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Methyl 6-methoxygambogate

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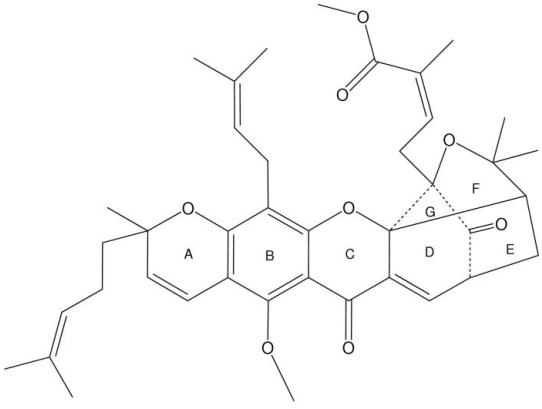
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; disorder in main residue; R factor = 0.049; wR factor = 0.153; data-to-parameter ratio = 7.5.

The title compound, $C_{40}H_{48}O_8$, is a derivative of gambogic acid, which is an effective telomerase inhibitor and displays potent anticancer activity both *in vitro* and *in vivo*. The three fused rings, *A*, *B* and *C*. are almost coplanar, with a mean deviation of 0.0670 (3) Å from their least-squares plane. The bridged structural unit including rings *D*, *E* and *G* forms a conformation like that of triethylenediamine. The structure is assembled *via* C—H···O hydrogen bonds. One methyl group is disordered over two positions; the site occupancy factors are *ca* 0.7 and 0.3.

Related literature

For related literature, see Liu *et al.* (2004).



Experimental

Crystal data

$C_{40}H_{48}O_8$
M_r = 656.78
Monoclinic, $P2_1$
a = 7.865 (3) Å
b = 17.017 (7) Å
c = 13.711 (6) Å
β = 105.322 (6)°
V = 1769.8 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.54 × 0.40 × 0.27 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.955, T_{max} = 0.977
8951 measured reflections
3235 independent reflections
2055 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.153
S = 1.05
3235 reflections
432 parameters
125 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O8^{i}	0.93	2.53	3.400 (8)	156
C31—H31C···O6^{ii}	0.96	2.58	3.535 (8)	176

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: BQ2036).

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supplementary materials

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Methyl 6-methoxygambogate

W. Liu, F. Feng, X. Wang, Q. Guo and Q. You

Comment

Garcinia L. Planch (*e.g. Garcinia hanburyi* Hook. and *Garcinia morella* Gesv.), is used in traditional Chinese medicine for detoxification, maintaining hemostasis, and as an anthelmintic. Gambogic acid (GBA), the primary component of Gamboge, is a prenylated xanthonoid that has the bridged structural moiety of tricyclo-4-oxa[4.3.1.0]decan-2-one. GBA is an effective telomerase inhibitor and displays potent anticancer activity both *in vitro* and *in vivo*. Moreover, GBA has been shown to induce apoptosis in a cell cycle independent manner. In order to study the structure–activity relationship of GBA, several derivates were synthesized and showed potential antitumor activity. The title compound, 6-methoxy-methyl-gambogate, is a derivative of them (Liu *et al.*, 2004). We report here the crystal structure of (I).

The crystal structure of the title compound is shown in Fig. 1. The three fused rings A(O1/C1–C4/C9), B(C4–C9) and C(O2/C6/C7/C10–C12) is almost coplanar with mean deviation being 0.0670 (3) Å from their least square plane. Rings D(C10/C11/C13–C16), E(C13–C15/C17–C19) and G(C10/C13–C15/C18/C19) formed a conformation like triethylenediamine. Three chains including H(C34–C39/O7/O8), I(C27–C31) and J(C21–C26) exists in the compound. Chain H is almost planar indicated by the mean deviations being 0.0436 (2) Å from their least square plane, and it have a dihedral angle of 12.1 (6)° with the plane of the fused rings A, B and C. The double bond C35═C36 has a *syn* conformation with torision angle C38—C35—C36—C37 being −177.1 (5)°.

The title compound is assembled *via* hydrogen bonds C2—H2···O8^{i} and C31—H31C···O6^{ii} [Symmetry code: (i) 1 − *x*, 1/2 + *y*, 1 − *z*; (ii) 1 − *x*, 1/2 + *y*, −*z*] as shown in Fig. 2. Details of the hydrogen bonds geometry is shown in Table 1.

Experimental

Gambogic acid (100 mg) dissolved in acetone (10 ml), anhydride K_2CO_3 (300 mg) and iodomethane (1 ml) was added and stirred at room temperature for a week. Inpoured the reagent into water (10 ml), and extracted with ether (60 ml) for 3 times. The ether layer was evaporated afrer being washed by water and dryed with anhydride Na_2SO_4. The residue was separated by silica chromatography, 6-methoxyl-methyl-gambogate (30 mg) was isolated as yellow needles. The title compound was dissolved in ethanol to get saturated solution, the cube crystals were obtained after a week's standing.

Refinement

The positions of the H atoms of the hydroxyl groups were located initially in a difference Fourier map and then constrained to ride on the parent O atom with O—H = 0.82–0.85, U_{iso}(H) = 1.5U_{eq}(O). The methyl H atoms were constrained to an ideal geometry with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C). Friedel pairs were averaged before the final refinement as the absolute could not be determined unambiguously.

Figures

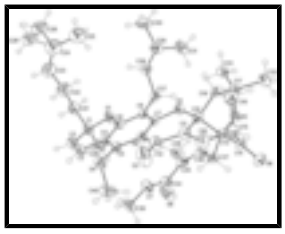


Fig. 1. A view of the asymmetric unit of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H-atoms are shown as spheres of arbitrary radii.

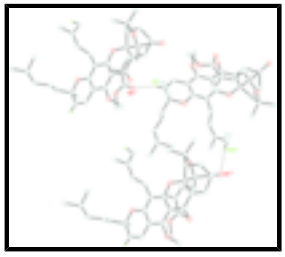


Fig. 2. Part of the packing diagram of the title compound shown the C—H···O hydrogen bonds. Atoms marked with (i) and (ii) are at the symmetric position of $(1 - x, 1/2 + y, 1 - z)$ and $(1 - x, 1/2 + y, -z)$, respectively. Some H atoms are omitted for the sake of clarity.

Methyl 6-methoxygambogate

Crystal data

$C_{40}H_{48}O_8$	$F_{000} = 704$
$M_r = 656.78$	$D_x = 1.232$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 2245 reflections
$a = 7.865$ (3) Å	$\theta = 2.4$–21.5°
$b = 17.017$ (7) Å	$\mu = 0.09$ mm^{-1}
$c = 13.711$ (6) Å	$T = 298$ (2) K
$\beta = 105.322$ (6)°	Prism, colourless
$V = 1769.8$ (13) Å^3	0.54 × 0.40 × 0.27 mm
$Z = 2$	

Data collection

Bruker SMART CCD area-detector diffractometer	3235 independent reflections
Radiation source: fine-focus sealed tube	2055 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.045$
$T = 298$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 1.5^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -9 \rightarrow 8$
$T_{min} = 0.955$, $T_{max} = 0.977$	$k = -20 \rightarrow 17$
8951 measured reflections	$l = -14 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0745P)^2 + 0.3642P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.153$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
3235 reflections	$\Delta\rho_{min} = -0.47$ e Å^{-3}
432 parameters	Extinction correction: none
125 restraints	Absolute structure: Flack (1983), with how many Friedel pairs?
Primary atom site location: structure-invariant direct methods	Flack parameter: ?
Secondary atom site location: difference Fourier map	

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	0.5187 (6)	0.4902 (2)	0.2713 (3)	0.0607 (11)	
O2	0.7046 (4)	0.23511 (19)	0.2426 (2)	0.0438 (8)	
O3	0.9237 (7)	0.2103 (3)	0.5463 (3)	0.1002 (18)	
O4	0.8083 (6)	0.3577 (2)	0.5729 (3)	0.0662 (12)	
O5	0.5472 (4)	0.0946 (2)	0.1436 (2)	0.0484 (9)	
O6	0.5658 (6)	−0.0343 (2)	0.2965 (3)	0.0706 (12)	
O7	0.1983 (6)	0.3344 (3)	0.3839 (3)	0.0737 (12)	
O8	0.3480 (8)	0.2268 (3)	0.4380 (4)	0.1069 (19)	
C1	0.4618 (9)	0.5561 (3)	0.3239 (4)	0.0602 (15)	
C2	0.5470 (10)	0.5550 (4)	0.4337 (4)	0.0739 (19)	
H2	0.5404	0.5999	0.4712	0.089*	
C3	0.6330 (9)	0.4927 (4)	0.4817 (4)	0.0626 (17)	
H3	0.6777	0.4939	0.5517	0.075*	
C4	0.6574 (8)	0.4233 (3)	0.4258 (4)	0.0478 (13)	
C5	0.7372 (7)	0.3556 (3)	0.4701 (4)	0.0462 (13)	

C6	0.7569 (7)	0.2895 (3)	0.4128 (4)	0.0433 (12)	
C7	0.6901 (6)	0.2951 (3)	0.3070 (3)	0.0383 (12)	
C8	0.6127 (6)	0.3626 (3)	0.2596 (4)	0.0389 (11)	
C9	0.5938 (7)	0.4244 (3)	0.3201 (4)	0.0433 (12)	
C10	0.7517 (6)	0.1570 (3)	0.2772 (4)	0.0413 (12)	
C11	0.8407 (7)	0.1514 (3)	0.3876 (4)	0.0504 (14)	
C12	0.8475 (7)	0.2180 (4)	0.4566 (4)	0.0592 (16)	
C13	0.5929 (6)	0.1014 (3)	0.2509 (4)	0.0422 (12)	
C14	0.6617 (8)	0.0210 (3)	0.2962 (4)	0.0512 (14)	
C15	0.8607 (8)	0.0173 (3)	0.3400 (5)	0.0608 (15)	
H15	0.9021	−0.0348	0.3664	0.073*	
C16	0.8921 (8)	0.0789 (3)	0.4205 (5)	0.0618 (16)	
H16	0.9434	0.0677	0.4883	0.074*	
C17	0.7047 (7)	0.1018 (3)	0.1087 (4)	0.0498 (13)	
C18	0.8537 (7)	0.1191 (3)	0.2049 (4)	0.0496 (13)	
H18	0.9377	0.1567	0.1900	0.060*	
C19	0.9515 (7)	0.0471 (4)	0.2605 (5)	0.0621 (16)	
H19A	1.0726	0.0612	0.2934	0.074*	
H19B	0.9529	0.0057	0.2122	0.074*	
C20	0.2619 (10)	0.5505 (5)	0.3030 (6)	0.095 (2)	
H20A	0.2094	0.5530	0.2314	0.143*	
H20B	0.2194	0.5933	0.3356	0.143*	
H20C	0.2312	0.5016	0.3288	0.143*	
C21	0.5154 (9)	0.6285 (4)	0.2739 (5)	0.0726 (16)	
H21A	0.4726	0.6749	0.3011	0.087*	
H21B	0.4580	0.6266	0.2020	0.087*	
C22	0.7133 (10)	0.6369 (4)	0.2884 (6)	0.090 (2)	
H22A	0.7716	0.6362	0.3602	0.109*	
H22B	0.7554	0.5920	0.2579	0.109*	
C23	0.7639 (11)	0.7107 (5)	0.2432 (6)	0.101 (2)	
H23	0.7458	0.7570	0.2752	0.121*	
C24	0.8284 (12)	0.7198 (6)	0.1670 (7)	0.1156 (18)	
C25	0.8507 (13)	0.6514 (6)	0.1007 (7)	0.1156 (18)	
H25A	0.9012	0.6698	0.0484	0.173*	
H25B	0.7378	0.6281	0.0706	0.173*	
H25C	0.9272	0.6128	0.1410	0.173*	
C26	0.888 (2)	0.7909 (10)	0.1209 (14)	0.111 (3)	0.66 (2)
H26A	0.8023	0.8037	0.0588	0.166*	0.66 (2)
H26B	0.9993	0.7803	0.1072	0.166*	0.66 (2)
H26C	0.9003	0.8343	0.1669	0.166*	0.66 (2)
C26'	0.858 (5)	0.8123 (12)	0.167 (3)	0.111 (3)	0.34 (2)
H26D	0.9077	0.8263	0.1127	0.166*	0.34 (2)
H26E	0.9377	0.8278	0.2304	0.166*	0.34 (2)
H26F	0.7475	0.8387	0.1590	0.166*	0.34 (2)
C27	0.5530 (7)	0.3704 (3)	0.1460 (3)	0.0426 (12)	
H27A	0.4452	0.4010	0.1275	0.051*	
H27B	0.5273	0.3186	0.1162	0.051*	
C28	0.6908 (7)	0.4095 (3)	0.1038 (4)	0.0527 (14)	
H28	0.7724	0.4406	0.1489	0.063*	

C29	0.7106 (8)	0.4053 (4)	0.0109 (5)	0.0641 (17)
C30	0.8509 (11)	0.4509 (5)	−0.0172 (6)	0.101 (3)
H30A	0.9261	0.4746	0.0423	0.151*
H30B	0.7984	0.4913	−0.0646	0.151*
H30C	0.9192	0.4165	−0.0475	0.151*
C31	0.5965 (11)	0.3570 (4)	−0.0713 (5)	0.085 (2)
H31A	0.4992	0.3365	−0.0492	0.128*
H31B	0.6640	0.3143	−0.0875	0.128*
H31C	0.5527	0.3891	−0.1302	0.128*
C32	0.7212 (10)	0.0255 (4)	0.0523 (5)	0.0759 (19)
H32A	0.7303	−0.0183	0.0975	0.114*
H32B	0.8247	0.0279	0.0278	0.114*
H32C	0.6190	0.0192	−0.0037	0.114*
C33	0.6785 (9)	0.1691 (4)	0.0333 (4)	0.0640 (16)
H33A	0.5662	0.1636	−0.0153	0.096*
H33B	0.7705	0.1681	−0.0008	0.096*
H33C	0.6822	0.2182	0.0684	0.096*
C34	0.2699 (8)	0.2663 (4)	0.3681 (5)	0.0580 (15)
C35	0.2475 (6)	0.2510 (3)	0.2599 (4)	0.0500 (13)
C36	0.3255 (6)	0.1904 (3)	0.2271 (4)	0.0493 (13)
H36	0.3111	0.1886	0.1576	0.059*
C37	0.4320 (7)	0.1257 (3)	0.2856 (4)	0.0508 (13)
H37A	0.3565	0.0801	0.2821	0.061*
H37B	0.4710	0.1415	0.3560	0.061*
C38	0.1371 (8)	0.3088 (4)	0.1866 (4)	0.0674 (17)
H38A	0.1286	0.2921	0.1186	0.101*
H38B	0.1912	0.3598	0.1974	0.101*
H38C	0.0213	0.3115	0.1970	0.101*
C39	0.2139 (10)	0.3574 (5)	0.4855 (5)	0.088 (2)
H39A	0.1879	0.3133	0.5229	0.131*
H39B	0.1325	0.3992	0.4865	0.131*
H39C	0.3320	0.3751	0.5157	0.131*
C40	0.7167 (10)	0.3145 (6)	0.6312 (5)	0.096 (3)
H40A	0.6355	0.3485	0.6521	0.144*
H40B	0.7994	0.2933	0.6898	0.144*
H40C	0.6529	0.2723	0.5912	0.144*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.096 (3)	0.043 (2)	0.043 (2)	0.015 (2)	0.019 (2)	0.0018 (18)
O2	0.054 (2)	0.0374 (19)	0.0374 (17)	0.0000 (16)	0.0084 (15)	−0.0019 (16)
O3	0.127 (4)	0.087 (4)	0.054 (3)	0.029 (3)	−0.035 (3)	−0.004 (2)
O4	0.086 (3)	0.068 (3)	0.037 (2)	−0.010 (2)	0.003 (2)	−0.0025 (18)
O5	0.0404 (19)	0.062 (2)	0.046 (2)	−0.0083 (17)	0.0171 (15)	−0.0095 (17)
O6	0.079 (3)	0.055 (2)	0.084 (3)	−0.013 (2)	0.031 (2)	0.001 (2)
O7	0.078 (3)	0.076 (3)	0.071 (3)	0.014 (2)	0.026 (2)	−0.014 (2)
O8	0.179 (6)	0.087 (3)	0.061 (3)	0.048 (4)	0.045 (3)	0.012 (3)

C1	0.087 (5)	0.041 (3)	0.057 (4)	0.007 (3)	0.026 (3)	−0.002 (3)
C2	0.128 (6)	0.046 (4)	0.053 (3)	0.006 (4)	0.033 (4)	−0.005 (3)
C3	0.097 (5)	0.052 (4)	0.043 (3)	−0.010 (3)	0.027 (3)	−0.009 (3)
C4	0.061 (3)	0.048 (3)	0.034 (3)	−0.014 (3)	0.012 (2)	−0.005 (2)
C5	0.047 (3)	0.053 (3)	0.033 (3)	−0.013 (3)	0.001 (2)	0.000 (2)
C6	0.045 (3)	0.044 (3)	0.036 (3)	−0.003 (2)	0.001 (2)	0.000 (2)
C7	0.038 (3)	0.037 (3)	0.040 (3)	−0.008 (2)	0.010 (2)	−0.004 (2)
C8	0.042 (3)	0.036 (3)	0.038 (3)	−0.006 (2)	0.010 (2)	0.000 (2)
C9	0.054 (3)	0.040 (3)	0.037 (3)	−0.004 (2)	0.013 (2)	0.003 (2)
C10	0.038 (3)	0.034 (3)	0.050 (3)	0.002 (2)	0.008 (2)	0.004 (2)
C11	0.046 (3)	0.050 (3)	0.049 (3)	0.002 (3)	0.002 (3)	0.005 (3)
C12	0.060 (4)	0.057 (4)	0.051 (3)	0.003 (3)	−0.004 (3)	0.005 (3)
C13	0.040 (3)	0.045 (3)	0.043 (3)	−0.006 (2)	0.013 (2)	−0.004 (2)
C14	0.062 (4)	0.041 (3)	0.057 (3)	−0.005 (3)	0.028 (3)	−0.002 (3)
C15	0.065 (4)	0.050 (3)	0.069 (4)	0.011 (3)	0.021 (3)	0.006 (3)
C16	0.057 (4)	0.057 (4)	0.062 (4)	0.009 (3)	−0.001 (3)	0.010 (3)
C17	0.055 (3)	0.049 (3)	0.054 (3)	0.002 (3)	0.030 (3)	−0.005 (3)
C18	0.038 (3)	0.047 (3)	0.071 (4)	0.000 (2)	0.027 (3)	0.003 (3)
C19	0.046 (3)	0.062 (4)	0.079 (4)	0.009 (3)	0.018 (3)	0.004 (3)
C20	0.098 (6)	0.097 (6)	0.101 (6)	0.006 (5)	0.044 (4)	−0.007 (5)
C21	0.101 (4)	0.055 (3)	0.063 (3)	0.005 (3)	0.024 (3)	0.005 (3)
C22	0.107 (4)	0.083 (4)	0.079 (4)	−0.011 (4)	0.020 (3)	0.019 (3)
C23	0.117 (4)	0.096 (4)	0.083 (4)	−0.012 (4)	0.015 (4)	0.014 (4)
C24	0.113 (3)	0.125 (4)	0.102 (4)	0.002 (3)	0.016 (3)	0.025 (3)
C25	0.113 (3)	0.125 (4)	0.102 (4)	0.002 (3)	0.016 (3)	0.025 (3)
C26	0.120 (6)	0.121 (6)	0.094 (6)	−0.016 (5)	0.035 (5)	0.035 (5)
C26'	0.120 (6)	0.121 (6)	0.094 (6)	−0.016 (5)	0.035 (5)	0.035 (5)
C27	0.049 (3)	0.040 (3)	0.038 (3)	0.000 (2)	0.010 (2)	0.000 (2)
C28	0.052 (3)	0.055 (4)	0.052 (3)	−0.003 (3)	0.015 (3)	0.005 (3)
C29	0.070 (4)	0.069 (4)	0.062 (4)	0.014 (3)	0.032 (3)	0.018 (3)
C30	0.107 (6)	0.108 (6)	0.113 (6)	0.002 (5)	0.076 (5)	0.024 (5)
C31	0.116 (6)	0.093 (5)	0.049 (4)	0.015 (5)	0.026 (4)	0.007 (4)
C32	0.086 (5)	0.064 (4)	0.089 (5)	0.006 (4)	0.043 (4)	−0.015 (4)
C33	0.080 (4)	0.066 (4)	0.053 (3)	−0.005 (3)	0.030 (3)	0.000 (3)
C34	0.057 (4)	0.056 (4)	0.068 (4)	0.002 (3)	0.029 (3)	−0.006 (3)
C35	0.030 (3)	0.061 (4)	0.058 (3)	−0.002 (3)	0.011 (2)	0.001 (3)
C36	0.035 (3)	0.063 (4)	0.047 (3)	−0.004 (3)	0.007 (2)	−0.002 (3)
C37	0.045 (3)	0.056 (3)	0.058 (3)	−0.001 (3)	0.026 (3)	0.004 (3)
C38	0.057 (4)	0.075 (4)	0.072 (4)	0.017 (3)	0.018 (3)	0.007 (3)
C39	0.091 (5)	0.100 (6)	0.086 (5)	0.001 (4)	0.046 (4)	−0.035 (4)
C40	0.094 (6)	0.149 (8)	0.044 (3)	−0.008 (5)	0.019 (3)	0.020 (4)

Geometric parameters (Å, °)

O1—C9	1.357 (6)	C22—C23	1.499 (11)
O1—C1	1.467 (7)	C22—H22A	0.9700
O2—C7	1.374 (6)	C22—H22B	0.9700
O2—C10	1.427 (6)	C23—C24	1.286 (10)
O3—C12	1.225 (6)	C23—H23	0.9300

O4—C5	1.372 (6)	C24—C26	1.497 (13)
O4—C40	1.416 (8)	C24—C25	1.516 (11)
O5—C13	1.424 (6)	C24—C26'	1.592 (18)
O5—C17	1.446 (6)	C25—H25A	0.9600
O6—C14	1.207 (6)	C25—H25B	0.9600
O7—C34	1.332 (7)	C25—H25C	0.9600
O7—C39	1.421 (8)	C26—H26A	0.9600
O8—C34	1.198 (7)	C26—H26B	0.9600
C1—C2	1.478 (8)	C26—H26C	0.9600
C1—C21	1.522 (9)	C26'—H26D	0.9600
C1—C20	1.524 (10)	C26'—H26E	0.9600
C2—C3	1.333 (8)	C26'—H26F	0.9600
C2—H2	0.9300	C27—C28	1.512 (8)
C3—C4	1.448 (8)	C27—H27A	0.9700
C3—H3	0.9300	C27—H27B	0.9700
C4—C5	1.374 (7)	C28—C29	1.326 (8)
C4—C9	1.403 (7)	C28—H28	0.9300
C5—C6	1.404 (7)	C29—C30	1.481 (10)
C6—C7	1.410 (6)	C29—C31	1.488 (9)
C6—C12	1.457 (7)	C30—H30A	0.9600
C7—C8	1.379 (6)	C30—H30B	0.9600
C8—C9	1.372 (7)	C30—H30C	0.9600
C8—C27	1.508 (7)	C31—H31A	0.9600
C10—C11	1.493 (7)	C31—H31B	0.9600
C10—C13	1.532 (7)	C31—H31C	0.9600
C10—C18	1.570 (7)	C32—H32A	0.9600
C11—C16	1.338 (7)	C32—H32B	0.9600
C11—C12	1.468 (8)	C32—H32C	0.9600
C13—C37	1.522 (7)	C33—H33A	0.9600
C13—C14	1.541 (8)	C33—H33B	0.9600
C14—C15	1.523 (8)	C33—H33C	0.9600
C15—C16	1.494 (8)	C34—C35	1.470 (8)
C15—C19	1.539 (9)	C35—C36	1.337 (7)
C15—H15	0.9800	C35—C38	1.508 (8)
C16—H16	0.9300	C36—C37	1.484 (7)
C17—C33	1.521 (8)	C36—H36	0.9300
C17—C32	1.534 (8)	C37—H37A	0.9700
C17—C18	1.543 (8)	C37—H37B	0.9700
C18—C19	1.537 (8)	C38—H38A	0.9600
C18—H18	0.9800	C38—H38B	0.9600
C19—H19A	0.9700	C38—H38C	0.9600
C19—H19B	0.9700	C39—H39A	0.9600
C20—H20A	0.9600	C39—H39B	0.9600
C20—H20B	0.9600	C39—H39C	0.9600
C20—H20C	0.9600	C40—H40A	0.9600
C21—C22	1.524 (10)	C40—H40B	0.9600
C21—H21A	0.9700	C40—H40C	0.9600
C21—H21B	0.9700		
C9—O1—C1	122.6 (4)	C21—C22—H22A	108.9

C7—O2—C10	122.5 (3)	C23—C22—H22B	108.9
C5—O4—C40	115.9 (5)	C21—C22—H22B	108.9
C13—O5—C17	109.3 (4)	H22A—C22—H22B	107.7
C34—O7—C39	117.9 (5)	C24—C23—C22	129.9 (9)
O1—C1—C2	112.1 (5)	C24—C23—H23	115.1
O1—C1—C21	104.0 (5)	C22—C23—H23	115.1
C2—C1—C21	111.4 (5)	C23—C24—C26	132.5 (13)
O1—C1—C20	107.0 (5)	C23—C24—C25	121.9 (9)
C2—C1—C20	111.0 (6)	C26—C24—C25	105.6 (12)
C21—C1—C20	111.2 (6)	C23—C24—C26'	101.9 (16)
C3—C2—C1	123.2 (5)	C26—C24—C26'	30.6 (11)
C3—C2—H2	118.4	C25—C24—C26'	136.1 (17)
C1—C2—H2	118.4	C24—C25—H25A	109.5
C2—C3—C4	120.5 (5)	C24—C25—H25B	109.5
C2—C3—H3	119.7	H25A—C25—H25B	109.5
C4—C3—H3	119.7	C24—C25—H25C	109.5
C5—C4—C9	117.7 (5)	H25A—C25—H25C	109.5
C5—C4—C3	124.0 (5)	H25B—C25—H25C	109.5
C9—C4—C3	118.3 (5)	C24—C26—H26A	109.5
O4—C5—C4	116.6 (5)	C24—C26—H26B	109.5
O4—C5—C6	121.3 (5)	C24—C26—H26C	109.5
C4—C5—C6	122.0 (4)	C24—C26'—H26D	109.5
C5—C6—C7	117.1 (4)	C24—C26'—H26E	109.5
C5—C6—C12	123.6 (4)	H26D—C26'—H26E	109.5
C7—C6—C12	119.2 (5)	C24—C26'—H26F	109.5
O2—C7—C8	114.6 (4)	H26D—C26'—H26F	109.5
O2—C7—C6	122.7 (4)	H26E—C26'—H26F	109.5
C8—C7—C6	122.6 (4)	C8—C27—C28	112.0 (4)
C9—C8—C7	117.2 (4)	C8—C27—H27A	109.2
C9—C8—C27	120.2 (4)	C28—C27—H27A	109.2
C7—C8—C27	122.6 (4)	C8—C27—H27B	109.2
O1—C9—C8	115.9 (4)	C28—C27—H27B	109.2
O1—C9—C4	120.7 (5)	H27A—C27—H27B	107.9
C8—C9—C4	123.3 (5)	C29—C28—C27	128.5 (5)
O2—C10—C11	114.1 (4)	C29—C28—H28	115.7
O2—C10—C13	111.9 (4)	C27—C28—H28	115.7
C11—C10—C13	109.5 (4)	C28—C29—C30	120.4 (7)
O2—C10—C18	107.7 (4)	C28—C29—C31	123.8 (6)
C11—C10—C18	115.6 (4)	C30—C29—C31	115.7 (6)
C13—C10—C18	96.8 (4)	C29—C30—H30A	109.5
C16—C11—C12	122.6 (5)	C29—C30—H30B	109.5
C16—C11—C10	114.8 (5)	H30A—C30—H30B	109.5
C12—C11—C10	122.0 (5)	C29—C30—H30C	109.5
O3—C12—C6	123.9 (5)	H30A—C30—H30C	109.5
O3—C12—C11	119.3 (5)	H30B—C30—H30C	109.5
C6—C12—C11	116.7 (4)	C29—C31—H31A	109.5
O5—C13—C37	110.0 (4)	C29—C31—H31B	109.5
O5—C13—C10	105.1 (4)	H31A—C31—H31B	109.5
C37—C13—C10	116.9 (4)	C29—C31—H31C	109.5

O5—C13—C14	108.1 (4)	H31A—C31—H31C	109.5
C37—C13—C14	110.3 (4)	H31B—C31—H31C	109.5
C10—C13—C14	106.0 (4)	C17—C32—H32A	109.5
O6—C14—C15	123.0 (5)	C17—C32—H32B	109.5
O6—C14—C13	122.8 (5)	H32A—C32—H32B	109.5
C15—C14—C13	114.3 (5)	C17—C32—H32C	109.5
C16—C15—C14	102.4 (5)	H32A—C32—H32C	109.5
C16—C15—C19	105.9 (5)	H32B—C32—H32C	109.5
C14—C15—C19	109.1 (5)	C17—C33—H33A	109.5
C16—C15—H15	112.9	C17—C33—H33B	109.5
C14—C15—H15	112.9	H33A—C33—H33B	109.5
C19—C15—H15	112.9	C17—C33—H33C	109.5
C11—C16—C15	115.0 (5)	H33A—C33—H33C	109.5
C11—C16—H16	122.5	H33B—C33—H33C	109.5
C15—C16—H16	122.5	O8—C34—O7	120.5 (6)
O5—C17—C33	108.3 (4)	O8—C34—C35	127.4 (6)
O5—C17—C32	106.8 (5)	O7—C34—C35	112.0 (6)
C33—C17—C32	108.0 (5)	C36—C35—C34	122.2 (5)
O5—C17—C18	104.9 (4)	C36—C35—C38	120.9 (5)
C33—C17—C18	111.9 (5)	C34—C35—C38	116.9 (5)
C32—C17—C18	116.5 (5)	C35—C36—C37	129.3 (5)
C19—C18—C17	116.1 (5)	C35—C36—H36	115.3
C19—C18—C10	106.6 (4)	C37—C36—H36	115.3
C17—C18—C10	102.6 (4)	C36—C37—C13	115.4 (4)
C19—C18—H18	110.4	C36—C37—H37A	108.4
C17—C18—H18	110.4	C13—C37—H37A	108.4
C10—C18—H18	110.4	C36—C37—H37B	108.4
C18—C19—C15	110.4 (5)	C13—C37—H37B	108.4
C18—C19—H19A	109.6	H37A—C37—H37B	107.5
C15—C19—H19A	109.6	C35—C38—H38A	109.5
C18—C19—H19B	109.6	C35—C38—H38B	109.5
C15—C19—H19B	109.6	H38A—C38—H38B	109.5
H19A—C19—H19B	108.1	C35—C38—H38C	109.5
C1—C20—H20A	109.5	H38A—C38—H38C	109.5
C1—C20—H20B	109.5	H38B—C38—H38C	109.5
H20A—C20—H20B	109.5	O7—C39—H39A	109.5
C1—C20—H20C	109.5	O7—C39—H39B	109.5
H20A—C20—H20C	109.5	H39A—C39—H39B	109.5
H20B—C20—H20C	109.5	O7—C39—H39C	109.5
C1—C21—C22	114.3 (6)	H39A—C39—H39C	109.5
C1—C21—H21A	108.7	H39B—C39—H39C	109.5
C22—C21—H21A	108.7	O4—C40—H40A	109.5
C1—C21—H21B	108.7	O4—C40—H40B	109.5
C22—C21—H21B	108.7	H40A—C40—H40B	109.5
H21A—C21—H21B	107.6	O4—C40—H40C	109.5
C23—C22—C21	113.5 (6)	H40A—C40—H40C	109.5
C23—C22—H22A	108.9	H40B—C40—H40C	109.5
C9—O1—C1—C2	19.3 (8)	C11—C10—C13—C37	73.1 (5)
C9—O1—C1—C21	139.7 (5)	C18—C10—C13—C37	−166.6 (4)

C9—O1—C1—C20	−102.6 (6)	O2—C10—C13—C14	−177.8 (4)
O1—C1—C2—C3	−14.1 (9)	C11—C10—C13—C14	−50.3 (5)
C21—C1—C2—C3	−130.0 (7)	C18—C10—C13—C14	70.0 (4)
C20—C1—C2—C3	105.5 (8)	O5—C13—C14—O6	−74.4 (6)
C1—C2—C3—C4	4.2 (10)	C37—C13—C14—O6	45.8 (7)
C2—C3—C4—C5	−176.9 (6)	C10—C13—C14—O6	173.3 (5)
C2—C3—C4—C9	2.2 (9)	O5—C13—C14—C15	105.3 (5)
C40—O4—C5—C4	108.0 (6)	C37—C13—C14—C15	−134.4 (5)
C40—O4—C5—C6	−76.5 (7)	C10—C13—C14—C15	−6.9 (6)
C9—C4—C5—O4	176.0 (5)	O6—C14—C15—C16	−120.0 (6)
C3—C4—C5—O4	−5.0 (8)	C13—C14—C15—C16	60.2 (6)
C9—C4—C5—C6	0.5 (8)	O6—C14—C15—C19	128.1 (6)
C3—C4—C5—C6	179.6 (5)	C13—C14—C15—C19	−51.7 (6)
O4—C5—C6—C7	−175.6 (5)	C12—C11—C16—C15	174.8 (5)
C4—C5—C6—C7	−0.3 (8)	C10—C11—C16—C15	3.4 (8)
O4—C5—C6—C12	2.0 (8)	C14—C15—C16—C11	−59.6 (7)
C4—C5—C6—C12	177.2 (5)	C19—C15—C16—C11	54.7 (7)
C10—O2—C7—C8	−168.6 (4)	C13—O5—C17—C33	−123.1 (5)
C10—O2—C7—C6	14.4 (7)	C13—O5—C17—C32	120.8 (5)
C5—C6—C7—O2	178.6 (5)	C13—O5—C17—C18	−3.4 (5)
C12—C6—C7—O2	0.9 (7)	O5—C17—C18—C19	91.1 (5)
C5—C6—C7—C8	1.8 (7)	C33—C17—C18—C19	−151.7 (5)
C12—C6—C7—C8	−175.9 (5)	C32—C17—C18—C19	−26.8 (7)
O2—C7—C8—C9	179.7 (4)	O5—C17—C18—C10	−24.8 (5)
C6—C7—C8—C9	−3.3 (7)	C33—C17—C18—C10	92.4 (5)
O2—C7—C8—C27	−1.5 (7)	C32—C17—C18—C10	−142.7 (5)
C6—C7—C8—C27	175.6 (5)	O2—C10—C18—C19	162.8 (4)
C1—O1—C9—C8	169.3 (5)	C11—C10—C18—C19	33.8 (6)
C1—O1—C9—C4	−14.7 (8)	C13—C10—C18—C19	−81.6 (5)
C7—C8—C9—O1	179.4 (4)	O2—C10—C18—C17	−74.7 (5)
C27—C8—C9—O1	0.5 (7)	C11—C10—C18—C17	156.4 (4)
C7—C8—C9—C4	3.5 (8)	C13—C10—C18—C17	40.9 (4)
C27—C8—C9—C4	−175.4 (5)	C17—C18—C19—C15	−89.9 (6)
C5—C4—C9—O1	−177.9 (5)	C10—C18—C19—C15	23.7 (6)
C3—C4—C9—O1	3.0 (8)	C16—C15—C19—C18	−68.9 (6)
C5—C4—C9—C8	−2.2 (8)	C14—C15—C19—C18	40.7 (6)
C3—C4—C9—C8	178.7 (5)	O1—C1—C21—C22	−63.2 (7)
C7—O2—C10—C11	−19.5 (6)	C2—C1—C21—C22	57.6 (8)
C7—O2—C10—C13	105.5 (5)	C20—C1—C21—C22	−178.1 (6)
C7—O2—C10—C18	−149.3 (4)	C1—C21—C22—C23	−177.0 (6)
O2—C10—C11—C16	−177.5 (5)	C21—C22—C23—C24	−111.6 (10)
C13—C10—C11—C16	56.3 (6)	C22—C23—C24—C26	−177.2 (11)
C18—C10—C11—C16	−51.7 (7)	C22—C23—C24—C25	5.6 (15)
O2—C10—C11—C12	11.1 (7)	C22—C23—C24—C26'	−176.9 (15)
C13—C10—C11—C12	−115.1 (5)	C9—C8—C27—C28	83.4 (6)
C18—C10—C11—C12	136.8 (5)	C7—C8—C27—C28	−95.4 (6)
C5—C6—C12—O3	−4.4 (9)	C8—C27—C28—C29	157.5 (6)
C7—C6—C12—O3	173.1 (6)	C27—C28—C29—C30	177.5 (6)
C5—C6—C12—C11	173.9 (5)	C27—C28—C29—C31	−1.7 (10)

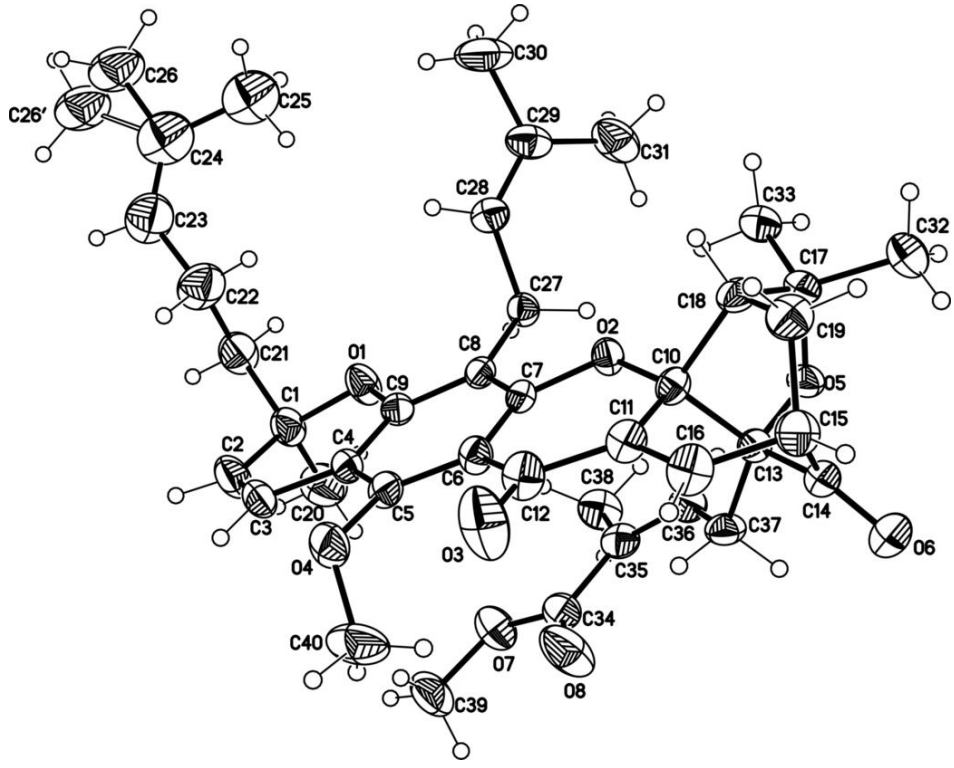
C7—C6—C12—C11	−8.6 (7)	C39—O7—C34—O8	−1.5 (9)
C16—C11—C12—O3	9.9 (9)	C39—O7—C34—C35	−178.6 (5)
C10—C11—C12—O3	−179.3 (6)	O8—C34—C35—C36	−4.4 (10)
C16—C11—C12—C6	−168.5 (6)	O7—C34—C35—C36	172.5 (5)
C10—C11—C12—C6	2.3 (8)	O8—C34—C35—C38	177.9 (6)
C17—O5—C13—C37	158.2 (4)	O7—C34—C35—C38	−5.2 (7)
C17—O5—C13—C10	31.5 (5)	C34—C35—C36—C37	5.3 (8)
C17—O5—C13—C14	−81.3 (5)	C38—C35—C36—C37	−177.1 (5)
O2—C10—C13—O5	67.9 (5)	C35—C36—C37—C13	−140.1 (6)
C11—C10—C13—O5	−164.6 (4)	O5—C13—C37—C36	−45.3 (6)
C18—C10—C13—O5	−44.3 (4)	C10—C13—C37—C36	74.4 (6)
O2—C10—C13—C37	−54.4 (6)	C14—C13—C37—C36	−164.4 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O8^{i}	0.93	2.53	3.400 (8)	156
C31—H31C···O6^{ii}	0.96	2.58	3.535 (8)	176

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

Fig. 1


C26
C26'
C25
C24
C23
C22
C21
C1
C2
C3
C20
C4
C5
O4
C40
C39
O7
O8
C34
C35
C36
C37
C38
O3
C12
C6
C9
O1
C8
C7
O2
C10
C11
C16
C13
C14
O6
C15
O5
C19
C18
C17
C32
C33
C27
C28
C29
C31
C30

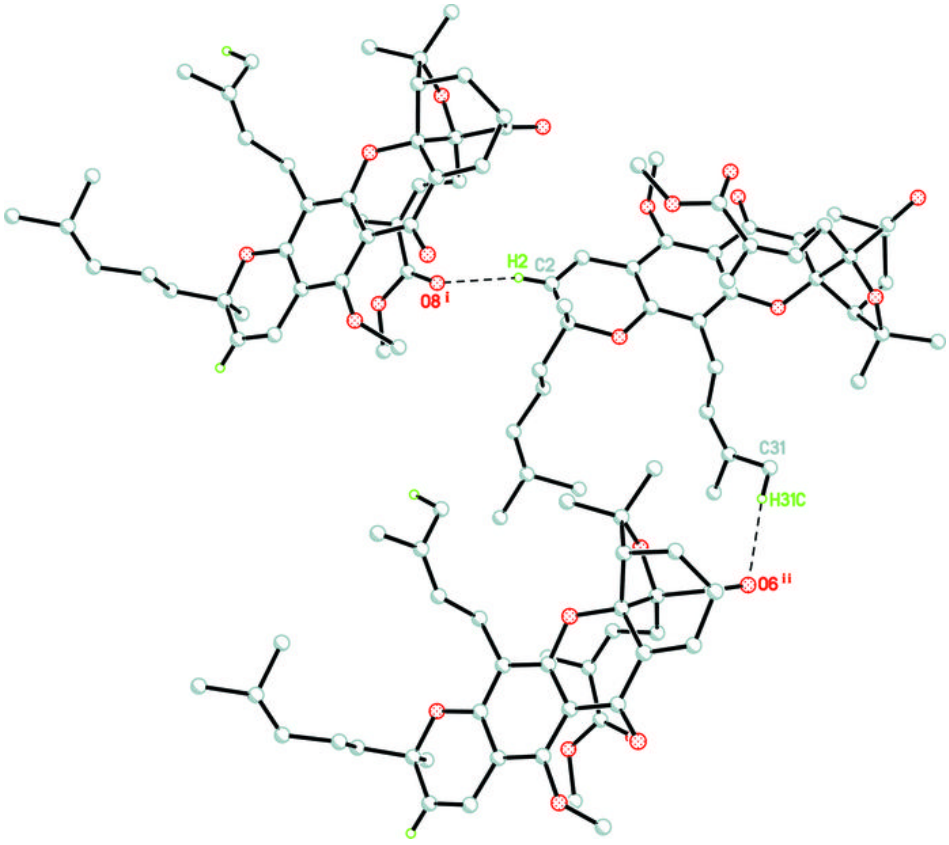



Fig. 2