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3,7-Dideacetylkhivorin

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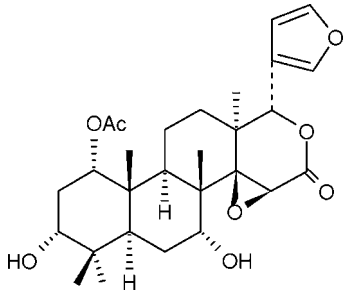
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.108; data-to-parameter ratio = 8.1.

The title compound, a D-*seco* limonoid (systematic name: 1α-acetyloxy-14,15β:21,23-diepoxy-3α,7α-dihydroxy-4,4,8-trimethyl-D-*homo*-24-*nor*-17-oxochola-20,22-dien-16-one), $C_{28}H_{38}O_8$, was isolated from the stem bark of African mahogany *Khaya senegalensis* (Meliaceae). The six-membered rings show chair, boat and half-chair conformations, while the furan ring is planar. The crystal packing is stabilized by both intra- and intermolecular O—H···O hydrogen bonds.

Related literature

Geometry: Allen *et al.* (1987); Background: Androulakis *et al.* (2006); Iwu (1993); Olayinka *et al.* (1992). Isolation, NMR and MS data, and biological activity of the title compound: Abdelgaleil *et al.* (2005); Adesida *et al.* (1971); Govindachari & Kumari (1998); Zhang *et al.* (2007). Similar D-*seco* limonoids (gedunin and 7-oxogedunin): Toscano *et al.* (1996); Waratchareeyakul *et al.* (2004).



Experimental

Crystal data

$C_{28}H_{38}O_8$
M_r = 502.58
Orthorhombic, $P2_12_12_1$
a = 10.907 (2) Å
b = 14.200 (3) Å
c = 17.391 (4) Å
V = 2693.5 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.48 × 0.46 × 0.29 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*REQAB*; Rigaku/MSC, 2006)
T_{min} = 0.958, T_{max} = 0.974
23488 measured reflections
2711 independent reflections
2485 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.07
2711 reflections
333 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1	0.83	2.10	2.810 (2)	143
O4—H4···O3^{i}	0.83	1.93	2.758 (2)	171

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z$.

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); 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: FL2161).

References

Abdelgaleil, S. A. M., Hashinaga, F. & Nakatani, M. (2005). *Pest Manag. Sci.* **61**, 186–190.
Adesida, G. A., Adesogan, E. K., Okorie, D. A., Taylor, D. A. H. & Styles, B. T. (1971). *Phytochemsitry*, **10**, 1845–1853.
Allen, F. H., Kennard, O., Waston, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Androulakis, X. M., Muga, S. J., Chen, F., Koita, Y., Toure, B. & Wargovich, M. J. (2006). *Anticancer Res.* **26**, 2397–2405.
Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Govindachari, T. R. & Kumari, G. N. K. (1998). *Phytochemistry*, **47**, 1423–1425.
Iwu, M. (1993). *Handbook of African Medicinal Plants*, *Pharmacognostical Profile of Selected Medicinal Plants*, pp. 196–197. Boca Raton, Florida: CRC Press Inc.
Olayinka, A. O., Onoruvwe, O. & Loti, T. Y. (1992). *Phytother. Res.* **6**, 282–284.
Rigaku/MSC (2006). *REQAB* and *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Toscano, R. A., Mata, R., Calderon, J. & Segura, R. (1996). *J. Chem. Crystallogr.* **26**, 707–711.
Waratchareeyakul, W., Chantrapromma, S., Fun, H.-K., Razak, I. A., Karalai, C. & Ponglimanont, C. (2004). *Acta Cryst.* E**60**, o1964–o1966.
Zhang, H. P., Wang, X., Chen, F., Androulakis, X. M. & Wargovich, M. J. (2007). *Phytother. Res.* **8**, 731–734.

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supplementary materials

Acta Cryst. (2007). E**63**, o4162 [**doi:10.1107/S1600536807046363**]

3,7-Dideacetylkhivorin

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Comment

African mahogany *Khaya senegalensis* is a large evergreen tree growing mainly in the sub-Saharan savannah forests from Senegal to Uganda (Adesida *et al.*, 1971). The plant is one of the most popular medicinal meliaceous plants used in African traditional remedies. It is used as a bitter tonic, folk and popular medicine against malaria, fever, mucous diarrhea, and venereal diseases as well as an anthelmintic and a taeniacide remedy (Olayinka *et al.*, 1992; Iwu, 1993). Our earlier study showed that its stem bark extract displayed anti-proliferative, anti-inflammatory and pro-apoptotic effects on HT-29, HCT-15, HCA-7 cells (Androulakis *et al.*, 2006). In the course of our chemical investigation of the plant bark, the title compound, was isolated as a major product from the $CHCl_3$ extract. This D-*seco* limonoid was originally isolated from the seeds of *Khaya nyasica* in Tanzania (Adesida *et al.*, 1971) and was also found to be present in the fresh seeds of *Khaya senegalensis* (Govindachari & Kumari, 1998) in India and in the stem bark of *khaya ivorensis* in the Democratic Republic of Congo (Abdelgaleil *et al.*, 2005). It was reported that this tetranortriterpenoid displayed moderate antifungal and antibacterial activities (Abdelgaleil *et al.*, 2005). Also, the title compound showed growth inhibitory activities against different cell lines in our preliminary anti-cancer bioassay (Zhang *et al.*, 2007). Although its structure was tentatively identified, no spectral data were presented in previous publications. Considering its biological importanc, we have undertaken the X-ray crystal analysis of this limonoid in order to firmly establish its structure and relative stereochemistry.

The title compound (Fig.1) contains four six-membered rings A—D, one three-membered ring E (C14/C15/O5), and one furan ring F linked to atom C17 of ring D through a C—C bond in an equatorial position, known as a D-*seco* type 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 fairly planar moieties. The bond lengths and angles are with normal ranges (Allen *et al.*, 1987), fairly close to their expected values and the data are comparable with the corresponding values in those of two similar D-*seco* limonoids: gedunin (Toscano *et al.*, 1996) and 7-oxogedunin (Waratchareeyakul *et al.*, 2004). The strong classical intra-molecular hydrogen bond, O3—H3···O1 and inter-molecular hydrogen-bonding interactions O4—H4···O3 (Table 1) connect the molecules together to form a network.

Experimental

The plant stem bark was collected from the martime plains near Conakry, Republic of Guinea (west Africa) in November 2005. Air-dried and powdered raw material (495 g) were extracted by 5*L* methanol using a Soxhlet extrator. The MeOH extract was dried under reduced pressure to yield a crude extract (51 g). The crude extract was suspended in 300 ml water and partitioned with 400 ml trichloromethane three times to yield $CHCl_3$ fraction (5 g). This part was separated by conventional chromatography on silica gel, eluting with mixtures of hexane and acetone (increasing polarity). The purified powder (32 mg) of the title compound was obtained from the hexane:acetone = 4:1 fractions. Transparent rod shaped crystals of the title compound were suitable for X-ray diffraction after recrystallization from a solution of 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 configuration. Therefore, Friedel reflections were merged before final refinement because of the absence of significant anomalous scattering effects. All H atoms were geometrically fixed 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}(C) of the attached C atom for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Figures

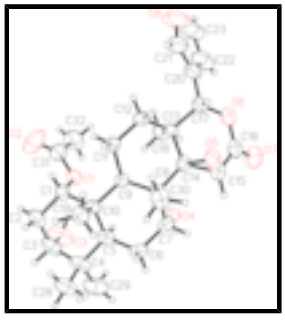


Fig. 1. view of the title compound, showing 50% probability displacement and the atom-numbering scheme

1α-acetyloxy-14,15β:21,23-diepoxy-3α,7α-dihydroxy-4,4,8-trimethyl-D-\ *homo*-24-*nor*-17-oxochola-20,22-dien-16-one

Crystal data

$C_{28}H_{38}O_8$	D_x = 1.239 Mg m^{-3}
M_r = 502.58	Melting point: _audit_update_record _audit_creation_method 'SHELXL-97' _chemical_name_systematic K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.7107 Å
a = 10.907 (2) Å	Cell parameters from 12261 reflections
b = 14.200 (3) Å	θ = 1.9–25.7º
c = 17.391 (4) Å	μ = 0.09 mm^{-1}
V = 2693.5 (9) Å^3	T = 298 (2) K
Z = 4	Rod, colourless
F_{000} = 1080	0.48 × 0.46 × 0.29 mm

Data collection

Rigaku Mercury CCD (2x2 bin mode) diffractometer	2711 independent reflections
Radiation source: Sealed Tube	2485 reflections with $I > 2\sigma(I)$
Monochromator: Graphite Monochromator	R_{int} = 0.030
Detector resolution: 14.6199 pixels mm^{-1}	θ_{max} = 25.1º
T = 298(2) K	θ_{min} = 2.2º
ω scans	h = −12→13
Absorption correction: multi-scan	k = −16→16

(REQAB; Rigaku/MSC, 2006)
T_{min} = 0.958, T_{max} = 0.974
23488 measured reflections

l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.07
2711 reflections
333 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
$w = 1/[s^2(F_o^2) + (0.063P)^2 + 0.4442P]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 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^ > 2sigma(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}
O1	−0.24761 (17)	0.03344 (13)	0.00185 (10)	0.0417 (4)
O2	−0.4201 (3)	−0.0522 (2)	0.00453 (17)	0.0943 (10)
O3	−0.2722 (3)	0.22356 (16)	−0.04014 (13)	0.0752 (8)
H3	−0.2628	0.1805	−0.0084	0.090*
O4	0.1384 (2)	0.09520 (13)	0.03037 (11)	0.0482 (5)
H4	0.1596	0.1513	0.0307	0.058*
O5	0.27970 (17)	−0.15088 (15)	0.01582 (12)	0.0533 (5)
O6	0.25238 (19)	−0.16923 (15)	0.17687 (12)	0.0583 (5)
O7	0.3671 (2)	−0.0430 (2)	0.18949 (16)	0.0853 (8)
O8	−0.0200 (3)	−0.3910 (3)	0.2456 (2)	0.1073 (11)
C1	−0.2334 (2)	0.0143 (2)	−0.08101 (13)	0.0390 (6)
H1	−0.2682	−0.0462	−0.0926	0.047*
C2	−0.3041 (3)	0.0904 (2)	−0.12480 (17)	0.0533 (7)
H2A	−0.3063	0.0739	−0.1783	0.064*
H2B	−0.3871	0.0916	−0.1064	0.064*

C3	−0.2496 (3)	0.1879 (2)	−0.11678 (17)	0.0593 (8)
H3A	−0.2930	0.2281	−0.1518	0.071*
C4	−0.1123 (3)	0.1934 (2)	−0.13845 (16)	0.0535 (7)
C5	−0.0427 (3)	0.11459 (17)	−0.09381 (15)	0.0402 (6)
H5	−0.0529	0.1310	−0.0407	0.048*
C6	0.0963 (3)	0.11405 (18)	−0.10582 (16)	0.0441 (6)
H6A	0.1147	0.0883	−0.1555	0.053*
H6B	0.1266	0.1775	−0.1044	0.053*
C7	0.1605 (2)	0.05603 (18)	−0.04411 (15)	0.0409 (6)
H7	0.2471	0.0569	−0.0539	0.049*
C8	0.1167 (2)	−0.04691 (16)	−0.04239 (13)	0.0327 (5)
C9	−0.0258 (2)	−0.04659 (15)	−0.03695 (13)	0.0305 (5)
H9	−0.0433	−0.0148	0.0105	0.037*
C10	−0.0948 (2)	0.01254 (16)	−0.09987 (13)	0.0327 (5)
C11	−0.0774 (2)	−0.14678 (17)	−0.02572 (16)	0.0401 (6)
H11A	−0.1611	−0.1420	−0.0091	0.048*
H11B	−0.0772	−0.1785	−0.0745	0.048*
C12	−0.0064 (3)	−0.20695 (17)	0.03248 (16)	0.0446 (6)
H12A	−0.0642	−0.2399	0.0643	0.053*
H12B	0.0404	−0.2532	0.0050	0.053*
C13	0.0805 (2)	−0.15048 (17)	0.08430 (13)	0.0346 (5)
C14	0.1681 (2)	−0.09790 (16)	0.03037 (14)	0.0346 (5)
C15	0.2863 (3)	−0.0707 (2)	0.06512 (18)	0.0507 (7)
H15	0.3256	−0.0155	0.0449	0.061*
C16	0.3058 (3)	−0.0916 (3)	0.14789 (19)	0.0581 (8)
C17	0.1596 (2)	−0.21977 (19)	0.13252 (16)	0.0449 (6)
H17	0.2003	−0.2623	0.0980	0.054*
C18	0.0126 (3)	−0.0847 (2)	0.14002 (15)	0.0453 (6)
H18A	0.0710	−0.0529	0.1721	0.068*
H18B	−0.0421	−0.1210	0.1715	0.068*
H18C	−0.0336	−0.0392	0.1113	0.068*
C19	−0.0872 (3)	−0.03207 (19)	−0.18109 (14)	0.0437 (6)
H19A	−0.0809	−0.0993	−0.1765	0.066*
H19B	−0.0163	−0.0082	−0.2074	0.066*
H19C	−0.1596	−0.0164	−0.2097	0.066*
C20	0.0914 (3)	−0.2766 (2)	0.19140 (18)	0.0543 (7)
C21	0.0267 (4)	−0.3566 (3)	0.1786 (2)	0.0758 (10)
H21	0.0153	−0.3851	0.1290	0.091*
C22	0.0852 (4)	−0.2612 (3)	0.2719 (2)	0.0794 (11)
H22	0.1235	−0.2106	0.2996	0.095*
C23	0.0167 (5)	−0.3296 (3)	0.3024 (3)	0.0912 (14)
H23	−0.0040	−0.3351	0.3558	0.109*
C28	−0.1018 (4)	0.1874 (3)	−0.22687 (18)	0.0718 (10)
H28A	−0.1493	0.1350	−0.2452	0.108*
H28B	−0.0175	0.1788	−0.2410	0.108*
H28C	−0.1322	0.2445	−0.2494	0.108*
C29	−0.0636 (4)	0.2901 (2)	−0.1140 (3)	0.0825 (12)
H29A	−0.1159	0.3385	−0.1342	0.124*
H29B	0.0180	0.2984	−0.1336	0.124*

H29C	−0.0623	0.2940	−0.0589	0.124*
C30	0.1665 (3)	−0.0979 (2)	−0.11413 (16)	0.0493 (7)
H30A	0.1459	−0.0624	−0.1593	0.074*
H30B	0.1307	−0.1595	−0.1175	0.074*
H30C	0.2540	−0.1035	−0.1103	0.074*
C31	−0.3440 (3)	−0.0070 (2)	0.03741 (18)	0.0533 (7)
C32	−0.3406 (3)	0.0131 (3)	0.12206 (19)	0.0743 (11)
H32A	−0.4218	0.0076	0.1429	0.112*
H32B	−0.3104	0.0757	0.1304	0.112*
H32C	−0.2875	−0.0314	0.1470	0.112*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0403 (9)	0.0501 (10)	0.0347 (8)	0.0061 (8)	0.0047 (8)	0.0034 (8)
O2	0.0707 (16)	0.131 (3)	0.0810 (18)	−0.0390 (18)	0.0204 (15)	−0.0125 (18)
O3	0.114 (2)	0.0517 (12)	0.0600 (13)	0.0383 (14)	0.0212 (14)	0.0016 (11)
O4	0.0665 (12)	0.0353 (9)	0.0427 (10)	−0.0147 (9)	−0.0038 (9)	−0.0048 (8)
O5	0.0383 (10)	0.0660 (13)	0.0555 (11)	0.0188 (9)	0.0077 (9)	0.0045 (10)
O6	0.0500 (11)	0.0700 (13)	0.0549 (11)	−0.0003 (10)	−0.0148 (10)	0.0165 (11)
O7	0.0725 (16)	0.108 (2)	0.0753 (16)	−0.0286 (16)	−0.0363 (14)	0.0087 (16)
O8	0.104 (2)	0.095 (2)	0.123 (3)	0.0002 (19)	0.026 (2)	0.061 (2)
C1	0.0392 (14)	0.0467 (14)	0.0312 (12)	0.0056 (11)	−0.0009 (10)	0.0004 (10)
C2	0.0527 (16)	0.0635 (18)	0.0438 (15)	0.0192 (15)	−0.0016 (13)	0.0097 (14)
C3	0.080 (2)	0.0537 (16)	0.0441 (15)	0.0302 (16)	0.0029 (15)	0.0086 (13)
C4	0.078 (2)	0.0381 (14)	0.0449 (15)	0.0115 (14)	0.0049 (15)	0.0105 (12)
C5	0.0540 (15)	0.0334 (12)	0.0330 (12)	0.0024 (11)	0.0043 (12)	0.0007 (10)
C6	0.0564 (16)	0.0353 (13)	0.0405 (14)	−0.0098 (12)	0.0065 (12)	0.0044 (11)
C7	0.0410 (13)	0.0412 (13)	0.0404 (13)	−0.0087 (11)	0.0057 (11)	0.0012 (11)
C8	0.0337 (11)	0.0320 (11)	0.0325 (11)	−0.0017 (10)	0.0035 (10)	−0.0029 (10)
C9	0.0353 (11)	0.0276 (11)	0.0286 (11)	−0.0011 (9)	−0.0006 (9)	−0.0025 (9)
C10	0.0373 (12)	0.0324 (12)	0.0285 (11)	0.0010 (10)	0.0009 (10)	0.0001 (9)
C11	0.0430 (13)	0.0310 (11)	0.0464 (14)	−0.0070 (10)	−0.0102 (11)	0.0009 (11)
C12	0.0486 (14)	0.0288 (12)	0.0563 (15)	−0.0069 (11)	−0.0076 (13)	0.0050 (11)
C13	0.0360 (12)	0.0314 (11)	0.0364 (12)	0.0026 (10)	0.0007 (10)	0.0019 (10)
C14	0.0307 (11)	0.0331 (11)	0.0400 (13)	0.0042 (10)	0.0025 (10)	−0.0043 (10)
C15	0.0364 (13)	0.0583 (17)	0.0575 (17)	−0.0052 (13)	−0.0058 (12)	0.0104 (15)
C16	0.0426 (15)	0.071 (2)	0.0601 (18)	−0.0019 (15)	−0.0149 (14)	0.0086 (16)
C17	0.0429 (13)	0.0433 (13)	0.0486 (15)	0.0090 (12)	−0.0014 (12)	0.0045 (12)
C18	0.0501 (15)	0.0463 (14)	0.0395 (13)	0.0143 (13)	0.0052 (12)	0.0036 (11)
C19	0.0525 (15)	0.0477 (14)	0.0310 (12)	0.0035 (12)	−0.0020 (12)	−0.0058 (11)
C20	0.0543 (16)	0.0491 (16)	0.0596 (18)	0.0119 (13)	0.0040 (14)	0.0208 (14)
C21	0.089 (3)	0.062 (2)	0.076 (2)	−0.0034 (19)	0.007 (2)	0.0257 (19)
C22	0.100 (3)	0.079 (2)	0.059 (2)	0.013 (2)	0.010 (2)	0.0194 (19)
C23	0.112 (3)	0.091 (3)	0.070 (2)	0.023 (3)	0.031 (3)	0.039 (2)
C28	0.098 (3)	0.068 (2)	0.0496 (17)	0.017 (2)	0.0084 (18)	0.0247 (16)
C29	0.115 (3)	0.0352 (15)	0.097 (3)	0.0076 (19)	0.006 (3)	0.0101 (17)
C30	0.0500 (16)	0.0561 (16)	0.0418 (14)	0.0093 (14)	0.0078 (12)	−0.0101 (13)

C31	0.0432 (15)	0.0650 (18)	0.0519 (16)	0.0034 (14)	0.0122 (14)	0.0070 (15)
C32	0.066 (2)	0.107 (3)	0.0494 (17)	0.013 (2)	0.0184 (16)	0.0155 (19)

Geometric parameters (Å, °)

O1—C31	1.348 (3)	C11—C12	1.534 (4)
O1—C1	1.475 (3)	C11—H11A	0.9600
O2—C31	1.195 (4)	C11—H11B	0.9600
O3—C3	1.447 (4)	C12—C13	1.534 (4)
O3—H3	0.8299	C12—H12A	0.9600
O4—C7	1.430 (3)	C12—H12B	0.9600
O4—H4	0.8299	C13—C14	1.534 (3)
O5—C15	1.427 (4)	C13—C18	1.536 (3)
O5—C14	1.453 (3)	C13—C17	1.554 (3)
O6—C16	1.345 (4)	C14—C15	1.475 (4)
O6—C17	1.461 (3)	C15—C16	1.485 (4)
O7—C16	1.203 (4)	C15—H15	0.9600
O8—C21	1.363 (5)	C17—C20	1.501 (4)
O8—C23	1.376 (6)	C17—H17	0.9600
C1—C2	1.530 (4)	C18—H18A	0.9599
C1—C10	1.547 (3)	C18—H18B	0.9599
C1—H1	0.9600	C18—H18C	0.9599
C2—C3	1.513 (5)	C19—H19A	0.9599
C2—H2A	0.9600	C19—H19B	0.9599
C2—H2B	0.9600	C19—H19C	0.9599
C3—C4	1.546 (5)	C20—C21	1.356 (5)
C3—H3A	0.9600	C20—C22	1.419 (5)
C4—C29	1.533 (5)	C21—H21	0.9600
C4—C28	1.544 (4)	C22—C23	1.335 (6)
C4—C5	1.560 (4)	C22—H22	0.9600
C5—C6	1.530 (4)	C23—H23	0.9600
C5—C10	1.560 (3)	C28—H28A	0.9599
C5—H5	0.9600	C28—H28B	0.9599
C6—C7	1.523 (4)	C28—H28C	0.9599
C6—H6A	0.9600	C29—H29A	0.9599
C6—H6B	0.9600	C29—H29B	0.9599
C7—C8	1.538 (3)	C29—H29C	0.9599
C7—H7	0.9600	C30—H30A	0.9599
C8—C30	1.541 (3)	C30—H30B	0.9599
C8—C9	1.558 (3)	C30—H30C	0.9599
C8—C14	1.562 (3)	C31—C32	1.500 (5)
C9—C11	1.542 (3)	C32—H32A	0.9599
C9—C10	1.571 (3)	C32—H32B	0.9599
C9—H9	0.9600	C32—H32C	0.9599
C10—C19	1.550 (3)		
C31—O1—C1	116.9 (2)	C14—C13—C12	106.3 (2)
C3—O3—H3	109.5	C14—C13—C18	113.0 (2)
C7—O4—H4	109.5	C12—C13—C18	113.0 (2)
C15—O5—C14	61.59 (16)	C14—C13—C17	107.0 (2)

C16—O6—C17	120.3 (2)	C12—C13—C17	109.2 (2)
C21—O8—C23	106.2 (3)	C18—C13—C17	108.2 (2)
O1—C1—C2	107.6 (2)	O5—C14—C15	58.35 (18)
O1—C1—C10	108.2 (2)	O5—C14—C13	112.13 (19)
C2—C1—C10	113.5 (2)	C15—C14—C13	114.9 (2)
O1—C1—H1	109.1	O5—C14—C8	113.55 (19)
C2—C1—H1	109.1	C15—C14—C8	121.6 (2)
C10—C1—H1	109.1	C13—C14—C8	119.83 (19)
C3—C2—C1	113.7 (2)	O5—C15—C14	60.06 (16)
C3—C2—H2A	108.8	O5—C15—C16	115.5 (3)
C1—C2—H2A	108.8	C14—C15—C16	118.0 (3)
C3—C2—H2B	108.8	O5—C15—H15	117.0
C1—C2—H2B	108.8	C14—C15—H15	117.0
H2A—C2—H2B	107.7	C16—C15—H15	117.0
O3—C3—C2	109.8 (3)	O7—C16—O6	119.0 (3)
O3—C3—C4	111.8 (3)	O7—C16—C15	123.2 (3)
C2—C3—C4	113.9 (2)	O6—C16—C15	117.7 (3)
O3—C3—H3A	107.0	O6—C17—C20	104.3 (2)
C2—C3—H3A	107.0	O6—C17—C13	111.0 (2)
C4—C3—H3A	107.0	C20—C17—C13	115.7 (2)
C29—C4—C28	107.5 (3)	O6—C17—H17	108.5
C29—C4—C3	108.3 (3)	C20—C17—H17	108.5
C28—C4—C3	108.2 (3)	C13—C17—H17	108.5
C29—C4—C5	109.6 (3)	C13—C18—H18A	109.5
C28—C4—C5	114.8 (3)	C13—C18—H18B	109.5
C3—C4—C5	108.3 (2)	H18A—C18—H18B	109.5
C6—C5—C4	114.7 (2)	C13—C18—H18C	109.5
C6—C5—C10	110.3 (2)	H18A—C18—H18C	109.5
C4—C5—C10	117.1 (2)	H18B—C18—H18C	109.5
C6—C5—H5	104.4	C10—C19—H19A	109.5
C4—C5—H5	104.4	C10—C19—H19B	109.5
C10—C5—H5	104.4	H19A—C19—H19B	109.5
C7—C6—C5	111.2 (2)	C10—C19—H19C	109.5
C7—C6—H6A	109.4	H19A—C19—H19C	109.5
C5—C6—H6A	109.4	H19B—C19—H19C	109.5
C7—C6—H6B	109.4	C21—C20—C22	105.5 (3)
C5—C6—H6B	109.4	C21—C20—C17	126.6 (3)
H6A—C6—H6B	108.0	C22—C20—C17	127.8 (3)
O4—C7—C6	110.5 (2)	C20—C21—O8	110.7 (4)
O4—C7—C8	107.44 (19)	C20—C21—H21	124.7
C6—C7—C8	112.6 (2)	O8—C21—H21	124.7
O4—C7—H7	108.7	C23—C22—C20	107.8 (4)
C6—C7—H7	108.7	C23—C22—H22	126.1
C8—C7—H7	108.7	C20—C22—H22	126.1
C7—C8—C30	108.7 (2)	C22—C23—O8	109.8 (4)
C7—C8—C9	108.0 (2)	C22—C23—H23	125.1
C30—C8—C9	113.7 (2)	O8—C23—H23	125.1
C7—C8—C14	110.2 (2)	C4—C28—H28A	109.5
C30—C8—C14	108.2 (2)	C4—C28—H28B	109.5

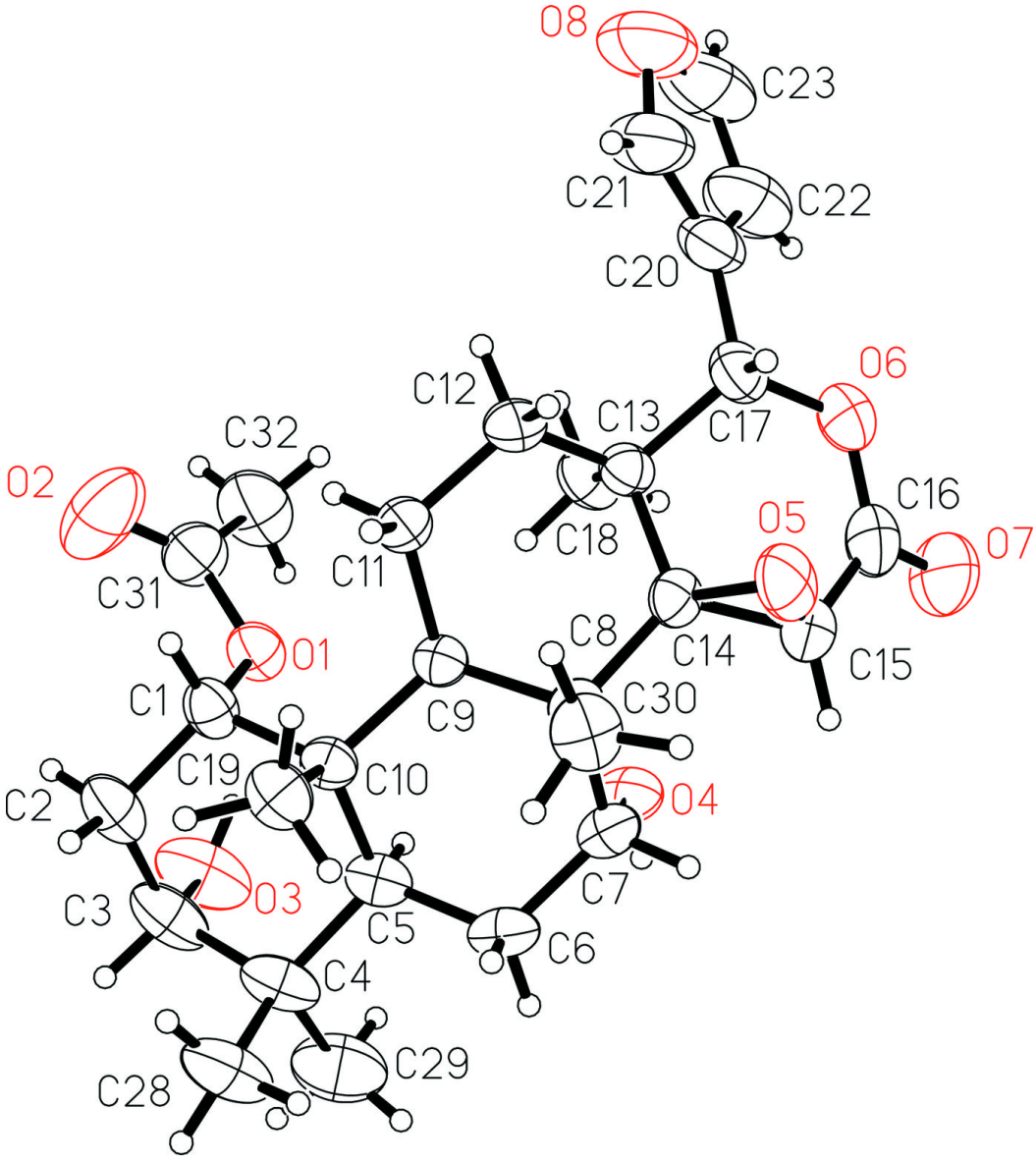
C9—C8—C14	108.09 (19)	H28A—C28—H28B	109.5
C11—C9—C8	111.68 (19)	C4—C28—H28C	109.5
C11—C9—C10	113.97 (19)	H28A—C28—H28C	109.5
C8—C9—C10	115.93 (19)	H28B—C28—H28C	109.5
C11—C9—H9	104.6	C4—C29—H29A	109.5
C8—C9—H9	104.6	C4—C29—H29B	109.5
C10—C9—H9	104.6	H29A—C29—H29B	109.5
C1—C10—C19	104.6 (2)	C4—C29—H29C	109.5
C1—C10—C5	109.1 (2)	H29A—C29—H29C	109.5
C19—C10—C5	114.9 (2)	H29B—C29—H29C	109.5
C1—C10—C9	109.20 (19)	C8—C30—H30A	109.5
C19—C10—C9	113.00 (19)	C8—C30—H30B	109.5
C5—C10—C9	105.96 (19)	H30A—C30—H30B	109.5
C12—C11—C9	114.4 (2)	C8—C30—H30C	109.5
C12—C11—H11A	108.7	H30A—C30—H30C	109.5
C9—C11—H11A	108.7	H30B—C30—H30C	109.5
C12—C11—H11B	108.7	O2—C31—O1	123.4 (3)
C9—C11—H11B	108.7	O2—C31—C32	126.1 (3)
H11A—C11—H11B	107.6	O1—C31—C32	110.5 (3)
C13—C12—C11	114.10 (19)	C31—C32—H32A	109.5
C13—C12—H12A	108.7	C31—C32—H32B	109.5
C11—C12—H12A	108.7	H32A—C32—H32B	109.5
C13—C12—H12B	108.7	C31—C32—H32C	109.5
C11—C12—H12B	108.7	H32A—C32—H32C	109.5
H12A—C12—H12B	107.6	H32B—C32—H32C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1	0.83	2.10	2.810 (2)	143
O4—H4···O3^{i}	0.83	1.93	2.758 (2)	171

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

Fig. 1


O8
C23
C21
C22
C20
C12
C13
C17
O6
C32
O2
C16
C18
O5
O7
C31
C11
C8
C14
O1
C15
C1
C9
C30
C19
C10
O4
C2
C7
O3
C3
C5
C6
C4
C29
C28