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Glucosilate kaurenoic acid sesquihydrate

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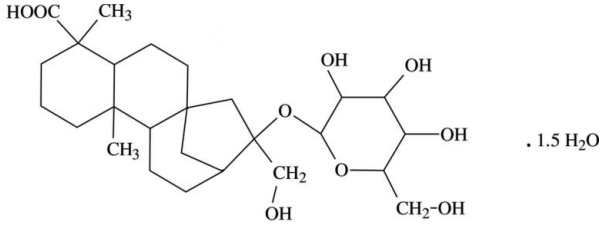
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.014 Å; H-atom completeness 96%; disorder in solvent or counterion; R factor = 0.071; wR factor = 0.219; data-to-parameter ratio = 7.3.

The title compound, $C_{26}H_{42}O_9\cdot1.5H_2O$, was extracted from the leaves and flowers of *Ageratina vacciniaefolia*, a Colombian native plant, which is called 'chilca' by locals. The molecule consists a kauranol group connected to a glucopyranosyl group by an O-atom bridge adjacent to a β-type anomeric center. In the crystal structure, intermolecular O—H···O hydrogen bonds link molecules to form a three-dimensional network. One solvent water molecule lies on a crystallographic twofold axis, while another is disordered over three sites with equal occupancies.

Related literature

The title molecule belongs to the diterpene family, which exhibit physiological effects as gibberellic, antitumoral and antiinflammatory activities (Guisalberti, 1997). Similar structures were found in the Cambridge Structural Database (Version 5.28; Allen, 2002) For the industrial production, see: Villalobos (1994). For puckering analyses, see: Cremer & Pople (1975); Nardelli (1983).



Experimental

Crystal data

$C_{26}H_{42}O_9\cdot1.5H_2O$
M_r = 525.62
Monoclinic, $C2$
a = 33.36 (2) Å
b = 7.365 (4) Å
c = 11.076 (8) Å
β = 100.52 (5)°
V = 2675 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.1 mm^{-1}
T = 293 (2) K
0.4 × 0.2 × 0.05 mm

Data collection

Rigaku AFC6 diffractometer
Absorption correction: none
3120 measured reflections
2559 independent reflections
1222 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
3 standard reflections
every 147 reflections
intensity decay: <0.1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.219
S = 1.00
2559 reflections
350 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O17—H17···O21	0.83 (2)	2.14 (7)	2.753 (9)	131 (7)
O17—H17···O26	0.83 (2)	2.29 (6)	3.033 (11)	149 (9)
O19—H19···O22^{i}	0.84 (6)	1.82 (5)	2.641 (13)	171.9 (3)
O22—H22···O17^{ii}	0.81 (2)	2.17 (4)	2.715 (6)	125.1 (3)
O24—H24···O20^{iii}	0.82 (5)	1.94 (5)	2.747 (6)	164.9 (3)
O26—H26···O1W	0.82	2.27	2.757 (15)	118
O1W—H1W···O24^{iv}	0.86 (5)	1.96 (4)	2.804 (6)	169.4 (3)

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

Data collection: *MSC/AFC6S Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC6S Diffractometer Control Software*; data reduction: *MSC/AFC6S Diffractometer Control Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-PC* (Sheldrick, 1994); software used to prepare material for publication: *SHELXTL-PC* and *PARST* (Nardelli, 1983).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2520).

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supplementary materials

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Glucosilate kaurenoic acid sesquihydrate

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Comment

The molecule contains kauranol and glucopyranosyl moieties (Fig. 1) and belongs to the diterpene family, which present physiological effects such as gibberellic, antitumoral and antiinflammatory activities. The kauranol group contains four fused rings, three of them are six-membered rings in chair conformations and one is a five-membered ring in an envelope conformation. Atoms C1/C2/C4/C5 determine a chair plane for the first ring (max. dev. from the l.s. plane: C2 0.000 (9) Å) and C3 and C10 located −0.685 (9)Å and 0.637 (9)Å from the mean plane respectively (ring puckering parameters are q_2=0.049 (9) Å, q_3=0.563 (9)Å and φ_2=127 (10)° (Cremer & Pople, 1975)). Atoms C6/C7/C9/C10 determine the chair plane for the second ring (max. dev. from the l.s. plane: C7 − 0.023 (9) Å), and C5 and C8 are located −0.718 (8)Å and 0.591 (8)Å from the mean plane respectively (ring puckering parameters are q_2= 0.101 (8) Å, q_3=0.563 (8)%A and φ_2=19 (4)°). The shared bond between these two fused rings does not form any part of either chair planes. A different situation is observed for the third ring, where the chair plane is determined by atoms C8/C9/C12/C13 (max. dev. from the l.s. plane: C13 − 0.022 (8) Å) and C11 and C14 located at −0.436 (8)Å and 0.859 (8)Å from the mean plane respectively (ring puckering parameters are q_2=0.270 (8) Å, q_3=-0.558 (8)Å and φ_2=112 (2)°). In this case, the shared bond between the last two six-membered fused rings is part of each of their chair planes forming an angle of 56.3 (3)° between them. The five-membered ring conformation can be described as an envelope, with C8/C13/C15/C16 lying almost on the same plane (max. dev. from the l.s. plane: C16 − 0.020 (9) Å) and C14 located at 0.738 (8)Å from this mean plane (ring puckering parameters are q_2=0.022 (8) Å, and φ_2=-1.7 (3)°). The angle between the C8/C9/C12/C13 and C8/C13/C15/C16 mean planes has a value of 69.0 (3)°. The kauranol group is connected to the glucopyranosyl group by means of atom O16 bonded to atom C1' atom, which in turn, is connected to a hydrogen atom. This anomeric center is beta type (torsional angle: O22—C2'-C1'-O16 66.6 (9)°).

A search using the CSD (ConQuest 1.9, CSD version 5.28, Allen, 2002) was carried out to find similar kauranol molecules, containing the four fused rings and CH_3 and COOH groups located at C10 and C4 respectively and this gave 37 hits. The CH_3 and COOH groups, located at C10 and C4 respectively are on the same side of the C1/C2/C4/C5 mean plane and the non-bonded torsion angle C19—C4···C10—C20 is 2.3 (9)°, and it is not very different to those values found in the 37 structures found in the CSD (mean value 1.8°, sample SD 0.001°). However, the angle formed by the mean plane determined by O19/O20/C19/C4 (max. dev. from the l.s. plane: C19 0.020 (8) Å) and that formed by atoms C19/C4/C10/C20 (max. dev. from the l.s. plane: C10 0.011 (8) Å) is 69.8 (9)°, very different from those values found in these 37 structures (mean value 81°, sample SD 8°), where only the fragments corresponding to FICDEB, VIMYIZ and VIFDAP refcodes have angle values below 70°.

The crystal structure is stabilized by an extensive H-bond scheme (Table 1). Two intra H-bond are observed (see Fig. 1), where O17 act as donor, sharing H17 between O21 and O26, in a bifurcated H-bond. The water molecule, O1W, is a bridge linking four different molecules *via* hydrogen bonds. O1W act as donor in two of these contacts and as receptor in another two, *via* H1W and H26 respectively. O24 connects different molecules through H24, and finally, O22 is a bridge, connecting two different molecules, acting as a donnor and a receptor, *via* H22 and H19 respectively.

Experimental

The kauranoic acid was obtained after processing, extraction and purification from leaves and inflorescence of Ageratina vacciniaefolia. The compound was recrystallized from metanol solution.

Refinement

Due to the absence of any significant anomalous scatterers in the molecule, 401 Friedel pairs were merged before the final refinement. The absolute configuration is unknown and the enantiomer was arbitrarilty assigned. Three significative peaks in the difference Fourier maps were modeled as a disordered water molecule: O2W1, O2W2 and O2W3 atoms constitute this model and were considered with occupancy fixed at 1/3 and freely refined isotropic displacement parameters. No H atoms were assigned to this disordered water molecule but they are included in the molecular formula. Some hydroxy H atoms, those bonded to O17, O19, O22 and O24 were found in difference Fourier maps and refined subject to O—H restraint 0.82 (4)Å and C—O—H restraint 110 (5)°. Water atom H1W also was found in difference Fourier maps and refined subject to O—H restraint 0.85Å and C—O—H restraint 109 (5)°. All other H atoms were treated as riding atoms, located at idealized positions, with C—H distances of 0.96 (CH_3), 0.97 (CH_2) or 0.98Å (CH), and O—H distances of 0.82 Å. All H atoms were assigned isotropic displacement parameters with U_{iso} (H) of 1.2 times of the U_{eq} of the parent non-H atoms, for CH_2 and CH, and 1.5 times, for CH_3 and OH.

Figures

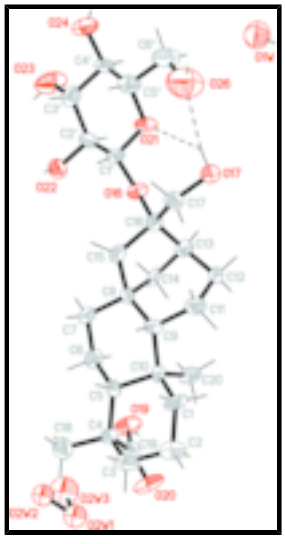

Fig. 1. View of the molecular structure showing the numbering scheme used and displacement ellipsoids drawn at the 50% probability level. (Intramolecular H-bonds are depicted as dashed lines).

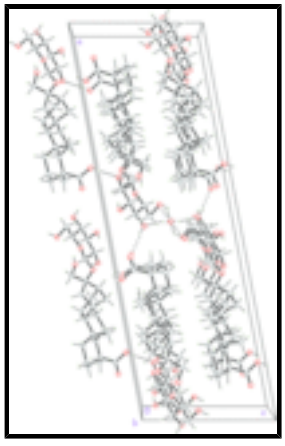

Fig. 2. Packing diagram showing part of the intermolecular hydrogen bonding scheme. (Hydrogen bonds represented as dashed lines). Disordered water molecule O2W has been omited.

(I)

Crystal data

$C_{26}H_{42}O_9 \cdot 1.5H_2O$	$F_{000} = 1140$
$M_r = 525.62$	$D_x = 1.305$ Mg m^{-3}
Monoclinic, *C*2	Melting point: 240 K
Hall symbol: C 2y	Mo *K*α radiation λ = 0.71073 Å
a = 33.36 (2) Å	Cell parameters from 20 reflections
b = 7.365 (4) Å	θ = 14–25º
c = 11.076 (8) Å	μ = 0.1 mm^{-1}
β = 100.52 (5)º	*T* = 293 (2) K
V = 2675 (3) Å^3	Plate, colourless
Z = 4	0.4 × 0.2 × 0.05 mm

Data collection

AFC6 Rigaku Diffractometer	$\theta_{max} = 25º$
Radiation source: fine-focus sealed tube	$\theta_{min} = 1.9º$
Monochromator: graphite	*h* = −1→39
ω scans	*k* = −1→8
Absorption correction: none	*l* = −13→12
3120 measured reflections	3 standard reflections
2559 independent reflections	every 147 reflections
1222 reflections with $I > 2\sigma(I)$	intensity decay: <0.1%
$R_{int} = 0.064$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.071$

$wR(F^2) = 0.219$

$S = 1.00$

2559 reflections

350 parameters

12 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.1157P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.55$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ 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 $F^2 > 2$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)
C1	0.3322 (2)	0.6045 (17)	0.3825 (9)	0.044 (3)	
H1A	0.3217	0.7276	0.3771	0.053*	
H1B	0.3296	0.5566	0.4622	0.053*	
C2	0.3769 (3)	0.6100 (18)	0.3739 (9)	0.048 (3)	
H2A	0.3802	0.6690	0.2979	0.057*	
H2B	0.3917	0.6803	0.4417	0.057*	
C3	0.3943 (3)	0.4209 (17)	0.3775 (9)	0.047 (3)	
H3A	0.3918	0.3639	0.4547	0.056*	
H3B	0.4230	0.4278	0.3735	0.056*	
C4	0.3724 (2)	0.3034 (16)	0.2705 (8)	0.034 (2)	
C5	0.3262 (2)	0.2991 (15)	0.2791 (8)	0.032 (2)	
H5	0.3260	0.2495	0.3611	0.039*	
C6	0.2987 (3)	0.1697 (15)	0.1935 (9)	0.038 (3)	
H6A	0.3128	0.0556	0.1883	0.046*	
H6B	0.2924	0.2218	0.1118	0.046*	
C7	0.2591 (3)	0.1356 (14)	0.2422 (9)	0.036 (2)	
H7A	0.2420	0.0534	0.1868	0.043*	
H7B	0.2657	0.0769	0.3217	0.043*	
C8	0.2353 (2)	0.3089 (14)	0.2549 (8)	0.030 (2)	
C9	0.2638 (2)	0.4573 (14)	0.3251 (8)	0.030 (2)	
H9	0.2712	0.4105	0.4091	0.036*	
C10	0.3062 (3)	0.4892 (14)	0.2824 (9)	0.033 (2)	

C11	0.2397 (3)	0.6293 (16)	0.3377 (9)	0.040 (3)
H11A	0.2587	0.7303	0.3491	0.048*
H11B	0.2283	0.6191	0.4118	0.048*
C12	0.2051 (3)	0.6764 (14)	0.2316 (8)	0.033 (2)
H12A	0.2165	0.7372	0.1679	0.040*
H12B	0.1867	0.7607	0.2610	0.040*
C13	0.1804 (3)	0.5096 (15)	0.1750 (8)	0.035 (3)
H13	0.1587	0.5453	0.1071	0.042*
C14	0.2104 (3)	0.3775 (14)	0.1314 (7)	0.032 (2)
H14A	0.2277	0.4393	0.0829	0.038*
H14B	0.1963	0.2787	0.0837	0.038*
C15	0.1999 (2)	0.2679 (14)	0.3255 (8)	0.034 (2)
H15A	0.2085	0.2927	0.4124	0.041*
H15B	0.1919	0.1414	0.3156	0.041*
C16	0.1635 (3)	0.3938 (14)	0.2700 (7)	0.030 (2)
C17	0.1461 (3)	0.4952 (15)	0.3674 (8)	0.036 (3)
H17A	0.1346	0.4081	0.4173	0.043*
H17B	0.1680	0.5582	0.4206	0.043*
C18	0.3905 (3)	0.1080 (18)	0.2865 (11)	0.059 (3)
H18A	0.3795	0.0364	0.2156	0.088*
H18B	0.3834	0.0533	0.3584	0.088*
H18C	0.4196	0.1140	0.2951	0.088*
C19	0.3834 (3)	0.3714 (15)	0.1500 (8)	0.038 (3)
C20	0.3017 (3)	0.5934 (17)	0.1613 (8)	0.038 (3)
H20A	0.2840	0.6957	0.1636	0.057*
H20B	0.2902	0.5148	0.0947	0.057*
H20C	0.3280	0.6349	0.1496	0.057*
C1'	0.1039 (2)	0.1856 (14)	0.2468 (8)	0.029 (2)
H1'	0.1172	0.1391	0.3270	0.035*
C2'	0.0868 (3)	0.0309 (15)	0.1649 (9)	0.038 (3)
H2'	0.0771	0.0807	0.0827	0.045*
C3'	0.0494 (3)	−0.0530 (15)	0.2098 (9)	0.042 (3)
H3'	0.0592	−0.1152	0.2879	0.050*
C4'	0.0182 (2)	0.0872 (16)	0.2302 (8)	0.035 (3)
H4'	0.0055	0.1413	0.1516	0.042*
C5'	0.0401 (3)	0.2343 (16)	0.3160 (9)	0.042 (3)
H5'	0.0521	0.1777	0.3943	0.051*
C6'	0.0142 (3)	0.3876 (19)	0.3406 (11)	0.061 (3)
H6'1	−0.0083	0.3457	0.3777	0.073*
H6'2	0.0034	0.4530	0.2657	0.073*
O16	0.13182 (15)	0.2820 (10)	0.1924 (5)	0.0346 (17)
O17	0.11503 (18)	0.6252 (11)	0.3182 (7)	0.049 (2)
H17	0.0934 (9)	0.574 (9)	0.322 (10)	0.073*
O19	0.35864 (19)	0.3112 (12)	0.0513 (6)	0.052 (2)
H19	0.366 (2)	0.346 (15)	−0.013 (3)	0.078*
O20	0.4128 (2)	0.4628 (13)	0.1414 (6)	0.060 (2)
O21	0.07224 (17)	0.3076 (11)	0.2613 (6)	0.0426 (19)
O22	0.11692 (18)	−0.1015 (10)	0.1549 (6)	0.0413 (18)
H22	0.1270 (18)	−0.128 (5)	0.225 (3)	0.062*

O23	0.0315 (2)	−0.1871 (13)	0.1205 (8)	0.088 (3)	
H23	0.0489	−0.2615	0.1107	0.131*	
O24	−0.0117 (2)	0.0061 (12)	0.2867 (6)	0.052 (2)	
H24	−0.0322 (14)	0.000 (17)	0.233 (5)	0.078*	
O26	0.0425 (3)	0.5035 (16)	0.4271 (10)	0.109 (4)	
H26	0.0326	0.5234	0.4884	0.164*	
O1W	0.0000	0.7924 (18)	0.5000	0.063 (3)	
H1W	0.000 (4)	0.858 (6)	0.436 (3)	0.094*	
O2W1	0.4565 (6)	0.129 (4)	0.0860 (19)	0.060 (6)*	0.33
O2W2	0.4415 (6)	−0.046 (4)	0.050 (2)	0.053 (6)*	0.33
O2W3	0.4347 (9)	0.027 (5)	−0.012 (3)	0.095 (10)*	0.33

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.031 (5)	0.046 (7)	0.053 (6)	−0.018 (6)	0.003 (4)	−0.013 (6)
C2	0.031 (5)	0.075 (10)	0.036 (6)	−0.013 (6)	0.004 (4)	−0.006 (6)
C3	0.027 (5)	0.066 (9)	0.048 (6)	−0.001 (6)	0.007 (5)	0.003 (7)
C4	0.026 (4)	0.035 (6)	0.044 (5)	0.001 (5)	0.013 (4)	0.008 (6)
C5	0.030 (5)	0.034 (6)	0.031 (5)	0.003 (5)	0.003 (4)	0.019 (5)
C6	0.044 (6)	0.030 (7)	0.043 (6)	−0.004 (5)	0.015 (5)	−0.009 (5)
C7	0.031 (5)	0.026 (6)	0.051 (6)	−0.009 (5)	0.008 (4)	−0.002 (6)
C8	0.029 (5)	0.021 (6)	0.041 (5)	0.004 (5)	0.010 (4)	0.004 (5)
C9	0.021 (4)	0.038 (6)	0.030 (5)	−0.001 (5)	0.002 (4)	−0.001 (5)
C10	0.029 (5)	0.024 (6)	0.046 (6)	0.001 (5)	0.006 (4)	0.011 (5)
C11	0.027 (5)	0.041 (8)	0.053 (6)	−0.005 (5)	0.006 (4)	−0.008 (6)
C12	0.028 (5)	0.020 (6)	0.051 (6)	0.002 (5)	0.009 (4)	0.001 (5)
C13	0.032 (5)	0.039 (7)	0.031 (5)	−0.005 (5)	0.002 (4)	0.010 (5)
C14	0.039 (5)	0.020 (6)	0.035 (5)	−0.012 (5)	0.002 (4)	0.004 (5)
C15	0.035 (5)	0.025 (6)	0.042 (5)	−0.008 (5)	0.005 (4)	0.006 (5)
C16	0.032 (5)	0.029 (6)	0.030 (5)	−0.001 (5)	0.007 (4)	−0.004 (5)
C17	0.032 (5)	0.044 (7)	0.035 (5)	0.004 (5)	0.014 (4)	0.009 (5)
C18	0.051 (6)	0.050 (8)	0.078 (8)	0.013 (7)	0.022 (6)	0.024 (7)
C19	0.044 (6)	0.036 (7)	0.034 (5)	0.006 (6)	0.007 (5)	−0.004 (6)
C20	0.030 (5)	0.057 (8)	0.029 (5)	−0.009 (6)	0.009 (4)	0.001 (5)
C1'	0.021 (5)	0.034 (6)	0.030 (5)	−0.008 (5)	0.001 (4)	0.000 (5)
C2'	0.024 (4)	0.044 (8)	0.046 (6)	0.006 (5)	0.007 (4)	0.002 (6)
C3'	0.047 (6)	0.032 (7)	0.046 (6)	−0.010 (6)	0.009 (5)	−0.001 (6)
C4'	0.023 (5)	0.047 (7)	0.036 (5)	0.002 (5)	0.011 (4)	0.004 (6)
C5'	0.038 (5)	0.057 (9)	0.035 (6)	0.001 (6)	0.013 (5)	0.003 (6)
C6'	0.040 (6)	0.067 (9)	0.079 (8)	−0.010 (7)	0.022 (6)	−0.024 (8)
O16	0.029 (3)	0.040 (5)	0.035 (3)	−0.009 (4)	0.006 (3)	−0.004 (4)
O17	0.033 (4)	0.039 (5)	0.079 (5)	0.003 (4)	0.019 (4)	0.010 (5)
O19	0.046 (4)	0.074 (6)	0.040 (4)	−0.022 (5)	0.016 (3)	−0.013 (5)
O20	0.048 (4)	0.088 (7)	0.046 (4)	−0.042 (5)	0.012 (3)	−0.003 (5)
O21	0.027 (3)	0.051 (5)	0.054 (4)	−0.001 (4)	0.020 (3)	0.001 (4)
O22	0.039 (4)	0.041 (5)	0.045 (4)	0.008 (4)	0.012 (3)	0.011 (4)
O23	0.064 (5)	0.072 (7)	0.134 (7)	−0.038 (5)	0.038 (5)	−0.065 (7)

O24	0.031 (4)	0.070 (6)	0.055 (4)	−0.014 (4)	0.009 (3)	0.006 (5)
O26	0.086 (7)	0.112 (10)	0.134 (8)	−0.010 (7)	0.030 (6)	−0.053 (8)
O1W	0.071 (7)	0.056 (8)	0.066 (7)	0.000	0.025 (7)	0.000

Geometric parameters (Å, °)

C1—C2	1.512 (12)	C16—O16	1.485 (10)
C1—C10	1.534 (14)	C16—C17	1.513 (12)
C1—H1A	0.9700	C17—O17	1.442 (11)
C1—H1B	0.9700	C17—H17A	0.9700
C2—C3	1.505 (17)	C17—H17B	0.9700
C2—H2A	0.9700	C18—H18A	0.9600
C2—H2B	0.9700	C18—H18B	0.9600
C3—C4	1.540 (14)	C18—H18C	0.9600
C3—H3A	0.9700	C19—O20	1.209 (12)
C3—H3B	0.9700	C19—O19	1.321 (11)
C4—C19	1.532 (13)	C20—H20A	0.9600
C4—C18	1.558 (16)	C20—H20B	0.9600
C4—C5	1.561 (11)	C20—H20C	0.9600
C5—C6	1.525 (13)	C1'—O16	1.393 (10)
C5—C10	1.555 (14)	C1'—O21	1.418 (11)
C5—H5	0.9800	C1'—C2'	1.503 (13)
C6—C7	1.537 (12)	C1'—H1'	0.9800
C6—H6A	0.9700	C2'—O22	1.420 (11)
C6—H6B	0.9700	C2'—C3'	1.551 (13)
C7—C8	1.523 (13)	C2'—H2'	0.9800
C7—H7A	0.9700	C3'—O23	1.447 (12)
C7—H7B	0.9700	C3'—C4'	1.513 (14)
C8—C14	1.549 (12)	C3'—H3'	0.9800
C8—C9	1.559 (13)	C4'—O24	1.404 (11)
C8—C15	1.560 (12)	C4'—C5'	1.535 (14)
C9—C11	1.520 (14)	C4'—H4'	0.9800
C9—C10	1.588 (11)	C5'—O21	1.430 (11)
C9—H9	0.9800	C5'—C6'	1.477 (16)
C10—C20	1.529 (13)	C5'—H5'	0.9800
C11—C12	1.529 (12)	C6'—O26	1.485 (14)
C11—H11A	0.9700	C6'—H6'1	0.9700
C11—H11B	0.9700	C6'—H6'2	0.9700
C12—C13	1.547 (13)	O17—H17	0.82 (2)
C12—H12A	0.9700	O19—H19	0.84 (2)
C12—H12B	0.9700	O22—H22	0.81 (2)
C13—C14	1.537 (13)	O23—H23	0.8200
C13—C16	1.538 (12)	O24—H24	0.82 (2)
C13—H13	0.9800	O26—H26	0.8200
C14—H14A	0.9700	O1W—H1W	0.85 (2)
C14—H14B	0.9700	O2W1—O2W3	1.41 (4)
C15—C16	1.561 (12)	O2W1—O2W2	1.41 (4)
C15—H15A	0.9700	O2W2—O2W3	0.87 (3)
C15—H15B	0.9700		

C2—C1—C10	113.9 (9)	C8—C14—H14B	111.4
C2—C1—H1A	108.8	H14A—C14—H14B	109.3
C10—C1—H1A	108.8	C8—C15—C16	107.0 (7)
C2—C1—H1B	108.8	C8—C15—H15A	110.3
C10—C1—H1B	108.8	C16—C15—H15A	110.3
H1A—C1—H1B	107.7	C8—C15—H15B	110.3
C3—C2—C1	110.6 (10)	C16—C15—H15B	110.3
C3—C2—H2A	109.5	H15A—C15—H15B	108.6
C1—C2—H2A	109.5	O16—C16—C17	111.1 (7)
C3—C2—H2B	109.5	O16—C16—C13	102.8 (6)
C1—C2—H2B	109.5	C17—C16—C13	116.8 (8)
H2A—C2—H2B	108.1	O16—C16—C15	108.6 (8)
C2—C3—C4	111.9 (8)	C17—C16—C15	112.6 (7)
C2—C3—H3A	109.2	C13—C16—C15	104.1 (7)
C4—C3—H3A	109.2	O17—C17—C16	113.7 (7)
C2—C3—H3B	109.2	O17—C17—H17A	108.8
C4—C3—H3B	109.2	C16—C17—H17A	108.8
H3A—C3—H3B	107.9	O17—C17—H17B	108.8
C19—C4—C3	109.3 (8)	C16—C17—H17B	108.8
C19—C4—C18	104.5 (8)	H17A—C17—H17B	107.7
C3—C4—C18	108.2 (8)	C4—C18—H18A	109.5
C19—C4—C5	117.2 (7)	C4—C18—H18B	109.5
C3—C4—C5	107.5 (8)	H18A—C18—H18B	109.5
C18—C4—C5	109.9 (9)	C4—C18—H18C	109.5
C6—C5—C10	111.5 (7)	H18A—C18—H18C	109.5
C6—C5—C4	117.5 (8)	H18B—C18—H18C	109.5
C10—C5—C4	114.6 (8)	O20—C19—O19	121.0 (9)
C6—C5—H5	103.8	O20—C19—C4	125.4 (9)
C10—C5—H5	103.8	O19—C19—C4	113.4 (9)
C4—C5—H5	103.8	C10—C20—H20A	109.5
C5—C6—C7	109.9 (7)	C10—C20—H20B	109.5
C5—C6—H6A	109.7	H20A—C20—H20B	109.5
C7—C6—H6A	109.7	C10—C20—H20C	109.5
C5—C6—H6B	109.7	H20A—C20—H20C	109.5
C7—C6—H6B	109.7	H20B—C20—H20C	109.5
H6A—C6—H6B	108.2	O16—C1'—O21	107.3 (8)
C8—C7—C6	113.1 (8)	O16—C1'—C2'	109.7 (7)
C8—C7—H7A	109.0	O21—C1'—C2'	109.9 (7)
C6—C7—H7A	109.0	O16—C1'—H1'	110.0
C8—C7—H7B	109.0	O21—C1'—H1'	110.0
C6—C7—H7B	109.0	C2'—C1'—H1'	110.0
H7A—C7—H7B	107.8	O22—C2'—C1'	111.8 (7)
C7—C8—C14	113.3 (8)	O22—C2'—C3'	111.9 (9)
C7—C8—C9	110.7 (7)	C1'—C2'—C3'	110.4 (8)
C14—C8—C9	113.0 (8)	O22—C2'—H2'	107.5
C7—C8—C15	109.8 (8)	C1'—C2'—H2'	107.5
C14—C8—C15	100.0 (7)	C3'—C2'—H2'	107.5
C9—C8—C15	109.4 (7)	O23—C3'—C4'	111.0 (8)
C11—C9—C8	110.2 (7)	O23—C3'—C2'	108.0 (8)

C11—C9—C10	114.7 (8)	C4'—C3'—C2'	113.0 (9)
C8—C9—C10	116.7 (7)	O23—C3'—H3'	108.3
C11—C9—H9	104.6	C4'—C3'—H3'	108.3
C8—C9—H9	104.6	C2'—C3'—H3'	108.3
C10—C9—H9	104.6	O24—C4'—C3'	109.9 (9)
C20—C10—C1	107.9 (9)	O24—C4'—C5'	108.7 (7)
C20—C10—C5	113.9 (8)	C3'—C4'—C5'	108.2 (7)
C1—C10—C5	109.1 (8)	O24—C4'—H4'	110.0
C20—C10—C9	112.7 (7)	C3'—C4'—H4'	110.0
C1—C10—C9	106.6 (7)	C5'—C4'—H4'	110.0
C5—C10—C9	106.4 (8)	O21—C5'—C6'	107.4 (9)
C9—C11—C12	117.2 (8)	O21—C5'—C4'	108.3 (7)
C9—C11—H11A	108.0	C6'—C5'—C4'	115.2 (8)
C12—C11—H11A	108.0	O21—C5'—H5'	108.6
C9—C11—H11B	108.0	C6'—C5'—H5'	108.6
C12—C11—H11B	108.0	C4'—C5'—H5'	108.6
H11A—C11—H11B	107.3	C5'—C6'—O26	103.6 (8)
C11—C12—C13	113.8 (8)	C5'—C6'—H6'1	111.0
C11—C12—H12A	108.8	O26—C6'—H6'1	111.0
C13—C12—H12A	108.8	C5'—C6'—H6'2	111.0
C11—C12—H12B	108.8	O26—C6'—H6'2	111.0
C13—C12—H12B	108.8	H6'1—C6'—H6'2	109.0
H12A—C12—H12B	107.7	C1'—O16—C16	119.7 (6)
C14—C13—C16	101.8 (8)	C17—O17—H17	104 (4)
C14—C13—C12	107.5 (7)	C19—O19—H19	111 (4)
C16—C13—C12	113.4 (7)	C1'—O21—C5'	116.1 (8)
C14—C13—H13	111.2	C2'—O22—H22	105 (4)
C16—C13—H13	111.2	C3'—O23—H23	109.5
C12—C13—H13	111.2	C4'—O24—H24	105 (4)
C13—C14—C8	101.7 (7)	C6'—O26—H26	109.5
C13—C14—H14A	111.4	O2W3—O2W1—O2W2	35.8 (15)
C8—C14—H14A	111.4	O2W3—O2W2—O2W1	72 (3)
C13—C14—H14B	111.4	O2W2—O2W3—O2W1	73 (3)
C10—C1—C2—C3	−56.6 (12)	C7—C8—C15—C16	−145.7 (8)
C1—C2—C3—C4	60.1 (11)	C14—C8—C15—C16	−26.3 (9)
C2—C3—C4—C19	70.2 (10)	C9—C8—C15—C16	92.7 (8)
C2—C3—C4—C18	−176.5 (8)	C14—C13—C16—O16	−81.5 (8)
C2—C3—C4—C5	−57.9 (11)	C12—C13—C16—O16	163.4 (7)
C19—C4—C5—C6	64.8 (13)	C14—C13—C16—C17	156.6 (7)
C3—C4—C5—C6	−171.7 (8)	C12—C13—C16—C17	41.5 (11)
C18—C4—C5—C6	−54.2 (11)	C14—C13—C16—C15	31.8 (8)
C19—C4—C5—C10	−68.9 (12)	C12—C13—C16—C15	−83.3 (9)
C3—C4—C5—C10	54.5 (11)	C8—C15—C16—O16	105.9 (8)
C18—C4—C5—C10	172.0 (9)	C8—C15—C16—C17	−130.6 (8)
C10—C5—C6—C7	−63.8 (11)	C8—C15—C16—C13	−3.2 (9)
C4—C5—C6—C7	161.1 (8)	O16—C16—C17—O17	−63.4 (10)
C5—C6—C7—C8	58.6 (11)	C13—C16—C17—O17	54.0 (10)
C6—C7—C8—C14	78.9 (10)	C15—C16—C17—O17	174.5 (8)
C6—C7—C8—C9	−49.2 (11)	C3—C4—C19—O20	20.2 (14)

C6—C7—C8—C15	−170.2 (7)	C18—C4—C19—O20	−95.4 (13)
C7—C8—C9—C11	−179.8 (8)	C5—C4—C19—O20	142.7 (11)
C14—C8—C9—C11	51.9 (10)	C3—C4—C19—O19	−163.8 (9)
C15—C8—C9—C11	−58.6 (10)	C18—C4—C19—O19	80.6 (10)
C7—C8—C9—C10	47.1 (11)	C5—C4—C19—O19	−41.3 (13)
C14—C8—C9—C10	−81.3 (10)	O16—C1'—C2'—O22	66.5 (9)
C15—C8—C9—C10	168.2 (7)	O21—C1'—C2'—O22	−175.8 (7)
C2—C1—C10—C20	−73.1 (11)	O16—C1'—C2'—C3'	−168.2 (8)
C2—C1—C10—C5	51.1 (12)	O21—C1'—C2'—C3'	−50.5 (10)
C2—C1—C10—C9	165.7 (9)	O22—C2'—C3'—O23	−61.1 (11)
C6—C5—C10—C20	−67.1 (9)	C1'—C2'—C3'—O23	173.8 (8)
C4—C5—C10—C20	69.4 (10)	O22—C2'—C3'—C4'	175.8 (8)
C6—C5—C10—C1	172.4 (7)	C1'—C2'—C3'—C4'	50.6 (11)
C4—C5—C10—C1	−51.2 (10)	O23—C3'—C4'—O24	66.8 (10)
C6—C5—C10—C9	57.7 (10)	C2'—C3'—C4'—O24	−171.8 (7)
C4—C5—C10—C9	−165.8 (7)	O23—C3'—C4'—C5'	−174.7 (8)
C11—C9—C10—C20	−56.1 (11)	C2'—C3'—C4'—C5'	−53.2 (11)
C8—C9—C10—C20	75.0 (11)	O24—C4'—C5'—O21	176.2 (8)
C11—C9—C10—C1	62.1 (10)	C3'—C4'—C5'—O21	56.9 (10)
C8—C9—C10—C1	−166.8 (9)	O24—C4'—C5'—C6'	−63.6 (12)
C11—C9—C10—C5	178.4 (8)	C3'—C4'—C5'—C6'	177.1 (9)
C8—C9—C10—C5	−50.5 (10)	O21—C5'—C6'—O26	−60.9 (11)
C8—C9—C11—C12	−34.5 (11)	C4'—C5'—C6'—O26	178.4 (8)
C10—C9—C11—C12	99.7 (9)	O21—C1'—O16—C16	83.3 (9)
C9—C11—C12—C13	39.1 (11)	C2'—C1'—O16—C16	−157.4 (7)
C11—C12—C13—C14	−57.6 (10)	C17—C16—O16—C1'	−41.3 (11)
C11—C12—C13—C16	54.1 (11)	C13—C16—O16—C1'	−166.9 (8)
C16—C13—C14—C8	−49.4 (8)	C15—C16—O16—C1'	83.2 (9)
C12—C13—C14—C8	70.0 (8)	O16—C1'—O21—C5'	179.9 (7)
C7—C8—C14—C13	163.0 (7)	C2'—C1'—O21—C5'	60.7 (10)
C9—C8—C14—C13	−70.1 (8)	C6'—C5'—O21—C1'	171.2 (8)
C15—C8—C14—C13	46.2 (8)	C4'—C5'—O21—C1'	−63.8 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O17—H17···O21	0.83 (2)	2.14 (7)	2.753 (9)	131 (7)
O17—H17···O26	0.83 (2)	2.29 (6)	3.033 (11)	149 (9)
O19—H19···O22^{i}	0.84 (6)	1.82 (5)	2.641 (13)	171.9 (3)
O22—H22···O17^{ii}	0.81 (2)	2.17 (4)	2.715 (6)	125.1 (3)
O24—H24···O20^{iii}	0.82 (5)	1.94 (5)	2.747 (6)	164.9 (3)
O26—H26···O1W	0.82	2.27	2.757 (15)	118
O1W—H1W···O24^{iv}	0.86 (5)	1.96 (4)	2.804 (6)	169.4 (3)

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

Fig. 1

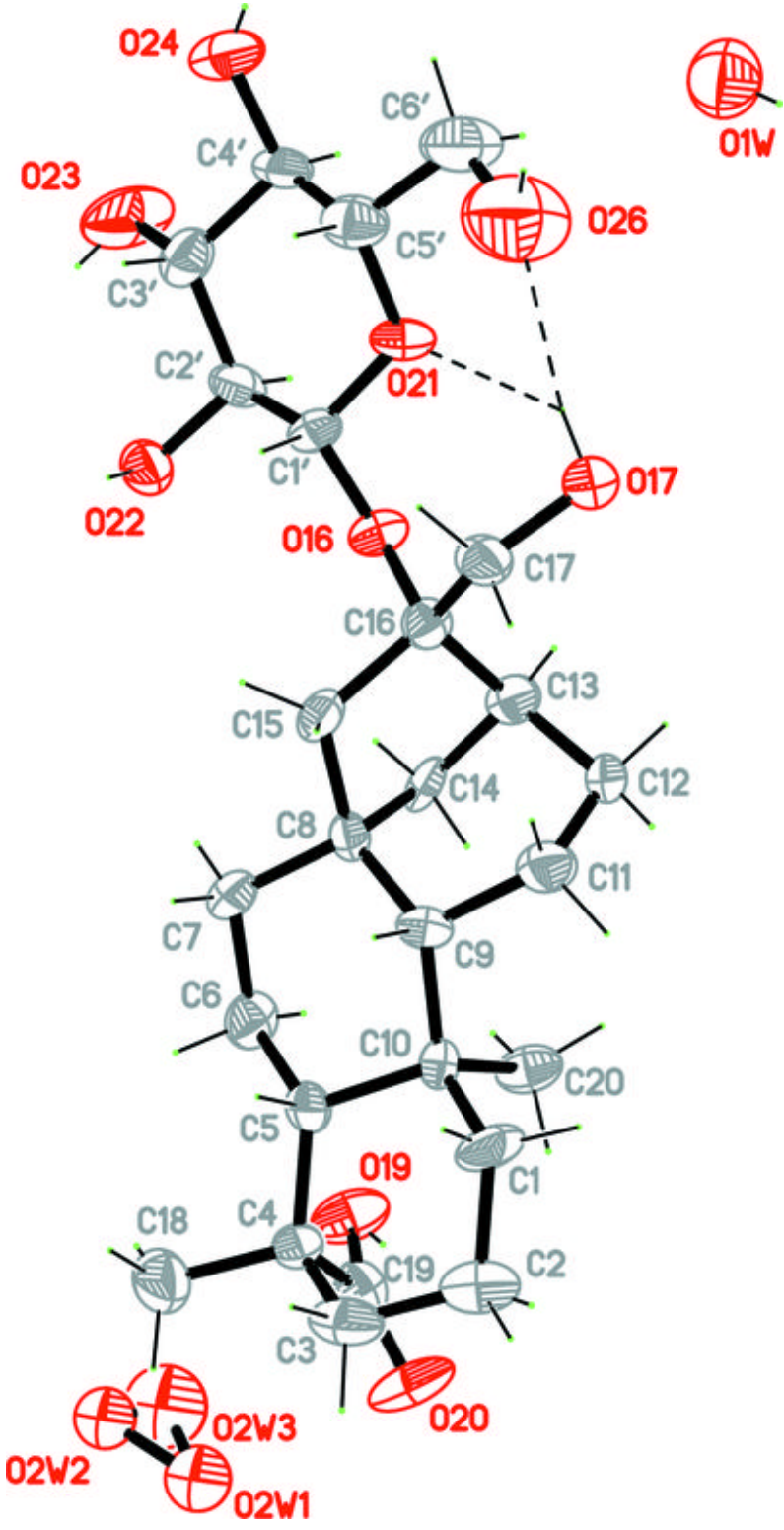
O24
C6′
O1W
C4′
O23
O26
C5′
C3′
O21
C2′
C1′
O17
O22
O16
C17
C16
C13
C15
C14
C12
C8
C11
C7
C9
C6
C10
C20
C5
C1
O19
C18
C4
C19
C2
C3
O20
O2W3
O2W2
O2W1

Fig. 2