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1,3,7-Trideacetylkhivorin

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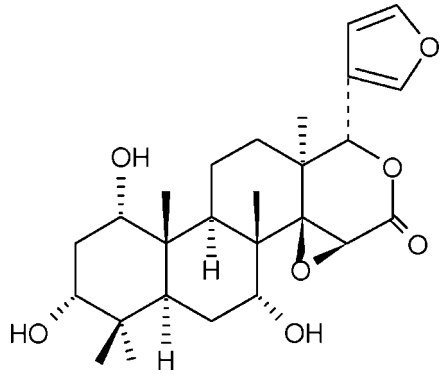
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.037; wR factor = 0.094; data-to-parameter ratio = 7.3.

The title D-*seco* limonoid, named 1,3,7-trideacetylkhivorin (systematic name: 14,15β:21,23-diepoxy-1α,3α,7α-trihydroxy-4,4,8-trimethyl-D-*homo*-24-*nor*-17-oxochola-20,22-diene-16-one), $C_{26}H_{36}O_7$, was isolated from the stem bark of African mahogany *Khaya senegalensis* (Meliaceae). The four fused six-membered rings adopt chair, chair, boat and half-chair conformations. The five-membered furan ring is disordered by a 180° rotation about the bond linking it to the pyran ring. The crystal structure is stabilized by strong classical O—H···O hydrogen-bond interactions to form a network.

Related literature

For general background, see: Androulakis *et al.* (2006). Details of the chemical modification from the original chemical khivorin are given by Bevan *et al.* (1963). Isolation from *Khaya ivorensis* and the antifungal activity of the title compound are described by Abdelgaleil *et al.* (2005). Details of the similar D-*seco* limonoids gedunin and 3,7-dideacetyl khivorin and their *in vitro* antitumor activities are given by: Toscano *et al.* (1996); Uddin *et al.* (2007); Zhang, Wang *et al.* (2007); Zhang, VanDerveer *et al.* (2007).



Experimental

Crystal data

$C_{26}H_{36}O_7$
M_r = 460.55
Orthorhombic, $P2_12_12_1$
a = 9.856 (2) Å
b = 14.492 (3) Å
c = 15.714 (3) Å
V = 2244.5 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 153 (2) K
0.41 × 0.41 × 0.31 mm

Data collection

Riguka Mercury CCD diffractometer
Absorption correction: multi-scan (*REQAB*; Rigaku/MSC, 2006)
T_{min} = 0.961, T_{max} = 0.970
16840 measured reflections
2222 independent reflections
2133 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.094
S = 1.07
2222 reflections
306 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O7^{i}	0.83	2.24	3.015 (3)	155
O4—H4···O3^{ii}	0.83	1.98	2.800 (2)	171

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

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: PK2059).

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supplementary materials

Acta Cryst. (2007). E**63**, o4599 [**doi:10.1107/S1600536807055110**]

1,3,7-Trideacetylkhivorin

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Comment

1,3,7-trideacetylkhivorin, a D-*seco* limonoid, has been isolated by trichloromethane extract of the stem bark of African mahogany *Khaya senegalensis* belonging to the Meliaceae family. Our previous study showed the stem bark extract displayed anti-proliferative effects on HT-29, HCT-15, and HCA-7 cells (Androulakis *et al.*, 2006). In addition, a major D-*seco* tetranortriterpenoid, 3,7-dideacetylkhivorin, isolated from the same extract, exhibited growth inhibitory activities against MCF-7, SiHa, and Caco-2 cells with IC_{50} values in the range of 35–69 µg/ml (Zhang, Wang *et al.*, 2007). In our furthur investigations of the anti-tumor constituents of this African mahogany, another minor tetranortriterpenoid, the title compound, named 14,15β:21,23-diepoxy-1α,3α,7α-trihydroxy-4,4,8-trimethyl-D-*homo*-24-*nor*-17-oxochola-20,22-diene-16-one, was purified from the plant extract for the first time. It was reported as a synthetic derivative by basic hydrolysis of khivorin (Bevan *et al.*, 1963) and isolated as a natural product from *Khaya ivorensis* recently (Abdelgaleil *et al.*, 2005). In our preliminary *in vitro* anti-cancer bioassay, the title compound showed similar bioactivies to those of two other D-*seco* limonoids, 3,7-dideacetyl khivorin and gedunin (Uddin *et al.*, 2007). Herein, the X-ray crystal analysis of the title compound was undertaken to establish its structure and relative stereochemistry, which will benefit further work on structure–activity relationships of anti-tumor bioactivities of D-*seco* limonoids.

Fig. 1 is a thermal ellipsoid plot with atomic labeling of the title compound. The structure contains four six-membered rings A–D, one three-membered ring E, and one furan ring F linked to ring D through a C—C bond in an equatorial position, known as a D-*seco* limonoid. The ring junctions A/B, B/C and C/D are all *trans*, while D/E is *cis*. The six-membered rings A–D adopt chair, chair, twist boat and half-chair conformations, while rings E and F are essentially planar. The five-membered furan ring is disordered by a 180° rotation about the C17—C20 bond.

All the bond lengths and angles are close to their expected values and the data are comparable with the corresponding values in those of gedunin (Toscano *et al.*, 1996) and 3,7-dideacetylkhivorin (Zhang, VanDerveer *et al.*, 2007).

The title compound exhibits strong classical intermolecular O1—H1A···O7, O4—H4···O3 hydrogen bond interactions (Table 1), which link the molecules together to form a stablized network.

Experimental

Air-dried and powdered barks of *Khaya senegalensis* (3.8 kg) were extracted with 95% alcohol at room temperature. The EtOH extract was dried under reduced pressure to yield a crude extract (310 g). The crude extract was suspended in 1000 ml water and partitioned with 2000 ml trichloromethane to yield a $CHCl_3$ fraction (45 g). This part was separated by chromatography on silica gel, eluting with mixtures of hexane and acetone (increasing polarity). The purified powder (30 mg) of the title compound was obtained from the hexane:acetone (3:1) fractions. Transparent rod shaped crystals of the title compound were obtained by recrystallization from 5% $CHCl_3$ in MeOH by slow evaporation at room temperature.

Refinement

Since the most electron-rich atom is oxygen it was not possible to determine the absolute configuation using Mo*K*α radiation. Therefore, Friedel pairs were merged before the final refinement because of the absence of significant anomalous scattering. All H atoms were placed geometrically and allowed to ride on the corresponding non-H atom with C—H = 0.96 Å, O—H = 0.83 Å, and U_{iso}(H) = 1.5U_{eq} of the attached atom for methyl and hydroxyl H atoms and 1.2U_{eq}(C) for other H atoms.

Figures

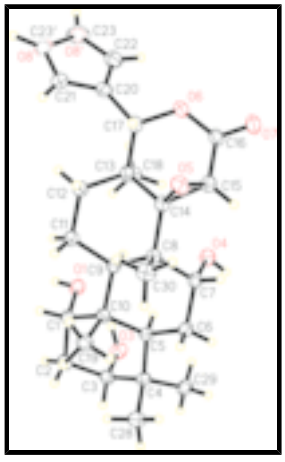


Fig. 1. view of the title compound, showing 50% probability displacement and the atom-numbering scheme

14,15β:21,23-diepoxy-1α,3α,7α-trihydroxy-4,4,8-trimethyl-D-homo-24- nor-17-oxochola-20,22-diene-16-one

Crystal data

$C_{26}H_{36}O_7$	F_{000} = 992
M_r = 460.55	D_x = 1.363 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 8564 reflections
a = 9.856 (2) Å	θ = 4.2–26.4º
b = 14.492 (3) Å	μ = 0.10 mm^{-1}
c = 15.714 (3) Å	*T* = 153 (2) K
V = 2244.5 (8) Å^3	Block, colourless
Z = 4	0.41 × 0.41 × 0.31 mm

Data collection

Riguka Mercury CCD diffractometer	2222 independent reflections
Radiation source: sealed tube	2133 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 153(2) K	θ_{max} = 25.1º
ω scans	θ_{min} = 3.3º

Absorption correction: multi-scan (Rigaku/MSC, 2006)	$h = -8 \rightarrow 11$
$T_{min} = 0.961$, $T_{max} = 0.970$	$k = -17 \rightarrow 17$
16840 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.9576P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.094$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
2222 reflections	Extinction correction: none
306 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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}	Occ. (<1)
O1	0.54360 (19)	0.79446 (11)	0.30374 (11)	0.0253 (4)	
H1A	0.6098	0.8242	0.2860	0.038*	
O3	0.5683 (2)	0.75585 (12)	0.47596 (10)	0.0283 (4)	
H3A	0.5492	0.7884	0.4342	0.042*	
O4	0.10161 (19)	0.74230 (12)	0.34713 (10)	0.0251 (4)	
H4	0.0843	0.7394	0.3987	0.038*	
O5	−0.01398 (18)	0.68425 (11)	0.12115 (11)	0.0248 (4)	
O6	−0.06666 (17)	0.87686 (12)	0.09534 (11)	0.0262 (4)	
O7	−0.22403 (19)	0.85936 (13)	0.19420 (13)	0.0328 (4)	
O8	0.2139 (2)	0.99496 (16)	−0.10650 (13)	0.0320 (5)	0.50
C23'	0.2139 (2)	0.99496 (16)	−0.10650 (13)	0.0320 (5)	0.50
H23'	0.2700	1.0078	−0.1550	0.038*	0.50

C1	0.5671 (3)	0.69684 (16)	0.29037 (15)	0.0222 (5)	
H1	0.6137	0.6891	0.2372	0.027*	
C2	0.6585 (3)	0.66042 (19)	0.36194 (15)	0.0257 (5)	
H2A	0.6850	0.5983	0.3489	0.031*	
H2B	0.7393	0.6973	0.3645	0.031*	
C3	0.5893 (3)	0.66186 (17)	0.44869 (15)	0.0238 (5)	
H3	0.6498	0.6333	0.4887	0.029*	
C4	0.4551 (3)	0.60729 (16)	0.45018 (15)	0.0216 (5)	
C5	0.3648 (2)	0.64328 (15)	0.37633 (14)	0.0184 (5)	
H5	0.3485	0.7070	0.3896	0.022*	
C6	0.2239 (3)	0.59939 (16)	0.37310 (15)	0.0214 (5)	
H6A	0.2321	0.5369	0.3535	0.026*	
H6B	0.1867	0.5978	0.4295	0.026*	
C7	0.1268 (2)	0.65128 (16)	0.31494 (15)	0.0205 (5)	
H7	0.0423	0.6183	0.3130	0.025*	
C8	0.1815 (2)	0.66078 (15)	0.22338 (14)	0.0190 (5)	
C9	0.3285 (2)	0.69989 (16)	0.22822 (14)	0.0192 (5)	
H9	0.3191	0.7586	0.2560	0.023*	
C10	0.4298 (2)	0.64452 (16)	0.28547 (14)	0.0199 (5)	
C11	0.3866 (3)	0.72283 (18)	0.13991 (15)	0.0243 (5)	
H11A	0.4206	0.6670	0.1148	0.029*	
H11B	0.4621	0.7640	0.1470	0.029*	
C12	0.2846 (3)	0.76720 (17)	0.07785 (14)	0.0231 (5)	
H12A	0.3297	0.8149	0.0462	0.028*	
H12B	0.2543	0.7213	0.0381	0.028*	
C13	0.1609 (2)	0.80870 (15)	0.12310 (14)	0.0201 (5)	
C14	0.0922 (2)	0.72936 (16)	0.17152 (14)	0.0196 (5)	
C15	−0.0535 (2)	0.74388 (17)	0.19000 (15)	0.0228 (5)	
H15	−0.0891	0.7148	0.2402	0.027*	
C16	−0.1213 (3)	0.83018 (17)	0.16126 (16)	0.0232 (5)	
C17	0.0590 (2)	0.84230 (16)	0.05511 (15)	0.0224 (5)	
H17	0.0360	0.7910	0.0193	0.027*	
C18	0.1966 (3)	0.88937 (16)	0.18393 (15)	0.0233 (5)	
H18A	0.1428	0.8847	0.2347	0.035*	
H18B	0.1782	0.9470	0.1562	0.035*	
H18C	0.2911	0.8863	0.1986	0.035*	
C19	0.4635 (3)	0.54764 (16)	0.24976 (15)	0.0246 (5)	
H19A	0.5558	0.5324	0.2631	0.037*	
H19B	0.4515	0.5476	0.1891	0.037*	
H19C	0.4040	0.5028	0.2749	0.037*	
C20	0.1080 (3)	0.92041 (16)	−0.00073 (15)	0.0244 (5)	
C21	0.1952 (3)	0.91246 (18)	−0.06903 (16)	0.0278 (6)	
H21	0.2367	0.8559	−0.0873	0.033*	
C22	0.0718 (3)	1.01360 (18)	0.00268 (17)	0.0308 (6)	
H22	0.0115	1.0408	0.0436	0.037*	
C23	0.1346 (3)	1.05895 (17)	−0.06020 (17)	0.0430 (6)	0.50
H23	0.1271	1.1238	−0.0719	0.052*	0.50
O8'	0.1346 (3)	1.05895 (17)	−0.06020 (17)	0.0430 (6)	0.50
C28	0.4863 (3)	0.50310 (17)	0.44778 (17)	0.0293 (6)	

H28A	0.5465	0.4902	0.4013	0.044*
H28B	0.4034	0.4693	0.4403	0.044*
H28C	0.5283	0.4850	0.5003	0.044*
C29	0.3843 (3)	0.62676 (18)	0.53597 (14)	0.0259 (5)
H29A	0.3106	0.5843	0.5436	0.039*
H29B	0.3501	0.6888	0.5362	0.039*
H29C	0.4484	0.6193	0.5815	0.039*
C30	0.1717 (3)	0.56622 (16)	0.17897 (16)	0.0261 (5)
H30A	0.2167	0.5205	0.2129	0.039*
H30B	0.2143	0.5696	0.1241	0.039*
H30C	0.0780	0.5496	0.1722	0.039*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0253 (9)	0.0231 (8)	0.0276 (9)	−0.0041 (7)	0.0017 (8)	0.0018 (7)
O3	0.0336 (11)	0.0306 (9)	0.0208 (8)	−0.0050 (9)	−0.0035 (8)	−0.0011 (8)
O4	0.0292 (9)	0.0268 (8)	0.0193 (8)	0.0045 (8)	0.0042 (8)	−0.0030 (7)
O5	0.0210 (9)	0.0256 (8)	0.0277 (9)	−0.0034 (7)	−0.0056 (8)	−0.0029 (7)
O6	0.0188 (9)	0.0282 (9)	0.0316 (9)	0.0038 (7)	0.0029 (8)	0.0060 (7)
O7	0.0224 (9)	0.0320 (10)	0.0440 (11)	0.0034 (8)	0.0093 (9)	0.0058 (9)
O8	0.0243 (11)	0.0430 (12)	0.0288 (11)	−0.0048 (10)	−0.0028 (9)	0.0132 (10)
C23'	0.0243 (11)	0.0430 (12)	0.0288 (11)	−0.0048 (10)	−0.0028 (9)	0.0132 (10)
C1	0.0195 (12)	0.0271 (12)	0.0202 (11)	0.0031 (10)	0.0009 (10)	−0.0004 (10)
C2	0.0177 (12)	0.0334 (13)	0.0262 (13)	0.0005 (11)	−0.0015 (10)	0.0029 (10)
C3	0.0200 (12)	0.0303 (12)	0.0211 (11)	0.0023 (11)	−0.0024 (10)	0.0019 (10)
C4	0.0205 (12)	0.0237 (11)	0.0207 (11)	0.0004 (10)	−0.0011 (10)	0.0045 (9)
C5	0.0189 (11)	0.0179 (10)	0.0184 (10)	0.0001 (9)	−0.0010 (9)	0.0009 (9)
C6	0.0218 (12)	0.0231 (11)	0.0192 (11)	−0.0017 (10)	0.0000 (10)	0.0022 (10)
C7	0.0180 (11)	0.0206 (11)	0.0228 (11)	−0.0019 (10)	0.0008 (10)	0.0004 (9)
C8	0.0204 (11)	0.0183 (10)	0.0182 (11)	0.0003 (10)	−0.0006 (9)	−0.0006 (9)
C9	0.0208 (12)	0.0200 (10)	0.0169 (11)	0.0021 (10)	0.0011 (9)	−0.0006 (9)
C10	0.0186 (12)	0.0224 (11)	0.0187 (11)	0.0019 (10)	0.0001 (9)	0.0002 (9)
C11	0.0213 (12)	0.0311 (12)	0.0204 (12)	0.0033 (11)	0.0019 (10)	0.0038 (10)
C12	0.0227 (12)	0.0291 (12)	0.0177 (11)	0.0035 (10)	0.0007 (10)	0.0009 (9)
C13	0.0193 (12)	0.0219 (11)	0.0190 (11)	−0.0001 (9)	−0.0001 (10)	0.0006 (9)
C14	0.0189 (11)	0.0215 (11)	0.0183 (11)	−0.0023 (10)	−0.0014 (9)	−0.0022 (9)
C15	0.0205 (12)	0.0244 (12)	0.0236 (11)	−0.0014 (10)	0.0017 (10)	0.0011 (10)
C16	0.0172 (12)	0.0237 (12)	0.0286 (12)	−0.0008 (10)	−0.0003 (11)	0.0003 (10)
C17	0.0187 (12)	0.0250 (11)	0.0235 (11)	0.0008 (10)	0.0017 (10)	0.0004 (10)
C18	0.0253 (13)	0.0213 (11)	0.0233 (11)	−0.0028 (10)	−0.0029 (10)	0.0004 (9)
C19	0.0260 (13)	0.0242 (12)	0.0236 (11)	0.0040 (11)	0.0003 (11)	−0.0004 (10)
C20	0.0194 (12)	0.0294 (12)	0.0245 (12)	0.0003 (10)	−0.0039 (10)	0.0037 (10)
C21	0.0267 (14)	0.0343 (13)	0.0223 (12)	0.0030 (11)	0.0003 (11)	0.0047 (10)
C22	0.0270 (14)	0.0297 (12)	0.0357 (13)	0.0032 (11)	0.0007 (11)	0.0038 (11)
C23	0.0393 (14)	0.0399 (13)	0.0497 (14)	−0.0050 (11)	−0.0100 (13)	0.0178 (12)
O8'	0.0393 (14)	0.0399 (13)	0.0497 (14)	−0.0050 (11)	−0.0100 (13)	0.0178 (12)
C28	0.0302 (13)	0.0280 (13)	0.0295 (13)	0.0052 (11)	−0.0020 (11)	0.0046 (11)

C29	0.0269 (13)	0.0315 (12)	0.0193 (11)	−0.0024 (11)	−0.0009 (10)	0.0035 (10)
C30	0.0305 (14)	0.0212 (11)	0.0266 (12)	0.0002 (11)	−0.0052 (11)	−0.0045 (10)

Geometric parameters (Å, °)

O1—C1	1.449 (3)	C10—C19	1.548 (3)
O1—H1A	0.8299	C11—C12	1.541 (3)
O3—C3	1.443 (3)	C11—H11A	0.9600
O3—H3A	0.8299	C11—H11B	0.9600
O4—C7	1.434 (3)	C12—C13	1.534 (3)
O4—H4	0.8299	C12—H12A	0.9600
O5—C15	1.438 (3)	C12—H12B	0.9600
O5—C14	1.466 (3)	C13—C14	1.536 (3)
O6—C16	1.349 (3)	C13—C17	1.545 (3)
O6—C17	1.478 (3)	C13—C18	1.551 (3)
O7—C16	1.213 (3)	C14—C15	1.480 (3)
O8—C23	1.414 (4)	C15—C16	1.488 (3)
C1—C2	1.534 (3)	C15—H15	0.9600
C1—C10	1.554 (3)	C17—C20	1.511 (3)
C1—H1	0.9600	C17—H17	0.9600
C2—C3	1.524 (3)	C18—H18A	0.9599
C2—H2A	0.9600	C18—H18B	0.9599
C2—H2B	0.9600	C18—H18C	0.9599
C3—C4	1.542 (3)	C19—H19A	0.9599
C3—H3	0.9600	C19—H19B	0.9599
C4—C28	1.541 (3)	C19—H19C	0.9599
C4—C29	1.544 (3)	C20—C21	1.380 (4)
C4—C5	1.553 (3)	C20—C22	1.398 (4)
C5—C6	1.528 (3)	C21—H21	0.9600
C5—C10	1.565 (3)	C22—C23	1.338 (4)
C5—H5	0.9600	C22—H22	0.9600
C6—C7	1.522 (3)	C23—H23	0.9600
C6—H6A	0.9600	C28—H28A	0.9599
C6—H6B	0.9600	C28—H28B	0.9599
C7—C8	1.543 (3)	C28—H28C	0.9599
C7—H7	0.9600	C29—H29A	0.9599
C8—C30	1.541 (3)	C29—H29B	0.9599
C8—C9	1.558 (3)	C29—H29C	0.9599
C8—C14	1.558 (3)	C30—H30A	0.9599
C9—C11	1.538 (3)	C30—H30B	0.9599
C9—C10	1.565 (3)	C30—H30C	0.9599
C9—H9	0.9600		
C1—O1—H1A	109.5	C13—C12—H12A	109.0
C3—O3—H3A	109.5	C11—C12—H12A	109.0
C7—O4—H4	109.5	C13—C12—H12B	109.0
C15—O5—C14	61.25 (15)	C11—C12—H12B	109.0
C16—O6—C17	119.54 (19)	H12A—C12—H12B	107.8
O1—C1—C2	108.9 (2)	C12—C13—C14	106.65 (19)
O1—C1—C10	110.14 (19)	C12—C13—C17	108.64 (19)

C2—C1—C10	112.32 (19)	C14—C13—C17	106.97 (19)
O1—C1—H1	108.5	C12—C13—C18	113.6 (2)
C2—C1—H1	108.5	C14—C13—C18	111.05 (19)
C10—C1—H1	108.5	C17—C13—C18	109.66 (19)
C3—C2—C1	112.9 (2)	O5—C14—C15	58.47 (15)
C3—C2—H2A	109.0	O5—C14—C13	112.40 (18)
C1—C2—H2A	109.0	C15—C14—C13	114.7 (2)
C3—C2—H2B	109.0	O5—C14—C8	113.67 (18)
C1—C2—H2B	109.0	C15—C14—C8	122.4 (2)
H2A—C2—H2B	107.8	C13—C14—C8	119.2 (2)
O3—C3—C2	110.0 (2)	O5—C15—C14	60.28 (15)
O3—C3—C4	110.86 (19)	O5—C15—C16	113.5 (2)
C2—C3—C4	113.0 (2)	C14—C15—C16	119.7 (2)
O3—C3—H3	107.6	O5—C15—H15	117.0
C2—C3—H3	107.6	C14—C15—H15	117.0
C4—C3—H3	107.6	C16—C15—H15	117.0
C28—C4—C3	109.3 (2)	O7—C16—O6	119.1 (2)
C28—C4—C29	106.9 (2)	O7—C16—C15	122.6 (2)
C3—C4—C29	107.9 (2)	O6—C16—C15	118.3 (2)
C28—C4—C5	115.2 (2)	O6—C17—C20	105.21 (19)
C3—C4—C5	107.97 (18)	O6—C17—C13	110.83 (18)
C29—C4—C5	109.42 (19)	C20—C17—C13	115.5 (2)
C6—C5—C4	113.95 (19)	O6—C17—H17	108.4
C6—C5—C10	110.27 (19)	C20—C17—H17	108.4
C4—C5—C10	116.80 (19)	C13—C17—H17	108.4
C6—C5—H5	104.8	C13—C18—H18A	109.5
C4—C5—H5	104.8	C13—C18—H18B	109.5
C10—C5—H5	104.8	H18A—C18—H18B	109.5
C7—C6—C5	112.69 (19)	C13—C18—H18C	109.5
C7—C6—H6A	109.1	H18A—C18—H18C	109.5
C5—C6—H6A	109.1	H18B—C18—H18C	109.5
C7—C6—H6B	109.1	C10—C19—H19A	109.5
C5—C6—H6B	109.1	C10—C19—H19B	109.5
H6A—C6—H6B	107.8	H19A—C19—H19B	109.5
O4—C7—C6	110.56 (19)	C10—C19—H19C	109.5
O4—C7—C8	107.90 (18)	H19A—C19—H19C	109.5
C6—C7—C8	112.57 (19)	H19B—C19—H19C	109.5
O4—C7—H7	108.6	C21—C20—C22	105.6 (2)
C6—C7—H7	108.6	C21—C20—C17	126.0 (2)
C8—C7—H7	108.6	C22—C20—C17	128.3 (2)
C30—C8—C7	108.73 (19)	C20—C21—H21	124.7
C30—C8—C9	113.84 (19)	C23—C22—C20	109.2 (3)
C7—C8—C9	108.21 (18)	C23—C22—H22	125.4
C30—C8—C14	107.14 (19)	C20—C22—H22	125.4
C7—C8—C14	110.29 (19)	C22—C23—O8	108.3 (2)
C9—C8—C14	108.62 (18)	C22—C23—H23	125.9
C11—C9—C8	112.40 (19)	O8—C23—H23	125.9
C11—C9—C10	113.09 (19)	C4—C28—H28A	109.5
C8—C9—C10	115.76 (18)	C4—C28—H28B	109.5

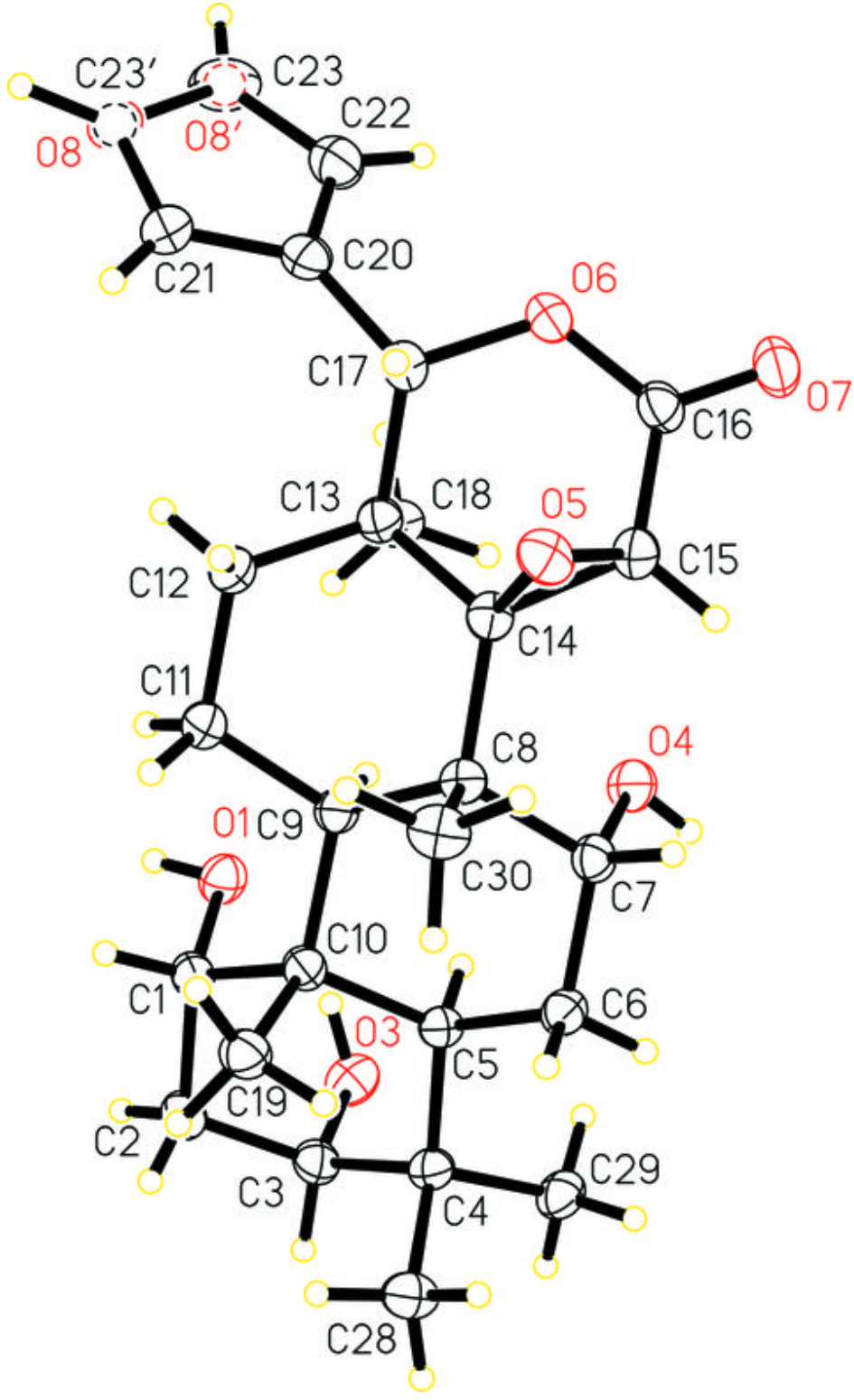
C11—C9—H9	104.7	H28A—C28—H28B	109.5
C8—C9—H9	104.7	C4—C28—H28C	109.5
C10—C9—H9	104.7	H28A—C28—H28C	109.5
C19—C10—C1	105.88 (18)	H28B—C28—H28C	109.5
C19—C10—C9	113.17 (18)	C4—C29—H29A	109.5
C1—C10—C9	109.50 (18)	C4—C29—H29B	109.5
C19—C10—C5	114.09 (18)	H29A—C29—H29B	109.5
C1—C10—C5	108.49 (18)	C4—C29—H29C	109.5
C9—C10—C5	105.62 (18)	H29A—C29—H29C	109.5
C9—C11—C12	114.7 (2)	H29B—C29—H29C	109.5
C9—C11—H11A	108.6	C8—C30—H30A	109.5
C12—C11—H11A	108.6	C8—C30—H30B	109.5
C9—C11—H11B	108.6	H30A—C30—H30B	109.5
C12—C11—H11B	108.6	C8—C30—H30C	109.5
H11A—C11—H11B	107.6	H30A—C30—H30C	109.5
C13—C12—C11	112.88 (19)	H30B—C30—H30C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O7^{i}	0.83	2.24	3.015 (3)	155
O4—H4···O3^{ii}	0.83	1.98	2.800 (2)	171

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

Fig. 1


C23′
C23
C22
O8
O8′
C21
C20
O6
C17
O7
C16
C13
C18
O5
C15
C12
C14
C11
C8
O4
O1
C9
C30
C7
C10
C1
C6
O3
C5
C19
C2
C29
C3
C4
C28