (19)	C4—C3—C2	105.9 (3)
C14—C13—C12	110.4 (2)	C4—C3—C8	122.5 (2)
O2—C13—C6	107.97 (18)	C2—C3—C8	131.6 (3)
C14—C13—C6	113.5 (2)	O3—C18—O4	122.4 (3)
C12—C13—C6	111.06 (19)	O3—C18—C17	125.6 (3)
C10—C11—C12	111.1 (2)	O4—C18—C17	111.7 (2)
C10—C11—H11A	109.4	C3—C4—O1	111.4 (2)
C12—C11—H11A	109.4	C3—C4—C5	129.0 (3)
C10—C11—H11B	109.4	O1—C4—C5	119.6 (2)
C12—C11—H11B	109.4	C2—C1—O1	111.2 (3)
H11A—C11—H11B	108.0	C2—C1—H1	124.4
C20—C17—C18	105.3 (2)	O1—C1—H1	124.4
C20—C17—C16	110.0 (2)	C1—C2—C3	106.4 (3)
C18—C17—C16	105.0 (2)	C1—C2—H2A	126.8
C20—C17—C12	111.5 (2)	C3—C2—H2A	126.8
O6—C21—O5—C16	−3.9 (4)	C21—O5—C16—C17	119.3 (3)
C22—C21—O5—C16	176.9 (3)	C20—C17—C16—O5	−53.6 (3)
C11—C12—C13—O2	−172.05 (19)	C18—C17—C16—O5	−166.4 (2)
C17—C12—C13—O2	60.6 (3)	C12—C17—C16—O5	71.8 (3)
C11—C12—C13—C14	73.6 (2)	C20—C17—C16—C15	−173.3 (2)
C17—C12—C13—C14	−53.8 (3)	C18—C17—C16—C15	73.8 (3)
C11—C12—C13—C6	−53.2 (3)	C12—C17—C16—C15	−48.0 (3)
C17—C12—C13—C6	179.41 (19)	O5—C16—C15—C14	−69.3 (3)
C7—C6—C13—O2	172.8 (2)	C17—C16—C15—C14	51.6 (3)
C5—C6—C13—O2	−58.2 (3)	C13—C14—C15—C16	−57.2 (3)
C7—C6—C13—C14	−71.6 (3)	C7—C6—C5—C4	−27.6 (4)
C5—C6—C13—C14	57.3 (3)	C13—C6—C5—C4	−156.2 (2)
C7—C6—C13—C12	53.4 (3)	C9—C8—C3—C4	−103.6 (3)
C5—C6—C13—C12	−177.6 (2)	C7—C8—C3—C4	23.2 (4)
C13—C12—C11—C10	55.8 (3)	C9—C8—C3—C2	74.5 (4)
C17—C12—C11—C10	−177.7 (2)	C7—C8—C3—C2	−158.7 (3)
C11—C12—C17—C20	47.8 (3)	C19—O4—C18—O3	−0.2 (4)

C13—C12—C17—C20	173.8 (2)	C19—O4—C18—C17	−174.3 (2)
C11—C12—C17—C18	165.3 (2)	C20—C17—C18—O3	−107.8 (3)
C13—C12—C17—C18	−68.8 (3)	C16—C17—C18—O3	8.3 (4)
C11—C12—C17—C16	−76.7 (3)	C12—C17—C18—O3	131.1 (3)
C13—C12—C17—C16	49.3 (3)	C20—C17—C18—O4	66.1 (3)
O2—C13—C14—C15	−59.1 (3)	C16—C17—C18—O4	−177.8 (2)
C12—C13—C14—C15	57.9 (3)	C12—C17—C18—O4	−55.1 (3)
C6—C13—C14—C15	−176.6 (2)	C2—C3—C4—O1	0.6 (3)
C5—C6—C7—C10	178.4 (2)	C8—C3—C4—O1	179.1 (2)
C13—C6—C7—C10	−54.1 (3)	C2—C3—C4—C5	180.0 (3)
C5—C6—C7—C8	51.8 (3)	C8—C3—C4—C5	−1.5 (5)
C13—C6—C7—C8	179.3 (2)	C1—O1—C4—C3	−0.1 (3)
C6—C7—C10—C11	56.6 (3)	C1—O1—C4—C5	−179.6 (3)
C8—C7—C10—C11	−175.6 (2)	C6—C5—C4—C3	3.3 (5)
C12—C11—C10—C7	−58.6 (3)	C6—C5—C4—O1	−177.4 (3)
C10—C7—C8—C3	−172.5 (2)	C4—O1—C1—C2	−0.4 (4)
C6—C7—C8—C3	−47.4 (3)	O1—C1—C2—C3	0.7 (4)
C10—C7—C8—C9	−48.3 (3)	C4—C3—C2—C1	−0.8 (4)
C6—C7—C8—C9	76.7 (3)	C8—C3—C2—C1	−179.1 (3)
C21—O5—C16—C15	−117.9 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O6^{i}	0.82	2.11	2.929 (3)	173
C11—H11B···O5	0.97	2.47	3.092 (3)	122
C15—H15B···O4^{ii}	0.97	2.56	3.460 (3)	154
C22—H22A···Cg^{iii}	0.96	2.70	3.54 (3)	147

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

Fig. 1

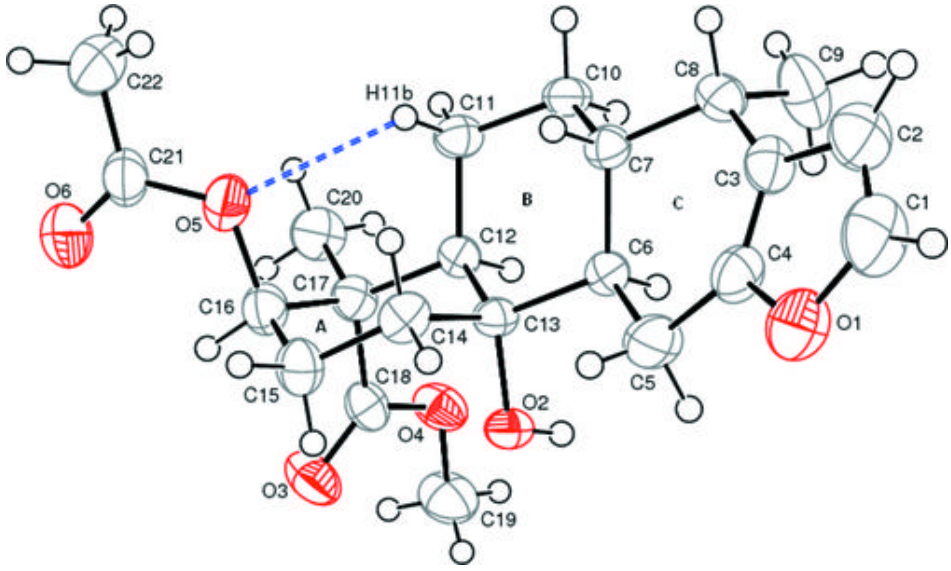
C22
C21
O6
O5
H11b
C11
C10
C8
C9
C2
C7
C3
C1
C20
B
C
C12
C6
C4
C17
C16
A
C14
C13
O1
C15
C18
C5
O2
O4
O3
C19

Fig. 2