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6α-Acetoxyepoxyazadiradione

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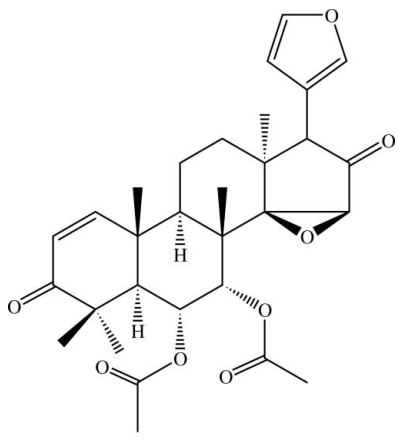
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.006 Å; R factor = 0.051; wR factor = 0.125; data-to-parameter ratio = 7.8.

The title limonoid compound [systematic name: (8*S**,10*R**)-17-(3-furyl)-4,4,8,10,13-pentamethyl-3,16-dioxo-6,7,9,11,12,17-hexahydro-14,15-epoxy-5*H*-cyclopenta[*a*]phenanthrene-6α,7α-diyl diacetate], $C_{30}H_{36}O_8$, was isolated from *Chisocheton siamensis*. The molecule contains four *trans*-fused rings. The cyclohexene ring adopts a screw-boat conformation, the two cyclohexane rings are in standard chair and boat conformations, and the cyclopentane ring is in an envelope conformation. Weak intramolecular C—H···O interactions generate S(5) and S(6) ring motifs. A weak C—H···O intermolecular interaction connects the molecules into dimers.

Related literature

For hydrogen-bond motifs, see Bernstein *et al.* (1995). For values of bond lengths, see Allen *et al.* (1987). For ring conformations, see Cremer & Pople (1975). For limonoid compounds and activities, see, for example, Bickii *et al.* (2000); Khalid *et al.* (1998); Koul *et al.* (2003); Nihei *et al.* (2002); Penido, Conte *et al.* (2006); Penido, Costa *et al.* (2006); Saewan *et al.* (2006); Takeya *et al.* (1996).



Experimental

Crystal data

$C_{30}H_{36}O_8$
M_r = 524.59
Orthorhombic, $P2_12_12$
a = 12.189 (3) Å
b = 29.983 (5) Å
c = 7.3372 (11) Å
V = 2681.5 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 297 (2) K
0.39 × 0.10 × 0.07 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.965, T_{max} = 0.994
20575 measured reflections
2724 independent reflections
1782 reflections with $I > 2\sigma(I)$
R_{int} = 0.100

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.125
S = 1.02
2724 reflections
351 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O7	0.98	2.45	2.900 (5)	107
C7—H7A···O8	0.98	2.31	2.705 (5)	103
C9—H9A···O7	0.98	2.54	2.936 (5)	104
C27—H27A···O2^{i}	0.96	2.51	3.422 (7)	160
C28—H28D···O5	0.96	2.53	3.121 (6)	120
C29—H29D···O5	0.96	2.29	2.973 (5)	127

Symmetry code: (i) $-x, -y, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2461).

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supplementary materials

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6α-Acetoxyepoxyazadiradione

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Comment

Meliaceae plants are known to be rich sources of limonoids. Some of these limonoids posses significant pharmocological effects such as antimalarial (Bickii *et al.*, 2000; Saewan *et al.*, 2006), cytotoxic (Takeya *et al.*, 1996), antiprotozoal (Khalid *et al.*, 1998) and antifeedant activities (Koul *et al.*, 2003; Nihei *et al.*, 2002). *Chisocheton siamensis* or "Ta Sua" in a local Thai name, is one of the Meliaceae plants, which is found in the northern part of Thailand. As part of our study of chemical constituents and bioactive compounds from *C. siamensis*, we herein report the crystal structure of the title compound which was isolated for the first time from the seed of *C. siamensis* collected from Chiang Mai province in the northern part of Thailand. The title compound was found to posses anti-allergic (Penido, Costa *et al.*, (2006) and antiinflammatory (Penido, Conte *et al.*, (2006) activities.

The title molecule (Fig. 1) contains a four *trans*-fused rings, the cyclohexene ring adopts a screw boat conformation with the puckering parameters (Cremer & Pople, 1975) Q = 0.552 (4) Å, θ = 70.6 (5)° and φ = 276.7 (5)°, the two cyclohexane rings are in standard chair and boat conformations and the cyclopentane adopts an envelope conformation, with atom C17 displaced from the C13/C14/C15/C16 plane by 0.238 (4) Å. The oxirane moiety (C14/C15/O3) makes the dihedral angle of 78.0 (4)° with the mean plane of C13/C14/C15/C16. The furan ring is planar and attached to the cyclopentane ring at atom C17, the dihedral angle between the furan ring and the C13/C14/C15/C16 mean plane is 56.7 (3)°. The two acetate groups are planar and in *cis*-configuration with respect to the C6—C7 bond, the dihedral angle between the mean plane of these two acetate moieties is 44.4 (3)°(Fig.1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In the structure, the weak C5—H5A···O7, C7—H7A···O8 and C9—H9A···O7 intramolecular interactions generate S(5) ring motifs, the C28—H28D···O5 and C29—H28D···O5 generate S(6) ring motifs (Bernstein *et al.*,1995), whereas the intermolecular C27—H27A···O2($-x$, $-y$, z) interaction connects the molecules into dimers (Fig. 2).

Experimental

Seeds of *C. siamensis* (600 g) were extracted with hexane-acetone (1:1 V/V) over the period of 3 days at room temperature. The mixture was filtered and concentrated under reduced pressure to provide the crude extract (62.21 g). This crude extract was subjected to quick column chromatography (QCC) over silica gel and eluted with a gradient of EtOAc-hexane to afford 13 factions (A1—A13). Fraction A8 (1.01 g) was subjected to repeated column chromatography using 25% EtOAc-hexane to give compound (I) (17.2 mg). Colorless plate shaped single crystals of the title compound were recrystallization from EtOAc-hexane (1:2 V/V) solution, Mp 493 K decomposition.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with the C—H distances in the range 0.93–0.98 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. A total of 2006 Friedel pairs were merged

before final refinement as there is no large anomalous dispersion for the determination of the absolute configuration. The absolute configuration of the title compound was selected arbitrarily.

Figures

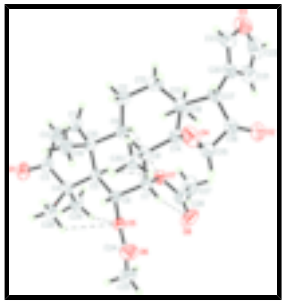

Fig. 1. The asymmetric unit of showing 40% probability displacement ellipsoids and the atomic numbering scheme. Intramolecular hydrogen bonds are shown as dashed lines.

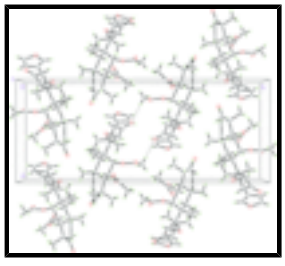

Fig. 2. The crystal packing viewed along the *c* axis. Weak C—H···O interactions are shown as dash lines.

(8*S**,10*R**)-17-(3-furyl)-4,4,8,10,13-pentamethyl-3,16-dioxo-6,7,9,11,12,17- hexahydro-14,15-epoxy-5*H*-cyclopenta[*a*]phenanthrene-6*α*,7*α*-diyl diacetate

Crystal data

$C_{30}H_{36}O_8$	D_x = 1.299 Mg m^{-3}
M_r = 524.59	Melting point: 493 K, decomposition K
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2 2ab	Cell parameters from 2724 reflections
a = 12.189 (3) Å	θ = 1.8–25.0º
b = 29.983 (5) Å	μ = 0.09 mm^{-1}
c = 7.3372 (11) Å	T = 297 (2) K
V = 2681.5 (9) Å^3	Plate, colourless
Z = 4	0.39 × 0.10 × 0.07 mm
F_{000} = 1120	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2724 independent reflections
Radiation source: fine-focus sealed tube	1782 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.100
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 25.0º
T = 297(2) K	θ_{min} = 1.8º

ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.965, T_{max} = 0.994

20575 measured reflections

h = −14→12

k = −35→32

l = −8→8

Refinement

Refinement on F^2

Least-squares matrix: full

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

$wR(F^2)$ = 0.125

S = 1.02

2724 reflections

351 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

$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.359P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.16 e Å^{-3}

$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0128 (16)

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 $F^2 > \sigma(F^2)$ 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}
O1	0.7277 (3)	0.20923 (12)	0.2888 (6)	0.0843 (11)
O2	−0.0801 (3)	0.04653 (14)	0.1717 (6)	0.0975 (13)
O3	−0.1807 (4)	0.1049 (2)	0.7665 (6)	0.1104 (15)
O4	0.0294 (3)	0.14448 (14)	0.0196 (5)	0.0819 (11)
O5	0.4887 (2)	0.10007 (9)	−0.1064 (4)	0.0495 (7)
O6	0.4156 (3)	0.11150 (12)	−0.3842 (5)	0.0733 (10)
O7	0.3165 (2)	0.08053 (8)	0.1306 (4)	0.0486 (7)
O8	0.2993 (4)	0.02858 (12)	−0.0864 (6)	0.1002 (13)
C1	0.4516 (4)	0.20161 (14)	0.4065 (6)	0.0577 (12)
H1A	0.4052	0.2069	0.5048	0.069*
C2	0.5586 (4)	0.20273 (15)	0.4328 (7)	0.0631 (13)

H2A	0.5858	0.2091	0.5484	0.076*
C3	0.6344 (4)	0.19424 (15)	0.2850 (7)	0.0586 (13)
C4	0.5981 (3)	0.16439 (13)	0.1284 (6)	0.0479 (11)
C5	0.4730 (3)	0.15332 (13)	0.1422 (5)	0.0412 (9)
H5A	0.4674	0.1289	0.2305	0.049*
C6	0.4207 (3)	0.13562 (13)	−0.0334 (6)	0.0425 (10)
H6A	0.4157	0.1598	−0.1228	0.051*
C7	0.3065 (3)	0.11623 (13)	0.0000 (6)	0.0454 (10)
H7A	0.2764	0.1047	−0.1145	0.054*
C8	0.2303 (3)	0.15175 (13)	0.0774 (6)	0.0453 (11)
C9	0.2831 (3)	0.17297 (13)	0.2505 (6)	0.0488 (11)
H9A	0.2926	0.1480	0.3353	0.059*
C10	0.4017 (3)	0.19194 (12)	0.2214 (6)	0.0438 (10)
C11	0.2019 (4)	0.20428 (15)	0.3457 (7)	0.0661 (13)
H11A	0.2360	0.2158	0.4555	0.079*
H11B	0.1872	0.2294	0.2661	0.079*
C12	0.0927 (4)	0.18242 (15)	0.3972 (7)	0.0664 (13)
H12A	0.0838	0.1841	0.5284	0.080*
H12B	0.0335	0.1994	0.3423	0.080*
C13	0.0817 (3)	0.13369 (15)	0.3387 (6)	0.0509 (11)
C14	0.1178 (3)	0.13163 (15)	0.1389 (6)	0.0525 (11)
C15	0.0509 (4)	0.0982 (2)	0.0452 (8)	0.0765 (16)
H15A	0.0804	0.0794	−0.0523	0.092*
C16	−0.0310 (4)	0.08116 (19)	0.1807 (7)	0.0706 (14)
C17	−0.0398 (3)	0.11626 (16)	0.3268 (6)	0.0574 (12)
H17A	−0.0844	0.1406	0.2773	0.069*
C18	0.1430 (4)	0.10127 (17)	0.4612 (7)	0.0669 (14)
H18A	0.2196	0.1087	0.4621	0.100*
H18B	0.1338	0.0715	0.4160	0.100*
H18C	0.1144	0.1031	0.5828	0.100*
C19	0.4068 (4)	0.23638 (13)	0.1148 (7)	0.0655 (14)
H19A	0.4793	0.2487	0.1237	0.098*
H19B	0.3549	0.2570	0.1655	0.098*
H19C	0.3893	0.2310	−0.0109	0.098*
C20	−0.0921 (4)	0.10193 (18)	0.5024 (7)	0.0623 (13)
C21	−0.0981 (4)	0.0586 (2)	0.5791 (8)	0.0827 (16)
H21A	−0.0702	0.0326	0.5279	0.099*
C22	−0.1507 (5)	0.0618 (3)	0.7375 (10)	0.100 (2)
H22A	−0.1647	0.0382	0.8163	0.119*
C23	−0.1448 (5)	0.1283 (2)	0.6193 (9)	0.0912 (18)
H23A	−0.1557	0.1587	0.6025	0.109*
C24	0.4794 (4)	0.09201 (16)	−0.2885 (7)	0.0582 (12)
C25	0.5585 (4)	0.05754 (19)	−0.3489 (8)	0.0854 (17)
H25A	0.5334	0.0444	−0.4606	0.128*
H25B	0.5645	0.0349	−0.2570	0.128*
H25C	0.6290	0.0710	−0.3680	0.128*
C26	0.3122 (4)	0.03785 (17)	0.0706 (9)	0.0687 (14)
C27	0.3269 (5)	0.00620 (17)	0.2258 (8)	0.0891 (17)
H27A	0.2609	−0.0108	0.2424	0.134*

H27B	0.3425	0.0227	0.3350	0.134*
H27C	0.3868	−0.0136	0.1999	0.134*
C28	0.6329 (4)	0.18517 (16)	−0.0557 (7)	0.0642 (14)
H28A	0.7092	0.1930	−0.0510	0.096*
H28B	0.5900	0.2114	−0.0785	0.096*
H28D	0.6211	0.1640	−0.1517	0.096*
C29	0.6656 (3)	0.12137 (14)	0.1587 (7)	0.0639 (14)
H29A	0.7420	0.1276	0.1397	0.096*
H29D	0.6420	0.0989	0.0741	0.096*
H29B	0.6548	0.1109	0.2810	0.096*
C30	0.2062 (4)	0.18580 (15)	−0.0755 (7)	0.0623 (13)
H30D	0.2742	0.1970	−0.1234	0.093*
H30A	0.1640	0.2101	−0.0270	0.093*
H30B	0.1657	0.1715	−0.1712	0.093*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.060 (2)	0.090 (2)	0.103 (3)	−0.0292 (19)	−0.007 (2)	−0.020 (2)
O2	0.083 (2)	0.112 (3)	0.098 (3)	−0.040 (2)	0.014 (2)	−0.026 (3)
O3	0.102 (3)	0.148 (4)	0.081 (3)	−0.035 (3)	0.029 (3)	−0.017 (3)
O4	0.0501 (18)	0.126 (3)	0.069 (2)	−0.006 (2)	−0.0200 (18)	0.018 (2)
O5	0.0452 (16)	0.0519 (16)	0.0514 (18)	−0.0011 (13)	0.0003 (14)	−0.0054 (15)
O6	0.078 (2)	0.089 (2)	0.052 (2)	−0.001 (2)	−0.0079 (19)	−0.0021 (19)
O7	0.0501 (15)	0.0375 (16)	0.0581 (18)	−0.0025 (13)	−0.0017 (15)	0.0014 (15)
O8	0.137 (4)	0.066 (2)	0.098 (3)	−0.015 (2)	−0.007 (3)	−0.027 (2)
C1	0.065 (3)	0.052 (3)	0.056 (3)	−0.011 (2)	0.001 (2)	−0.011 (2)
C2	0.072 (3)	0.063 (3)	0.054 (3)	−0.021 (3)	−0.012 (3)	−0.004 (3)
C3	0.058 (3)	0.051 (3)	0.067 (3)	−0.014 (2)	−0.013 (3)	0.000 (3)
C4	0.043 (2)	0.046 (2)	0.054 (3)	−0.007 (2)	0.001 (2)	0.002 (2)
C5	0.041 (2)	0.039 (2)	0.044 (2)	−0.0046 (18)	−0.003 (2)	0.000 (2)
C6	0.046 (2)	0.037 (2)	0.045 (2)	0.0048 (19)	0.0000 (19)	−0.002 (2)
C7	0.040 (2)	0.048 (2)	0.048 (3)	−0.0015 (19)	−0.003 (2)	0.004 (2)
C8	0.043 (2)	0.047 (2)	0.046 (2)	0.004 (2)	−0.005 (2)	0.003 (2)
C9	0.049 (2)	0.045 (2)	0.052 (3)	0.001 (2)	0.000 (2)	−0.004 (2)
C10	0.047 (2)	0.038 (2)	0.047 (3)	−0.0009 (19)	−0.005 (2)	−0.004 (2)
C11	0.065 (3)	0.058 (3)	0.075 (3)	0.001 (2)	0.009 (3)	−0.016 (3)
C12	0.059 (3)	0.068 (3)	0.072 (3)	0.004 (2)	0.006 (3)	−0.008 (3)
C13	0.040 (2)	0.058 (3)	0.055 (3)	0.002 (2)	−0.005 (2)	−0.003 (2)
C14	0.043 (2)	0.064 (3)	0.051 (3)	0.002 (2)	−0.006 (2)	0.001 (2)
C15	0.059 (3)	0.105 (5)	0.065 (3)	−0.023 (3)	0.001 (3)	−0.028 (3)
C16	0.049 (3)	0.093 (4)	0.069 (3)	−0.018 (3)	0.005 (3)	−0.014 (3)
C17	0.037 (2)	0.073 (3)	0.062 (3)	0.002 (2)	−0.001 (2)	0.000 (3)
C18	0.050 (3)	0.089 (4)	0.062 (3)	−0.005 (3)	−0.006 (2)	0.018 (3)
C19	0.066 (3)	0.037 (2)	0.093 (4)	−0.003 (2)	0.001 (3)	0.006 (3)
C20	0.042 (2)	0.078 (4)	0.067 (3)	−0.003 (3)	0.000 (2)	−0.005 (3)
C21	0.066 (3)	0.098 (4)	0.084 (4)	−0.003 (3)	0.003 (3)	0.015 (4)
C22	0.078 (4)	0.130 (6)	0.091 (5)	−0.033 (4)	−0.004 (4)	0.028 (5)

C23	0.088 (4)	0.101 (4)	0.085 (4)	−0.019 (3)	0.037 (4)	−0.013 (4)
C24	0.052 (3)	0.064 (3)	0.058 (3)	−0.010 (2)	0.002 (3)	−0.010 (3)
C25	0.086 (4)	0.088 (4)	0.082 (4)	0.005 (3)	0.010 (3)	−0.030 (3)
C26	0.065 (3)	0.051 (3)	0.090 (4)	−0.007 (2)	−0.007 (3)	−0.010 (3)
C27	0.092 (4)	0.054 (3)	0.122 (4)	−0.004 (3)	−0.009 (4)	0.020 (4)
C28	0.055 (3)	0.069 (3)	0.069 (3)	−0.013 (2)	−0.001 (2)	0.013 (3)
C29	0.048 (2)	0.057 (3)	0.086 (4)	0.001 (2)	−0.013 (3)	−0.001 (3)
C30	0.058 (3)	0.060 (3)	0.069 (3)	0.010 (2)	−0.003 (2)	0.006 (3)

Geometric parameters (Å, °)

O1—C3	1.224 (5)	C12—H12B	0.9700
O2—C16	1.200 (6)	C13—C18	1.520 (6)
O3—C22	1.359 (8)	C13—C14	1.532 (6)
O3—C23	1.360 (7)	C13—C17	1.573 (6)
O4—C15	1.424 (7)	C14—C15	1.464 (6)
O4—C14	1.440 (5)	C15—C16	1.499 (7)
O5—C24	1.362 (5)	C15—H15A	0.9800
O5—C6	1.453 (5)	C16—C17	1.506 (7)
O6—C24	1.199 (5)	C17—C20	1.500 (7)
O7—C26	1.354 (6)	C17—H17A	0.9800
O7—C7	1.442 (5)	C18—H18A	0.9600
O8—C26	1.195 (6)	C18—H18B	0.9600
C1—C2	1.319 (6)	C18—H18C	0.9600
C1—C10	1.516 (6)	C19—H19A	0.9600
C1—H1A	0.9300	C19—H19B	0.9600
C2—C3	1.447 (7)	C19—H19C	0.9600
C2—H2A	0.9300	C20—C23	1.331 (7)
C3—C4	1.522 (6)	C20—C21	1.419 (7)
C4—C29	1.546 (6)	C21—C22	1.331 (8)
C4—C28	1.547 (6)	C21—H21A	0.9300
C4—C5	1.564 (5)	C22—H22A	0.9300
C5—C6	1.532 (5)	C23—H23A	0.9300
C5—C10	1.560 (5)	C24—C25	1.482 (7)
C5—H5A	0.9800	C25—H25A	0.9600
C6—C7	1.528 (5)	C25—H25B	0.9600
C6—H6A	0.9800	C25—H25C	0.9600
C7—C8	1.523 (6)	C26—C27	1.493 (7)
C7—H7A	0.9800	C27—H27A	0.9600
C8—C30	1.545 (6)	C27—H27B	0.9600
C8—C9	1.559 (6)	C27—H27C	0.9600
C8—C14	1.564 (6)	C28—H28A	0.9600
C9—C11	1.532 (6)	C28—H28B	0.9600
C9—C10	1.568 (6)	C28—H28D	0.9600
C9—H9A	0.9800	C29—H29A	0.9600
C10—C19	1.546 (6)	C29—H29D	0.9600
C11—C12	1.531 (6)	C29—H29B	0.9600
C11—H11A	0.9700	C30—H30D	0.9600
C11—H11B	0.9700	C30—H30A	0.9600

C12—C13	1.529 (6)	C30—H30B	0.9600
C12—H12A	0.9700		
C22—O3—C23	106.2 (5)	C15—C14—C8	128.1 (4)
C15—O4—C14	61.5 (3)	C13—C14—C8	120.8 (3)
C24—O5—C6	116.4 (3)	O4—C15—C14	59.8 (3)
C26—O7—C7	118.8 (4)	O4—C15—C16	107.3 (5)
C2—C1—C10	122.2 (4)	C14—C15—C16	107.0 (4)
C2—C1—H1A	118.9	O4—C15—H15A	122.2
C10—C1—H1A	118.9	C14—C15—H15A	122.2
C1—C2—C3	121.1 (5)	C16—C15—H15A	122.2
C1—C2—H2A	119.4	O2—C16—C15	126.2 (5)
C3—C2—H2A	119.4	O2—C16—C17	127.5 (5)
O1—C3—C2	120.8 (5)	C15—C16—C17	106.3 (4)
O1—C3—C4	120.2 (5)	C20—C17—C16	116.2 (4)
C2—C3—C4	118.9 (4)	C20—C17—C13	116.6 (4)
C3—C4—C29	103.2 (4)	C16—C17—C13	101.8 (3)
C3—C4—C28	110.0 (3)	C20—C17—H17A	107.2
C29—C4—C28	108.4 (4)	C16—C17—H17A	107.2
C3—C4—C5	111.1 (4)	C13—C17—H17A	107.2
C29—C4—C5	109.4 (3)	C13—C18—H18A	109.5
C28—C4—C5	114.1 (4)	C13—C18—H18B	109.5
C6—C5—C10	109.8 (3)	H18A—C18—H18B	109.5
C6—C5—C4	115.2 (3)	C13—C18—H18C	109.5
C10—C5—C4	114.2 (3)	H18A—C18—H18C	109.5
C6—C5—H5A	105.6	H18B—C18—H18C	109.5
C10—C5—H5A	105.6	C10—C19—H19A	109.5
C4—C5—H5A	105.6	C10—C19—H19B	109.5
O5—C6—C7	107.4 (3)	H19A—C19—H19B	109.5
O5—C6—C5	109.1 (3)	C10—C19—H19C	109.5
C7—C6—C5	112.1 (3)	H19A—C19—H19C	109.5
O5—C6—H6A	109.4	H19B—C19—H19C	109.5
C7—C6—H6A	109.4	C23—C20—C21	105.3 (5)
C5—C6—H6A	109.4	C23—C20—C17	126.1 (5)
O7—C7—C8	108.8 (3)	C21—C20—C17	128.7 (5)
O7—C7—C6	108.2 (3)	C22—C21—C20	107.7 (6)
C8—C7—C6	110.5 (3)	C22—C21—H21A	126.1
O7—C7—H7A	109.8	C20—C21—H21A	126.1
C8—C7—H7A	109.8	C21—C22—O3	109.6 (6)
C6—C7—H7A	109.8	C21—C22—H22A	125.2
C7—C8—C30	107.9 (3)	O3—C22—H22A	125.2
C7—C8—C9	109.7 (3)	C20—C23—O3	111.1 (6)
C30—C8—C9	113.6 (3)	C20—C23—H23A	124.4
C7—C8—C14	111.9 (3)	O3—C23—H23A	124.4
C30—C8—C14	107.3 (3)	O6—C24—O5	122.8 (4)
C9—C8—C14	106.5 (3)	O6—C24—C25	125.9 (5)
C11—C9—C8	110.8 (3)	O5—C24—C25	111.3 (5)
C11—C9—C10	115.8 (3)	C24—C25—H25A	109.5
C8—C9—C10	114.7 (3)	C24—C25—H25B	109.5
C11—C9—H9A	104.7	H25A—C25—H25B	109.5

C8—C9—H9A	104.7	C24—C25—H25C	109.5
C10—C9—H9A	104.7	H25A—C25—H25C	109.5
C1—C10—C19	105.8 (3)	H25B—C25—H25C	109.5
C1—C10—C5	104.6 (3)	O8—C26—O7	122.5 (5)
C19—C10—C5	115.4 (4)	O8—C26—C27	127.1 (5)
C1—C10—C9	108.5 (3)	O7—C26—C27	110.4 (5)
C19—C10—C9	114.7 (3)	C26—C27—H27A	109.5
C5—C10—C9	107.2 (3)	C26—C27—H27B	109.5
C12—C11—C9	114.3 (4)	H27A—C27—H27B	109.5
C12—C11—H11A	108.7	C26—C27—H27C	109.5
C9—C11—H11A	108.7	H27A—C27—H27C	109.5
C12—C11—H11B	108.7	H27B—C27—H27C	109.5
C9—C11—H11B	108.7	C4—C28—H28A	109.5
H11A—C11—H11B	107.6	C4—C28—H28B	109.5
C13—C12—C11	114.6 (4)	H28A—C28—H28B	109.5
C13—C12—H12A	108.6	C4—C28—H28D	109.5
C11—C12—H12A	108.6	H28A—C28—H28D	109.5
C13—C12—H12B	108.6	H28B—C28—H28D	109.5
C11—C12—H12B	108.6	C4—C29—H29A	109.5
H12A—C12—H12B	107.6	C4—C29—H29D	109.5
C18—C13—C12	113.7 (4)	H29A—C29—H29D	109.5
C18—C13—C14	113.5 (4)	C4—C29—H29B	109.5
C12—C13—C14	106.4 (4)	H29A—C29—H29B	109.5
C18—C13—C17	106.4 (4)	H29D—C29—H29B	109.5
C12—C13—C17	114.6 (4)	C8—C30—H30D	109.5
C14—C13—C17	101.8 (3)	C8—C30—H30A	109.5
O4—C14—C15	58.7 (3)	H30D—C30—H30A	109.5
O4—C14—C13	110.9 (3)	C8—C30—H30B	109.5
C15—C14—C13	108.5 (4)	H30D—C30—H30B	109.5
O4—C14—C8	112.2 (3)	H30A—C30—H30B	109.5
C10—C1—C2—C3	−0.6 (7)	C11—C12—C13—C14	−47.8 (5)
C1—C2—C3—O1	155.2 (5)	C11—C12—C13—C17	−159.4 (4)
C1—C2—C3—C4	−27.8 (7)	C15—O4—C14—C13	−99.5 (4)
O1—C3—C4—C29	68.6 (5)	C15—O4—C14—C8	122.1 (4)
C2—C3—C4—C29	−108.4 (4)	C18—C13—C14—O4	151.9 (4)
O1—C3—C4—C28	−46.8 (6)	C12—C13—C14—O4	−82.3 (4)
C2—C3—C4—C28	136.1 (4)	C17—C13—C14—O4	37.9 (5)
O1—C3—C4—C5	−174.2 (4)	C18—C13—C14—C15	89.2 (4)
C2—C3—C4—C5	8.8 (6)	C12—C13—C14—C15	−145.0 (4)
C3—C4—C5—C6	162.7 (3)	C17—C13—C14—C15	−24.8 (5)
C29—C4—C5—C6	−84.1 (4)	C18—C13—C14—C8	−73.9 (5)
C28—C4—C5—C6	37.6 (5)	C12—C13—C14—C8	51.9 (5)
C3—C4—C5—C10	34.3 (5)	C17—C13—C14—C8	172.1 (4)
C29—C4—C5—C10	147.6 (4)	C7—C8—C14—O4	−108.7 (4)
C28—C4—C5—C10	−90.8 (4)	C30—C8—C14—O4	9.4 (5)
C24—O5—C6—C7	82.0 (4)	C9—C8—C14—O4	131.4 (4)
C24—O5—C6—C5	−156.3 (3)	C7—C8—C14—C15	−41.9 (6)
C10—C5—C6—O5	179.4 (3)	C30—C8—C14—C15	76.3 (6)
C4—C5—C6—O5	48.8 (4)	C9—C8—C14—C15	−161.8 (5)

C10—C5—C6—C7	−61.8 (4)	C7—C8—C14—C13	117.6 (4)
C4—C5—C6—C7	167.7 (3)	C30—C8—C14—C13	−124.2 (4)
C26—O7—C7—C8	136.6 (4)	C9—C8—C14—C13	−2.3 (5)
C26—O7—C7—C6	−103.4 (4)	C14—O4—C15—C16	100.0 (4)
O5—C6—C7—O7	60.8 (4)	C13—C14—C15—O4	103.7 (4)
C5—C6—C7—O7	−59.0 (4)	C8—C14—C15—O4	−94.8 (5)
O5—C6—C7—C8	179.8 (3)	O4—C14—C15—C16	−100.5 (5)
C5—C6—C7—C8	60.1 (4)	C13—C14—C15—C16	3.2 (6)
O7—C7—C8—C30	−171.2 (3)	C8—C14—C15—C16	164.7 (4)
C6—C7—C8—C30	70.2 (4)	O4—C15—C16—O2	137.6 (6)
O7—C7—C8—C9	64.6 (4)	C14—C15—C16—O2	−159.5 (5)
C6—C7—C8—C9	−54.0 (4)	O4—C15—C16—C17	−41.7 (5)
O7—C7—C8—C14	−53.4 (4)	C14—C15—C16—C17	21.2 (6)
C6—C7—C8—C14	−172.0 (3)	O2—C16—C17—C20	17.0 (8)
C7—C8—C9—C11	−172.5 (3)	C15—C16—C17—C20	−163.7 (4)
C30—C8—C9—C11	66.8 (5)	O2—C16—C17—C13	144.8 (5)
C14—C8—C9—C11	−51.2 (4)	C15—C16—C17—C13	−35.9 (5)
C7—C8—C9—C10	54.2 (4)	C18—C13—C17—C20	44.8 (5)
C30—C8—C9—C10	−66.6 (5)	C12—C13—C17—C20	−81.8 (5)
C14—C8—C9—C10	175.5 (3)	C14—C13—C17—C20	163.9 (4)
C2—C1—C10—C19	−80.6 (5)	C18—C13—C17—C16	−82.7 (5)
C2—C1—C10—C5	41.7 (5)	C12—C13—C17—C16	150.7 (4)
C2—C1—C10—C9	155.8 (4)	C14—C13—C17—C16	36.4 (5)
C6—C5—C10—C1	171.7 (3)	C16—C17—C20—C23	−153.2 (5)
C4—C5—C10—C1	−57.3 (4)	C13—C17—C20—C23	86.7 (6)
C6—C5—C10—C19	−72.5 (4)	C16—C17—C20—C21	25.9 (7)
C4—C5—C10—C19	58.5 (5)	C13—C17—C20—C21	−94.2 (6)
C6—C5—C10—C9	56.6 (4)	C23—C20—C21—C22	−1.6 (6)
C4—C5—C10—C9	−172.3 (3)	C17—C20—C21—C22	179.0 (5)
C11—C9—C10—C1	61.7 (5)	C20—C21—C22—O3	0.9 (6)
C8—C9—C10—C1	−167.3 (3)	C23—O3—C22—C21	0.2 (7)
C11—C9—C10—C19	−56.3 (5)	C21—C20—C23—O3	1.8 (6)
C8—C9—C10—C19	74.7 (4)	C17—C20—C23—O3	−178.9 (4)
C11—C9—C10—C5	174.2 (4)	C22—O3—C23—C20	−1.3 (7)
C8—C9—C10—C5	−54.8 (4)	C6—O5—C24—O6	−3.1 (6)
C8—C9—C11—C12	55.8 (5)	C6—O5—C24—C25	175.7 (3)
C10—C9—C11—C12	−171.4 (4)	C7—O7—C26—O8	−0.7 (7)
C9—C11—C12—C13	−2.1 (6)	C7—O7—C26—C27	178.6 (4)
C11—C12—C13—C18	77.9 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5A···O7	0.98	2.45	2.900 (5)	107
C7—H7A···O8	0.98	2.31	2.705 (5)	103
C9—H9A···O7	0.98	2.54	2.936 (5)	104
C27—H27A···O2^{i}	0.96	2.51	3.422 (7)	160
C28—H28D···O5	0.96	2.53	3.121 (6)	120
C29—H29D···O5	0.96	2.29	2.973 (5)	127

Symmetry codes: (i) −*x*, −*y*, *z*.

Fig. 1

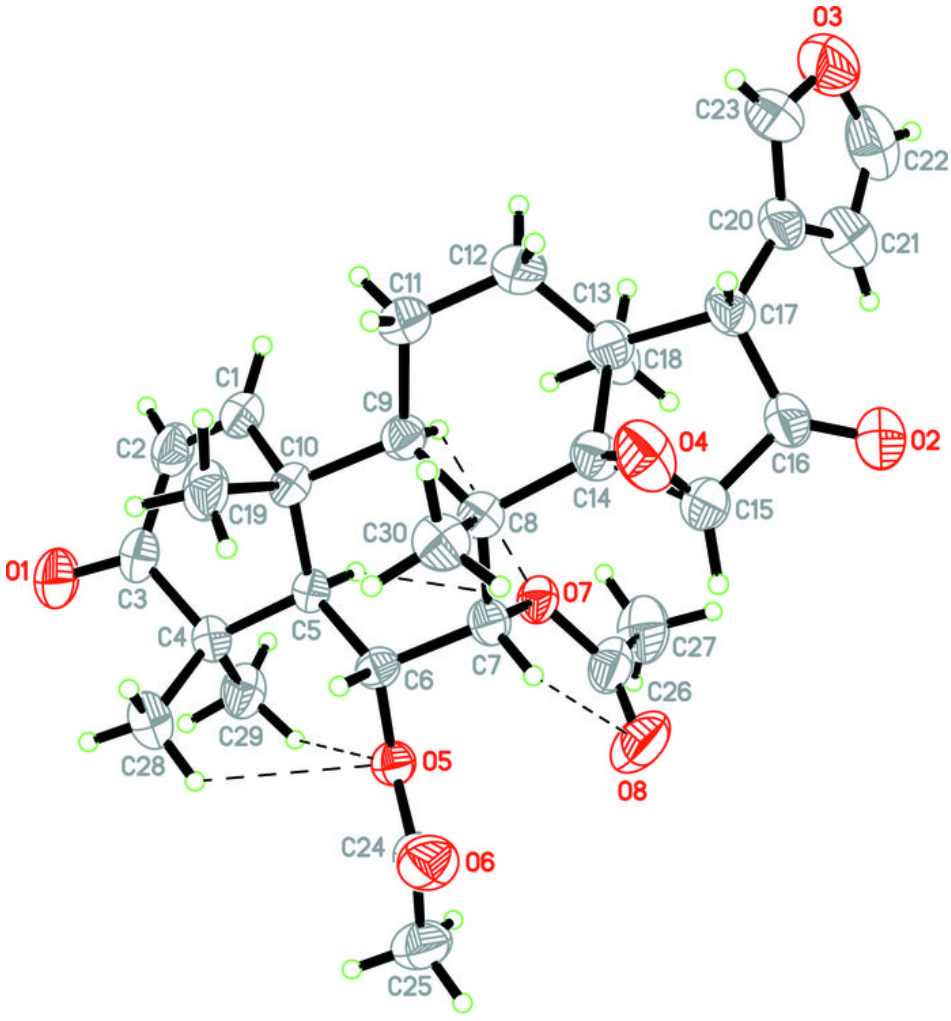

Fig. 2