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Alisol F 24-acetate: (24*R*)-24-acetoxy-11β,25-dihydroxy-16β,23β-epoxy-protost-13(17)-en-3-one

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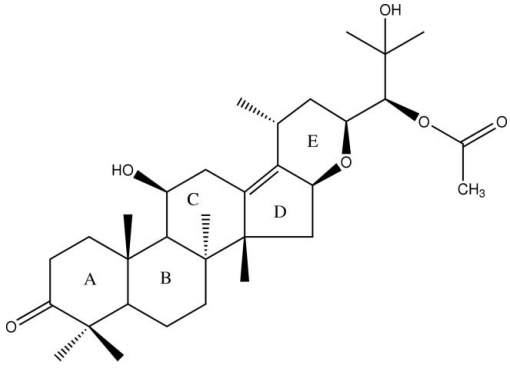
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.011 Å; R factor = 0.089; wR factor = 0.236; data-to-parameter ratio = 7.9.

The title compound, $C_{32}H_{50}O_6$, a known protostane-type triterpene, was isolated from the Chinese herbal medicine *Lobelia chinensis* Lour. The molecule contains five fused rings: four six-membered rings (*A*, *B*, *C* and *E*) and a five-membered ring (*D*). Rings *A* and *B* have slightly distorted boat conformations, while rings *C* and *E* adopt chair conformations. Ring *D* is almost planar. Rings *A* and *B* and rings *B* and *C* are both *trans*-fused. A chain running along the *b* axis is formed *via* classical intermolecular O—H···O hydrogen bonds.

Related literature

For related literature, see: Peng & Lou (2001); Zhou *et al.* (2005).



Experimental

Crystal data

$C_{32}H_{50}O_6$
M_r = 530.72
Monoclinic, $P2_1$
a = 6.525 (4) Å
b = 15.885 (10) Å
c = 14.401 (9) Å
β = 100.475 (10)°
V = 1467.8 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.50 × 0.47 × 0.40 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.959, T_{max} = 0.968
7306 measured reflections
2697 independent reflections
1811 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.089
$wR(F^2)$ = 0.236
S = 1.00
2697 reflections
343 parameters
37 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O6^{i}	0.82	2.09	2.904 (7)	172
O6—H6···O1^{ii}	0.82	2.07	2.867 (8)	165

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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, 2001); software used to prepare material for publication: *SHELXTL*.

The use of the Bruker X-ray Crystallographic Services at Liaocheng University and valuable assistance from Professor Daqi Wang are gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2209).

References

Bruker (2001). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Peng, G. P. & Lou, F. C. (2001). *Nat. Prod. Res. Dev.* **13**, 1–4.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Zhou, X. F., Yin, R. J. & Ruan, H. L. (2005). *Chin. J. Magn. Reson.* **22**, 195–200.

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supplementary materials

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Alisol F 24-acetate: (24*R*)-24-acetoxy-11β,25-dihydroxy-16β,23β-epoxyprotost-13(17)-en-3-one

X.-B. Wang and L.-Y. Kong

Comment

The title compound, $C_{32}H_{50}O_6$, a known protostane-type triterpene, named alisol F 24-acetate [systematic name: 16β, 23β-epoxy-11β, 25-dihydroxy-24(*R*)-acetoxy-protost-13 (17)-en-3-one], was isolated from the Chinese herbal medicine *Lobelia chinensis* Lour firstly obtained by Peng & Lou (2001) and found to possess activity for inhibiting formation of urinary oxalate calcium calculi. Its structure was elucidated on the basis of spectroscopic methods such as ^{1}H NMR, ^{13}C NMR, two-dimensional NMR and chemical correlation (Peng & Lou, 2001; Zhou *et al.*, 2005).

The molecule contains a five-fused ring system including four six-membered rings, *A*(C1—C5/C10), *B*(C5—C10), *C*(C8—C14) and *E*(O3/C16—C20) and a five-membered ring *D*(C13—C17) (Fig. 1). The junctions of the three fused six-membered rings *A*, *B* and *C*, two of which, *A* and *B*, adopt slightly distorted boat conformations and the *C* exhibits a perfect chair conformation, are all *trans*. The five-membered ring *D* is almost planar. Ring *E* adopts a chair conformation. A chain running along the *b* axis was formed *via* the classic intermolecular O—H···O hydrogen bonds as shown in Fig. 2.

Experimental

The dried herb of *Lobelia chinensis* (18 kg) was extracted with refluxing 95% ethanol (3 × 20 *L*), after removal of the solvent under reduced pressure, the extract was suspended in water and then partitioned with light petroleum, EtOAc and n-BuOH successively. The EtOAc-soluble part (150 g) was chromatographed on a silica gel column, using a gradient mixture of light petroleum-EtOAc as eluent. The fraction eluted with petrol-EtOAc (9: 2, *v/v*) was further purified by sephadex LH-20 column chromatography using $CHCl_3$—CH_3OH (1:1 *v/v*) as eluent, to yield the title compound (10 mg). IR (KBr) cm^{-1}: 3450, 3310, 1740, 1698, 1459, 1375, 1248, 1100, 1053. ^{1}H NMR (500 MHz, DMSO-d$_6$) δ: 2.47 (1*H*, m, H-1a), 2.32 (1*H*, m, H-1 b), 2.87 (1*H*, m, H-2a), 2.44 (1*H*, m, H-2 b), 2.25 (1*H*, dd, *J* = 11.0, 5.1 Hz, H-5),1.41 (1*H*, m, H-6a), 1.98 (1*H*, m, H-7a), 1.26 (2*H*, m, H-6 b, 7 b), 1.95 (1*H*, d, *J* = 11.0 Hz, H-9), 4.0 4 (1*H*, m, H-11), 2.91 (1*H*, dd, *J* = 13.0, 5.8 Hz, H-12*a*), 2.39 (1*H*, dd, *J* = 13.0, 12.8 Hz, H-12*b*), 2.28 (1*H*, dd, *J* = 13.8, 6.5 Hz, H-15*a*), 1.40 (1*H*, dd, *J* = 13.8, 5.5 Hz, H-15*b*), 4.58 (1*H*, dd, *J*=9.1, 7.1 Hz, H-16), 1.01 (3*H*, s, H-18), 1.38 (3*H*, s, H-19), 2.84 (1*H*, m, H-20), 1.14 (3*H*, d, *J* = 7.1 Hz, H-21), 1.88 (1*H*, m, H-22*a*), 1.51 (1*H*, m, H-22*b*), 4.48 (1*H*, m, H-23), 5.21 (1*H*, d, *J* = 2.6 Hz, H-24), 1.56 (3*H*, s, H-26), 1.60 (3*H*, s, H-27), 1.11 (3*H*, s, H-28), 1.18 (3*H*, s, H-29), 1.19 (3*H*, s, H-30), 2.10 (3*H*, s, 24-OAc); ^{13}C NMR (125 MHz, in DMSO-d$_6$) δ: 30.9 (C-1), 34.4 (C-2), 219.1 (C-3), 47.1 (C-4), 48.4 (C-5), 20.2 (C-6), 34.0 (C-7), 40.9 (C-8), 49.8 (C-9), 37.5 (C-10), 69.8 (C-11), 34.2 (C-12), 137.6 (C-13), 55.5 (C-14), 39.8 (C-15), 80.9 (C-16), 133.1 (C-17), 23.8 (C-18), 25.6 (C-19), 26.9 (C-20), 18.4 (C-21), 35.6 (C-22), 72.0 (C-23), 79.9 (C-24), 72.3 (C-25), 28.4 (C-26), 27.3 (C-27), 29.4 (C-28), 20.9 (C-29), 24.4 (C-30), 171.0 (24-OAc). Crystals suitable for X-ray structure analysis were obtained by slow evaporation of a methanol solution at room temperature.

Refinement

H atoms were positioned geometrically, with O—H = 0.82 and C—H = 0.98 (methine H), 0.97 (methylene H) or 0.96 Å (methyl H), and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*O*), where x = 1.2 for methine and methylene H and x = 1.5 for all other H. The acetyl group was restrained to keep their atomic displacement parameters close to those of the spatially adjacent atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged, and the absolute configuration is assigned arbitrarily.

Figures

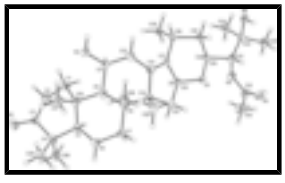

Fig. 1. A drawing of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

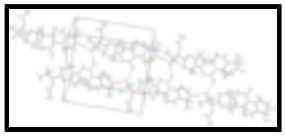

Fig. 2. A packing diagram of (I) showing the formation of the chain running along the *b* axis *via* the classic intermolecular O—H···O hydrogen bonds.

(24*R*)-24-acetoxy-11*β*, 25-dihydroxy-16*β*,23*β*-epoxyprotost-13 (17)-en-3-one

Crystal data

$C_{32}H_{50}O_6$	F_{000} = 580
M_r = 530.72	D_x = 1.201 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1829 reflections
a = 6.525 (4) Å	θ = 2.5–20.9º
b = 15.885 (10) Å	μ = 0.08 mm^{-1}
c = 14.401 (9) Å	T = 298 (2) K
β = 100.475 (10)º	Prism, colourless
V = 1467.8 (16) Å^3	0.50 × 0.47 × 0.40 mm
Z = 2	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2697 independent reflections
Radiation source: fine-focus sealed tube	1811 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.072
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	h = −7→7

T_{min} = 0.959, T_{max} = 0.968	k = −16→18
7306 measured reflections	l = −17→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.089	H-atom parameters constrained
$wR(F^2)$ = 0.236	$w = 1/[\sigma^2(F_o^2) + (0.1619P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
2697 reflections	$\Delta\rho_{max}$ = 0.48 e Å^{-3}
343 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
37 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.5063 (9)	1.1516 (4)	0.2720 (4)	0.0555 (15)
O2	0.2109 (9)	0.8135 (3)	0.4045 (3)	0.0460 (14)
H2	0.2120	0.7918	0.4562	0.069*
O3	0.7078 (9)	0.4694 (3)	0.2211 (3)	0.0371 (13)
O4	1.0287 (8)	0.3572 (3)	0.2271 (4)	0.0428 (13)
O5	0.8746 (14)	0.2837 (6)	0.1019 (4)	0.085 (2)
O6	0.7817 (9)	0.2528 (3)	0.4048 (4)	0.0444 (13)
H6	0.6889	0.2314	0.3659	0.067*
C1	0.5384 (12)	0.9359 (5)	0.3534 (5)	0.0383 (18)
H1A	0.4649	0.9368	0.4062	0.046*
H1B	0.6501	0.8949	0.3675	0.046*
C2	0.6326 (15)	1.0232 (5)	0.3431 (6)	0.052 (2)
H2A	0.6314	1.0541	0.4010	0.062*
H2B	0.7773	1.0155	0.3374	0.062*
C3	0.5303 (11)	1.0768 (5)	0.2622 (5)	0.0349 (17)

C4	0.4809 (12)	1.0350 (5)	0.1647 (5)	0.0383 (18)
C5	0.4810 (11)	0.9379 (5)	0.1782 (5)	0.0331 (16)
H5	0.6285	0.9222	0.1935	0.040*
C6	0.3929 (13)	0.8873 (5)	0.0909 (5)	0.0389 (18)
H6A	0.4321	0.9132	0.0357	0.047*
H6B	0.2420	0.8864	0.0822	0.047*
C7	0.4770 (14)	0.7981 (5)	0.1020 (5)	0.0410 (19)
H7A	0.6161	0.7979	0.0871	0.049*
H7B	0.3903	0.7627	0.0559	0.049*
C8	0.4879 (10)	0.7578 (5)	0.2008 (5)	0.0314 (15)
C9	0.3477 (11)	0.8093 (5)	0.2591 (4)	0.0301 (16)
H9	0.2051	0.8026	0.2242	0.036*
C10	0.3867 (11)	0.9081 (5)	0.2639 (5)	0.0317 (16)
C11	0.3472 (12)	0.7679 (5)	0.3552 (5)	0.0331 (16)
H11	0.4887	0.7704	0.3923	0.040*
C12	0.2799 (12)	0.6763 (5)	0.3456 (5)	0.0385 (18)
H12A	0.2947	0.6509	0.4077	0.046*
H12B	0.1343	0.6730	0.3158	0.046*
C13	0.4099 (12)	0.6295 (4)	0.2876 (5)	0.0335 (17)
C14	0.4086 (11)	0.6647 (5)	0.1904 (5)	0.0327 (16)
C15	0.5620 (13)	0.6021 (5)	0.1531 (5)	0.0394 (18)
H15A	0.4849	0.5622	0.1090	0.047*
H15B	0.6580	0.6326	0.1214	0.047*
C16	0.6790 (12)	0.5568 (5)	0.2389 (5)	0.0351 (17)
H16	0.8152	0.5836	0.2591	0.042*
C17	0.5477 (12)	0.5702 (4)	0.3132 (5)	0.0345 (17)
C18	0.5973 (14)	0.5198 (5)	0.4025 (5)	0.0396 (19)
H18	0.4731	0.5186	0.4319	0.047*
C19	0.6453 (12)	0.4289 (5)	0.3749 (5)	0.0361 (17)
H19A	0.7017	0.3974	0.4315	0.043*
H19B	0.5168	0.4018	0.3451	0.043*
C20	0.7985 (12)	0.4269 (4)	0.3083 (5)	0.0333 (16)
H20	0.9246	0.4572	0.3375	0.040*
C21	0.8607 (11)	0.3405 (5)	0.2789 (5)	0.0356 (17)
H21	0.7430	0.3145	0.2366	0.043*
C22	0.9502 (11)	0.2777 (5)	0.3590 (5)	0.0343 (17)
C23	1.0319 (14)	0.2005 (5)	0.3158 (6)	0.050 (2)
H23A	0.9253	0.1784	0.2671	0.075*
H23B	1.0709	0.1585	0.3637	0.075*
H23C	1.1514	0.2157	0.2891	0.075*
C24	1.1135 (13)	0.3150 (6)	0.4342 (6)	0.052 (2)
H24A	1.0593	0.3647	0.4591	0.079*
H24B	1.2336	0.3296	0.4078	0.079*
H24C	1.1524	0.2748	0.4841	0.079*
C25	0.2861 (14)	1.0721 (5)	0.1050 (6)	0.053 (2)
H25A	0.2588	1.0445	0.0447	0.080*
H25B	0.3064	1.1312	0.0962	0.080*
H25C	0.1698	1.0640	0.1365	0.080*
C26	0.6698 (13)	1.0584 (6)	0.1182 (6)	0.050 (2)

H26A	0.6526	1.0337	0.0565	0.075*
H26B	0.7955	1.0375	0.1564	0.075*
H26C	0.6782	1.1185	0.1131	0.075*
C27	0.1778 (13)	0.9489 (6)	0.2643 (5)	0.045 (2)
H27A	0.1942	1.0090	0.2670	0.068*
H27B	0.1237	0.9300	0.3183	0.068*
H27C	0.0828	0.9338	0.2077	0.068*
C28	0.7171 (11)	0.7592 (5)	0.2526 (6)	0.0403 (18)
H28A	0.7263	0.7351	0.3143	0.060*
H28B	0.7663	0.8162	0.2582	0.060*
H28C	0.8012	0.7270	0.2173	0.060*
C29	0.1928 (12)	0.6566 (6)	0.1275 (5)	0.0452 (19)
H29A	0.1967	0.6800	0.0664	0.068*
H29B	0.0921	0.6865	0.1559	0.068*
H29C	0.1545	0.5983	0.1210	0.068*
C30	0.7728 (18)	0.5592 (6)	0.4713 (5)	0.067 (3)
H30A	0.7997	0.5264	0.5281	0.100*
H30B	0.7346	0.6154	0.4859	0.100*
H30C	0.8960	0.5611	0.4436	0.100*
C31	1.0144 (18)	0.3278 (7)	0.1390 (6)	0.060 (2)
C32	1.193 (2)	0.3555 (10)	0.0980 (8)	0.102 (4)
H32A	1.2844	0.3889	0.1434	0.153*
H32B	1.1447	0.3885	0.0425	0.153*
H32C	1.2671	0.3071	0.0814	0.153*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.056 (4)	0.039 (4)	0.072 (4)	0.002 (3)	0.015 (3)	0.002 (3)
O2	0.064 (4)	0.040 (3)	0.040 (3)	0.006 (3)	0.025 (3)	−0.004 (2)
O3	0.059 (4)	0.028 (3)	0.027 (2)	0.008 (2)	0.015 (2)	−0.001 (2)
O4	0.046 (3)	0.043 (3)	0.045 (3)	0.007 (2)	0.023 (2)	−0.005 (2)
O5	0.121 (7)	0.095 (6)	0.037 (3)	0.004 (5)	0.007 (4)	−0.010 (4)
O6	0.053 (3)	0.039 (3)	0.047 (3)	−0.006 (3)	0.023 (3)	−0.012 (2)
C1	0.042 (5)	0.040 (4)	0.031 (4)	0.000 (4)	0.003 (3)	0.001 (3)
C2	0.068 (6)	0.044 (5)	0.037 (4)	−0.016 (4)	−0.004 (4)	−0.008 (4)
C3	0.033 (4)	0.035 (5)	0.039 (4)	−0.004 (3)	0.012 (3)	0.003 (3)
C4	0.028 (4)	0.039 (4)	0.049 (4)	0.005 (3)	0.010 (3)	0.002 (3)
C5	0.033 (4)	0.034 (4)	0.032 (4)	0.007 (3)	0.007 (3)	0.001 (3)
C6	0.049 (5)	0.034 (4)	0.033 (4)	−0.005 (4)	0.007 (3)	−0.004 (3)
C7	0.050 (5)	0.045 (5)	0.030 (4)	0.004 (4)	0.012 (3)	−0.004 (3)
C8	0.021 (4)	0.029 (4)	0.044 (4)	0.004 (3)	0.004 (3)	0.004 (3)
C9	0.023 (4)	0.038 (4)	0.029 (3)	0.000 (3)	0.006 (3)	0.001 (3)
C10	0.025 (4)	0.038 (4)	0.033 (3)	0.002 (3)	0.008 (3)	−0.004 (3)
C11	0.039 (4)	0.029 (4)	0.033 (4)	0.001 (3)	0.010 (3)	−0.004 (3)
C12	0.041 (5)	0.037 (4)	0.043 (4)	−0.002 (3)	0.021 (3)	−0.001 (3)
C13	0.039 (4)	0.030 (4)	0.036 (4)	−0.001 (3)	0.018 (3)	0.002 (3)
C14	0.035 (4)	0.037 (4)	0.026 (3)	0.004 (3)	0.005 (3)	0.004 (3)

C15	0.050 (5)	0.037 (4)	0.035 (4)	0.003 (4)	0.017 (4)	0.001 (3)
C16	0.044 (5)	0.034 (4)	0.029 (3)	−0.002 (3)	0.013 (3)	−0.004 (3)
C17	0.052 (5)	0.026 (4)	0.029 (3)	−0.001 (3)	0.016 (3)	−0.001 (3)
C18	0.067 (6)	0.029 (4)	0.026 (3)	0.006 (3)	0.017 (4)	0.003 (3)
C19	0.045 (5)	0.033 (4)	0.034 (4)	0.003 (3)	0.017 (3)	0.003 (3)
C20	0.042 (4)	0.019 (3)	0.039 (4)	0.003 (3)	0.008 (3)	0.000 (3)
C21	0.031 (4)	0.038 (4)	0.038 (4)	0.005 (3)	0.006 (3)	−0.008 (3)
C22	0.027 (4)	0.031 (4)	0.044 (4)	0.000 (3)	0.007 (3)	−0.002 (3)
C23	0.055 (6)	0.044 (5)	0.051 (5)	0.018 (4)	0.011 (4)	0.010 (4)
C24	0.044 (5)	0.062 (6)	0.048 (5)	−0.003 (4)	−0.001 (4)	0.008 (4)
C25	0.053 (6)	0.045 (5)	0.059 (5)	0.010 (4)	0.003 (4)	0.015 (4)
C26	0.048 (5)	0.046 (5)	0.060 (5)	0.005 (4)	0.020 (4)	−0.002 (4)
C27	0.041 (5)	0.053 (5)	0.042 (4)	0.006 (4)	0.007 (4)	0.003 (4)
C28	0.031 (4)	0.038 (4)	0.055 (4)	0.010 (3)	0.020 (3)	0.006 (4)
C29	0.040 (5)	0.049 (5)	0.046 (4)	0.000 (4)	0.007 (4)	−0.006 (4)
C30	0.131 (9)	0.039 (5)	0.026 (4)	0.010 (5)	0.005 (5)	−0.008 (3)
C31	0.080 (7)	0.065 (6)	0.040 (5)	0.016 (5)	0.026 (5)	0.003 (4)
C32	0.118 (10)	0.134 (11)	0.070 (7)	−0.007 (9)	0.061 (7)	−0.006 (7)

Geometric parameters (Å, °)

O1—C3	1.210 (9)	C15—C16	1.511 (11)
O2—C11	1.432 (8)	C15—H15A	0.9700
O2—H2	0.8200	C15—H15B	0.9700
O3—C16	1.430 (9)	C16—C17	1.502 (9)
O3—C20	1.455 (8)	C16—H16	0.9800
O4—C31	1.340 (11)	C17—C18	1.499 (10)
O4—C21	1.458 (9)	C18—C30	1.507 (13)
O5—C31	1.196 (13)	C18—C19	1.546 (10)
O6—C22	1.435 (9)	C18—H18	0.9800
O6—H6	0.8200	C19—C20	1.505 (10)
C1—C2	1.535 (12)	C19—H19A	0.9700
C1—C10	1.540 (10)	C19—H19B	0.9700
C1—H1A	0.9700	C20—C21	1.513 (10)
C1—H1B	0.9700	C20—H20	0.9800
C2—C3	1.499 (11)	C21—C22	1.557 (10)
C2—H2A	0.9700	C21—H21	0.9800
C2—H2B	0.9700	C22—C24	1.497 (11)
C3—C4	1.533 (11)	C22—C23	1.515 (11)
C4—C25	1.518 (11)	C23—H23A	0.9600
C4—C26	1.551 (10)	C23—H23B	0.9600
C4—C5	1.554 (11)	C23—H23C	0.9600
C5—C6	1.515 (10)	C24—H24A	0.9600
C5—C10	1.550 (10)	C24—H24B	0.9600
C5—H5	0.9800	C24—H24C	0.9600
C6—C7	1.518 (11)	C25—H25A	0.9600
C6—H6A	0.9700	C25—H25B	0.9600
C6—H6B	0.9700	C25—H25C	0.9600
C7—C8	1.549 (10)	C26—H26A	0.9600

C7—H7A	0.9700	C26—H26B	0.9600
C7—H7B	0.9700	C26—H26C	0.9600
C8—C28	1.545 (10)	C27—H27A	0.9600
C8—C14	1.564 (10)	C27—H27B	0.9600
C8—C9	1.579 (10)	C27—H27C	0.9600
C9—C11	1.533 (9)	C28—H28A	0.9600
C9—C10	1.589 (10)	C28—H28B	0.9600
C9—H9	0.9800	C28—H28C	0.9600
C10—C27	1.511 (10)	C29—H29A	0.9600
C11—C12	1.518 (10)	C29—H29B	0.9600
C11—H11	0.9800	C29—H29C	0.9600
C12—C13	1.492 (10)	C30—H30A	0.9600
C12—H12A	0.9700	C30—H30B	0.9600
C12—H12B	0.9700	C30—H30C	0.9600
C13—C17	1.308 (10)	C31—C32	1.466 (15)
C13—C14	1.507 (9)	C32—H32A	0.9600
C14—C29	1.535 (11)	C32—H32B	0.9600
C14—C15	1.573 (10)	C32—H32C	0.9600
C11—O2—H2	109.5	C17—C16—H16	109.4
C16—O3—C20	110.0 (5)	C15—C16—H16	109.4
C31—O4—C21	119.4 (7)	C13—C17—C18	131.0 (7)
C22—O6—H6	109.5	C13—C17—C16	110.7 (6)
C2—C1—C10	112.7 (6)	C18—C17—C16	118.2 (6)
C2—C1—H1A	109.1	C17—C18—C30	111.2 (7)
C10—C1—H1A	109.1	C17—C18—C19	107.5 (6)
C2—C1—H1B	109.1	C30—C18—C19	113.1 (7)
C10—C1—H1B	109.1	C17—C18—H18	108.3
H1A—C1—H1B	107.8	C30—C18—H18	108.3
C3—C2—C1	117.3 (7)	C19—C18—H18	108.3
C3—C2—H2A	108.0	C20—C19—C18	111.9 (6)
C1—C2—H2A	108.0	C20—C19—H19A	109.2
C3—C2—H2B	108.0	C18—C19—H19A	109.2
C1—C2—H2B	108.0	C20—C19—H19B	109.2
H2A—C2—H2B	107.2	C18—C19—H19B	109.2
O1—C3—C2	121.0 (7)	H19A—C19—H19B	107.9
O1—C3—C4	121.5 (7)	O3—C20—C19	109.1 (6)
C2—C3—C4	117.1 (7)	O3—C20—C21	105.5 (5)
C25—C4—C3	111.4 (6)	C19—C20—C21	116.1 (6)
C25—C4—C26	108.2 (7)	O3—C20—H20	108.6
C3—C4—C26	103.9 (6)	C19—C20—H20	108.6
C25—C4—C5	115.9 (7)	C21—C20—H20	108.6
C3—C4—C5	108.6 (6)	O4—C21—C20	104.0 (6)
C26—C4—C5	108.0 (6)	O4—C21—C22	106.3 (5)
C6—C5—C10	110.7 (6)	C20—C21—C22	117.2 (6)
C6—C5—C4	115.5 (6)	O4—C21—H21	109.7
C10—C5—C4	114.4 (6)	C20—C21—H21	109.7
C6—C5—H5	105.0	C22—C21—H21	109.7
C10—C5—H5	105.0	O6—C22—C24	106.3 (6)
C4—C5—H5	105.0	O6—C22—C23	108.8 (6)

C5—C6—C7	109.4 (6)	C24—C22—C23	111.1 (7)
C5—C6—H6A	109.8	O6—C22—C21	107.5 (5)
C7—C6—H6A	109.8	C24—C22—C21	113.8 (6)
C5—C6—H6B	109.8	C23—C22—C21	109.0 (6)
C7—C6—H6B	109.8	C22—C23—H23A	109.5
H6A—C6—H6B	108.2	C22—C23—H23B	109.5
C6—C7—C8	116.0 (6)	H23A—C23—H23B	109.5
C6—C7—H7A	108.3	C22—C23—H23C	109.5
C8—C7—H7A	108.3	H23A—C23—H23C	109.5
C6—C7—H7B	108.3	H23B—C23—H23C	109.5
C8—C7—H7B	108.3	C22—C24—H24A	109.5
H7A—C7—H7B	107.4	C22—C24—H24B	109.5
C28—C8—C7	108.4 (6)	H24A—C24—H24B	109.5
C28—C8—C14	109.5 (6)	C22—C24—H24C	109.5
C7—C8—C14	110.1 (6)	H24A—C24—H24C	109.5
C28—C8—C9	109.8 (6)	H24B—C24—H24C	109.5
C7—C8—C9	109.9 (6)	C4—C25—H25A	109.5
C14—C8—C9	109.1 (5)	C4—C25—H25B	109.5
C11—C9—C8	110.8 (6)	H25A—C25—H25B	109.5
C11—C9—C10	114.3 (5)	C4—C25—H25C	109.5
C8—C9—C10	115.5 (5)	H25A—C25—H25C	109.5
C11—C9—H9	105.0	H25B—C25—H25C	109.5
C8—C9—H9	105.0	C4—C26—H26A	109.5
C10—C9—H9	105.0	C4—C26—H26B	109.5
C27—C10—C1	108.5 (6)	H26A—C26—H26B	109.5
C27—C10—C5	111.1 (6)	C4—C26—H26C	109.5
C1—C10—C5	106.9 (6)	H26A—C26—H26C	109.5
C27—C10—C9	106.7 (6)	H26B—C26—H26C	109.5
C1—C10—C9	113.1 (6)	C10—C27—H27A	109.5
C5—C10—C9	110.6 (5)	C10—C27—H27B	109.5
O2—C11—C12	109.6 (6)	H27A—C27—H27B	109.5
O2—C11—C9	109.5 (6)	C10—C27—H27C	109.5
C12—C11—C9	112.1 (6)	H27A—C27—H27C	109.5
O2—C11—H11	108.5	H27B—C27—H27C	109.5
C12—C11—H11	108.5	C8—C28—H28A	109.5
C9—C11—H11	108.5	C8—C28—H28B	109.5
C13—C12—C11	110.1 (6)	H28A—C28—H28B	109.5
C13—C12—H12A	109.6	C8—C28—H28C	109.5
C11—C12—H12A	109.6	H28A—C28—H28C	109.5
C13—C12—H12B	109.6	H28B—C28—H28C	109.5
C11—C12—H12B	109.6	C14—C29—H29A	109.5
H12A—C12—H12B	108.2	C14—C29—H29B	109.5
C17—C13—C12	129.3 (7)	H29A—C29—H29B	109.5
C17—C13—C14	114.6 (6)	C14—C29—H29C	109.5
C12—C13—C14	115.3 (6)	H29A—C29—H29C	109.5
C13—C14—C29	111.5 (6)	H29B—C29—H29C	109.5
C13—C14—C8	108.4 (6)	C18—C30—H30A	109.5
C29—C14—C8	112.8 (6)	C18—C30—H30B	109.5
C13—C14—C15	100.7 (6)	H30A—C30—H30B	109.5

C29—C14—C15	108.5 (6)	C18—C30—H30C	109.5
C8—C14—C15	114.3 (6)	H30A—C30—H30C	109.5
C16—C15—C14	106.3 (5)	H30B—C30—H30C	109.5
C16—C15—H15A	110.5	O5—C31—O4	122.9 (9)
C14—C15—H15A	110.5	O5—C31—C32	126.4 (10)
C16—C15—H15B	110.5	O4—C31—C32	110.6 (10)
C14—C15—H15B	110.5	C31—C32—H32A	109.5
H15A—C15—H15B	108.7	C31—C32—H32B	109.5
O3—C16—C17	112.1 (6)	H32A—C32—H32B	109.5
O3—C16—C15	112.2 (6)	C31—C32—H32C	109.5
C17—C16—C15	104.2 (6)	H32A—C32—H32C	109.5
O3—C16—H16	109.4	H32B—C32—H32C	109.5
C10—C1—C2—C3	14.8 (10)	C17—C13—C14—C8	−112.1 (7)
C1—C2—C3—O1	139.2 (8)	C12—C13—C14—C8	58.6 (8)
C1—C2—C3—C4	−47.6 (10)	C17—C13—C14—C15	8.2 (9)
O1—C3—C4—C25	−38.8 (10)	C12—C13—C14—C15	178.8 (6)
C2—C3—C4—C25	148.1 (7)	C28—C8—C14—C13	64.1 (7)
O1—C3—C4—C26	77.4 (9)	C7—C8—C14—C13	−176.7 (6)
C2—C3—C4—C26	−95.7 (8)	C9—C8—C14—C13	−56.1 (7)
O1—C3—C4—C5	−167.7 (7)	C28—C8—C14—C29	−171.9 (6)
C2—C3—C4—C5	19.1 (9)	C7—C8—C14—C29	−52.8 (8)
C25—C4—C5—C6	41.8 (9)	C9—C8—C14—C29	67.9 (7)
C3—C4—C5—C6	168.2 (6)	C28—C8—C14—C15	−47.2 (7)
C26—C4—C5—C6	−79.7 (8)	C7—C8—C14—C15	71.9 (7)
C25—C4—C5—C10	−88.3 (8)	C9—C8—C14—C15	−167.5 (5)
C3—C4—C5—C10	38.0 (8)	C13—C14—C15—C16	−16.3 (8)
C26—C4—C5—C10	150.1 (6)	C29—C14—C15—C16	−133.5 (7)
C10—C5—C6—C7	−70.2 (8)	C8—C14—C15—C16	99.7 (7)
C4—C5—C6—C7	157.9 (6)	C20—O3—C16—C17	−56.3 (8)
C5—C6—C7—C8	41.4 (9)	C20—O3—C16—C15	−173.2 (6)
C6—C7—C8—C28	−103.5 (8)	C14—C15—C16—O3	140.1 (6)
C6—C7—C8—C14	136.8 (7)	C14—C15—C16—C17	18.6 (8)
C6—C7—C8—C9	16.6 (9)	C12—C13—C17—C18	11.1 (14)
C28—C8—C9—C11	−63.7 (8)	C14—C13—C17—C18	−179.8 (7)
C7—C8—C9—C11	177.1 (6)	C12—C13—C17—C16	−165.5 (7)
C14—C8—C9—C11	56.3 (7)	C14—C13—C17—C16	3.6 (9)
C28—C8—C9—C10	68.2 (7)	O3—C16—C17—C13	−135.9 (7)
C7—C8—C9—C10	−51.0 (8)	C15—C16—C17—C13	−14.3 (9)
C14—C8—C9—C10	−171.7 (6)	O3—C16—C17—C18	47.0 (9)
C2—C1—C10—C27	−81.3 (8)	C15—C16—C17—C18	168.6 (7)
C2—C1—C10—C5	38.6 (8)	C13—C17—C18—C30	−94.0 (11)
C2—C1—C10—C9	160.6 (6)	C16—C17—C18—C30	82.4 (8)
C6—C5—C10—C27	−83.3 (8)	C13—C17—C18—C19	141.7 (8)
C4—C5—C10—C27	49.3 (8)	C16—C17—C18—C19	−41.9 (9)
C6—C5—C10—C1	158.6 (6)	C17—C18—C19—C20	48.9 (9)
C4—C5—C10—C1	−68.9 (8)	C30—C18—C19—C20	−74.2 (8)
C6—C5—C10—C9	35.0 (8)	C16—O3—C20—C19	65.3 (7)
C4—C5—C10—C9	167.5 (6)	C16—O3—C20—C21	−169.3 (6)
C11—C9—C10—C27	−84.3 (7)	C18—C19—C20—O3	−62.6 (8)

C8—C9—C10—C27	145.4 (6)	C18—C19—C20—C21	178.4 (6)
C11—C9—C10—C1	34.9 (8)	C31—O4—C21—C20	−125.2 (7)
C8—C9—C10—C1	−95.4 (7)	C31—O4—C21—C22	110.5 (8)
C11—C9—C10—C5	154.8 (6)	O3—C20—C21—O4	66.9 (7)
C8—C9—C10—C5	24.5 (8)	C19—C20—C21—O4	−172.1 (6)
C8—C9—C11—O2	−177.1 (5)	O3—C20—C21—C22	−176.1 (6)
C10—C9—C11—O2	50.3 (8)	C19—C20—C21—C22	−55.1 (9)
C8—C9—C11—C12	−55.3 (8)	O4—C21—C22—O6	−173.8 (5)
C10—C9—C11—C12	172.1 (6)	C20—C21—C22—O6	70.5 (8)
O2—C11—C12—C13	175.4 (6)	O4—C21—C22—C24	68.7 (7)
C9—C11—C12—C13	53.7 (8)	C20—C21—C22—C24	−47.0 (9)
C11—C12—C13—C17	112.2 (9)	O4—C21—C22—C23	−56.0 (8)
C11—C12—C13—C14	−56.8 (9)	C20—C21—C22—C23	−171.7 (7)
C17—C13—C14—C29	123.1 (8)	C21—O4—C31—O5	−4.5 (13)
C12—C13—C14—C29	−66.2 (9)	C21—O4—C31—C32	176.9 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O6^{i}	0.82	2.09	2.904 (7)	172
O6—H6···O1^{ii}	0.82	2.07	2.867 (8)	165

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

Fig. 1

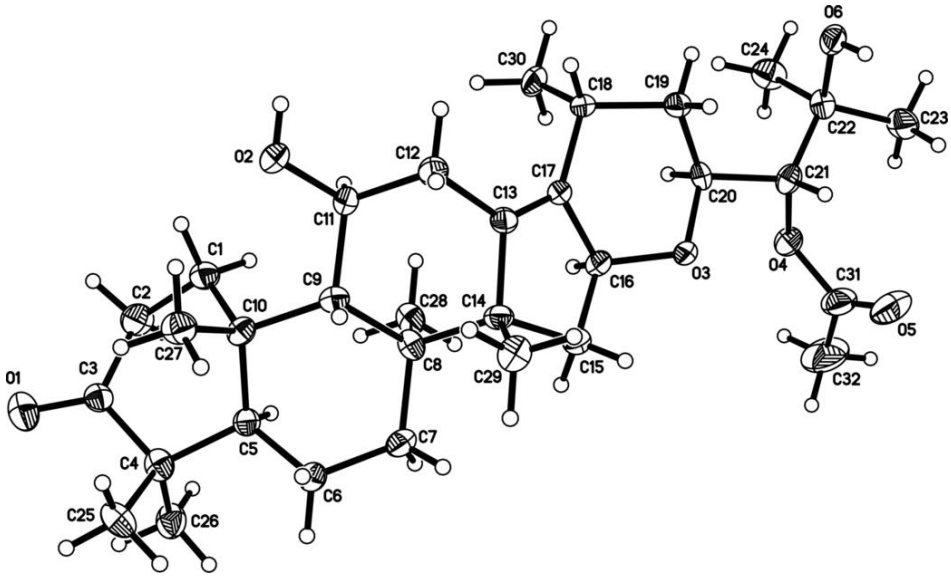
O6
C24
C30
C18
C19
C22
C23
O2
C12
C17
C21
C13
C20
C11
C1
O4
C31
C9
C16
O3
C2
C10
C28
C14
O5
C27
C8
C29
C15
C32
O1
C3
C5
C7
C4
C6
C25
C26

Fig. 2