Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,19-Dihydroxy-3-oxo-(2*α*,4*α*,19*α*)-24-nor-olean-12-en-28-oic acid monohydrate

Watcharee Waratchareeyakul,[a] Kittisak Seatyoa,[a] Suchada Chantrapromma[b]‡ and Hoong-Kun Fun[c]*

[a]Department of Chemistry, Faculty of Science and Technology, Rambhai Barni Rajabhat University, Muang, Chantaburi 22000, Thailand, [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

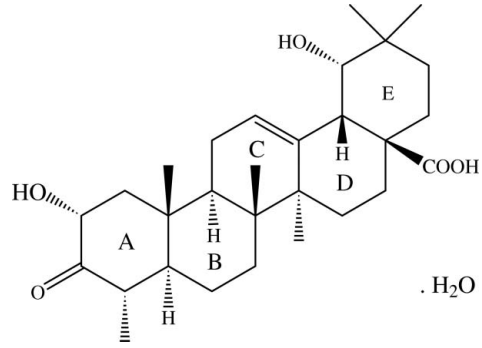
Received 9 September 2007; accepted 11 September 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.110; data-to-parameter ratio = 17.0.

The title triterpenoid compound, $C_{29}H_{44}O_5\cdot H_2O$, was isolated from *Terminalia triptera*. The molecule contains five fused six-membered rings, with three rings in chair, one in an envelope and one in half-chair conformations. The *D* and *E* rings are *cis*-fused, while the other ring junctions are *trans*-fused. An intramolecular O—H···O hydrogen bond generates an *S*(5) ring motif. In the crystal structure, the molecules are linked into a three-dimensional network by O—H···O hydrogen bonds and a weak C—H···O intermolecular interaction. The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds and weak C—H···O interactions.

Related literature

For related literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related literature on values of bond lengths, see: Allen *et al.* (1987). For related literature on ring conformations, see: Cremer & Pople (1975). For related structures, see: *e.g.* Chantrapromma *et al.* (2003); Chen *et al.* (2006); Rahman *et al.* (2002). For related literature on alkaloids, flavanoids, triterpenoids and their biological activities, see: *e.g.* Ahn *et al.* (2002); Batawila *et al.* (2005); Chen *et al.* (2006); Malekzadeh *et al.* (2001); Pawar & Bhutani (2005); Kaur *et al.* (2002); Rahman *et al.* (2002); Sabu & Kuttan (2002); Saleem *et al.* (2001, 2002); Sumitra *et al.* (2001); Yukawa *et al.* (1996).



Experimental

Crystal data

$C_{29}H_{44}O_5\cdot H_2O$
M_r = 490.66
Orthorhombic, $P2_12_12_1$
a = 11.8983 (3) Å
b = 14.3772 (3) Å
c = 15.1835 (3) Å
V = 2597.35 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100.0 (1) K
0.58 × 0.41 × 0.18 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.952, T_{max} = 0.985
37602 measured reflections
5485 independent reflections
5078 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.110
S = 1.04
5485 reflections
322 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O1···O2	0.75	2.39	2.6933 (17)	106
O1—H1O1···O1W^{i}	0.75	1.96	2.692 (2)	167
O1W—H1W1···O5	0.87	1.80	2.6389 (19)	163
O4—H1O4···O1^{ii}	0.80	1.84	2.6357 (16)	169
O5—H1O5···O3^{iii}	0.84	1.95	2.7642 (15)	161
O1W—H2W1···O2^{iv}	0.91	2.42	2.8426 (19)	109
C6—H6A···O4^{v}	0.97	2.50	3.3849 (18)	152
C18—H18A···O3	0.98	2.40	2.8006 (18)	104
C26—H26A···O5	0.96	2.45	3.0012 (18)	116

Symmetry codes: (i) $-x+\frac{1}{2}, -y, z-\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+2$; (iv) $-x+\frac{1}{2}, -y, z+\frac{1}{2}$; (v) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); 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).

WW and KS thank Rambhai Barni Rajabhat University for financial support. The authors also thank the Prince of Songkla University, the Malaysian Government and Universiti Sains Malaysia for Scientific Advancement Grant Allocation (SAGA) No. 304/PFIZIK/653003/A118.

‡ Additional correspondence author, email: suchada.c@psu.ac.th.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2205).

References

Ahn, M. J., Kim, C. Y., Lee, J. S., Kim, T. G., Kim, S. H., Lee, C. K., Lee, B. B., Shin, C. G., Huh, H. & Kim, J. (2002). *Planta Med.* **68**, 457–459.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Batawila, K., Kokou, K., Koumaglo, K., Gbeassor, M., de Foucault, B., Bouchet, Ph. & Akpagana, K. (2005). *Fitoterapia*, **76**, 264–268.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12a) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Fun, H.-K., Razak, I. A., Laphookhieo, S. & Karalai, C. (2003). *Acta Cryst.* E**59**, o1864–o1866.

Chen, H.-D., Yang, S.-P., Liao, S.-G., Zhang, C.-R. & Yue, J.-M. (2006). *Helv. Chim. Acta*, **89**, 1971–1977.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Kaur, K., Arora, S., Kumar, S. & Nagpal, A. (2002). *Food. Chem. Toxicol.* **40**, 1475–1482.

Malekzadeh, F., Ehsanifar, H., Shahamat, M., Levin, M. & Colwell, R. R. (2001). *Int. J. Antimicrob. Agents*, **18**, 85–88.

Pawar, R. S. & Bhutani, K. K. (2005). *Phytomedicine*, **12**, 391–393.

Rahman, A., Zareen, S., Choudhary, M. I., Ngounou, F. N., Yasin, A. & Parvez, M. (2002). *Tetrahedron Lett.* **43**, 6233–6236.

Sabu, M. C. & Kuttan, R. (2002). *J. Ethnopharmacol.* **81**, 155–160.

Saleem, A., Ahotupa, M. & Pihlaja, K. (2001). *Z. Naturforsch. Teil C*, **56**, 973–978.

Saleem, A., Husheem, M., Harkonen, P. & Pihlaja, K. (2002). *J. Ethnopharmacol.* **81**, 327–336.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Sumitra, M., Manikandan, P., Kumar, D. A., Arutselvan, N., Balakrishna, K., Manohar, B. M. & Puvanakrishnan, R. (2001). *Mol. Cell. Biochem.* **224**, 135–142.

Yukawa, T. A., Kurokawa, M., Sato, H., Yoshida, Y., Kageyama, S., Hasegawa, T., Namba, T., Imakita, M., Hozumi, T. & Shiraki, K. (1996). *Antiviral Res.* **32**, 63–70.

supplementary materials

Acta Cryst. (2007). E**63**, o4062-o4063 [**doi:10.1107/S1600536807044339**]

2,19-Dihydroxy-3-oxo-(2α,4α,19α)-24-nor-olean-12-en-28-oic acid monohydrate

W. Waratchareeyakul, K. Seatyoa, S. Chantrapromma and H.-K. Fun

Comment

Terminalia triptera is a large tree of the flowering plant family, Combretaceae, comprising around 100 species distributed throughout the tropical regions of the world. This genus takes its name from the Latin word, "terminus", referring to the fact that the leaves appear at the very tips of their shoots. Plants of this genus are known especially as a source of secondary metabolites, *e.g.* triterpenoids, flavonoids, alkaloids and tannins. Some of these substances have antifungal (Batawila *et al.*, 2005), antibacterial (Malekzadeh *et al.*, 2001), antiviral (Ahn *et al.*, 2002; Saleem *et al.*, 2001; Yukawa *et al.*, 1996), antioxidant (Sabu & Kuttan, 2002), antimutagenic (Kaur *et al.*, 2002), anticancer (Saleem *et al.*, 2002), antidiabetic activities (Sabu & Kuttan, 2002) and some exhibit cardiac protective action (Pawar & Bhutani, 2005; Sumitra *et al.*, 2001). In our ongoing research on finding bioactive compounds from natural products, we have examined the barks of *Terminalia triptera*, collected from Chantaburi province in the eastern part of Thailand. The title compound was isolated from a dichloromethane extract. It was previously isolated from the *Quercus aliena* Blume, Fagaceae family (Chen *et al.*, 2006) and has shown several biological activities (Chen *et al.* (2006). We report here the crystal structure of the title compound.

The title molecule (Fig. 1) has five fused six-membered rings (*A*/*B*/*C*/*D*/*E*); the cyclohexane rings *A*, *B* and *E* are in standard chair conformation, cyclohexene ring *C* is in an envelope conformation with the puckering parameter (Cremer & Pople, 1975) Q = 0.579 (1) Å, θ = 53.5 (1)° and φ = 354.97 (17)°, atom C8 having the maximum deviation of 0.4084 (13) Å, cyclohexane ring *D* is in a half-chair conformation with Q = 0.502 (1) Å, θ = 150.2 (1)° and φ = 7.1 (3)°. The *D* and *E* rings are *cis*-fused. The other ring junctions are *tran*-fused. The orientation of the carboxylic acid group with respect to cyclohexane ring D is indicated by the torsion angles C16—C17—C27—O3 = -134.99 (15)° and C16—C17—C27—O4 = 48.93 (14)°. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

An intramolecular O1—H1O1···O2 hydrogen bond generates an S(5) ring motif (Bernstein *et al.*, 1995). The water molecule forms an intermolecular O—H···O hydrogen bond with one of the hydroxyl groups (O1W—H1W1···O5, Fig. 1). In the crystal packing (Fig. 2), the triterpenoid molecules are linked with the water molecule by O—H···O hydrogen bonds, forming sheets parallel to the *bc* plane and these sheets are further connected into a three dimensional network by O—H···O hydrogen bonds and weak C—H···O interactions (Fig. 2 and Table 1). The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds and weak C—H···O intramolecular interactions (Table 1).

Experimental

Air-dried barks of *Terminalia triptera* (5.2 kg) were extracted with hexane, CH_2Cl_2 and MeOH, successively. The CH_2Cl_2 extract was dried under reduced pressure to yield a crude extract (13.50 g) which was subjected to quick column chromatography (QCC) over silica gel and eluted initially with CH_2Cl_2 enriched with EtOAc, followed by an increasing amount of MeOH in EtOAc and finally with MeOH. The eluents were separated into 11 fractions (F1—F11) on the basis of TLC analysis. Fraction F7 (1.25 g) was separated by QCC over silica gel and eluted initially with EtOAc:hexane (2:3 *v*/*v*), to give 6 fractions (F7A—F7F). Fraction F7B was further purified by crystallization to give the title compound (0.1691 g).

Colorless single crystals of the title compound were recrystallized from hexane:CH_2Cl_2:EtOAC (3:3:4 *v*/*v*) after a few days (*M*.p. 512–514 K).

Refinement

Water and hydroxyl H atoms were located in a difference map whereas the remaining H atoms were positioned geometrically. All hydrogen atoms were allowed to ride on their parent atoms, with the C—H distances in the range 0.93 - 0.98 Å and O—H in the range 0.75 - 0.91 Å. The U_{iso}(H) values were set equal to $1.5U_{eq}$ of the carrier atom for methyl, hydroxyl and water H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. A total of 4444 Friedel pairs were merged before the final refinement as there are no significant anomalous dispersion effects. The configuration was assigned on the basis of the earlier literature absolute configuration (Chen *et al.*, 2006).

Figures

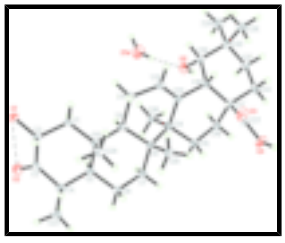


Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme. Hydrogen bonds are shown as dashed lines.

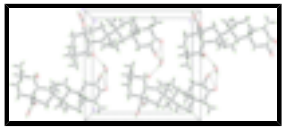


Fig. 2. The crystal packing of the title compound, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

2,19-Dihydroxy-3-oxo-(2*α*,4*α*,19*α*)-24-nor-olean-12-en-28-oic acid monohydrate

Crystal data

$C_{29}H_{44}O_5 \cdot H_2O$	D_x = 1.255 Mg m^{-3}
M_r = 490.66	Melting point: 512-514 K K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5485 reflections
a = 11.8983 (3) Å	θ = 2.0–33.2°
b = 14.3772 (3) Å	μ = 0.09 mm^{-1}
c = 15.1835 (3) Å	*T* = 100.0 (1) K
V = 2597.35 (10) Å^3	Block, colorless
Z = 4	0.58 × 0.41 × 0.18 mm
F_{000} = 1072	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	5485 independent reflections
Radiation source: fine-focus sealed tube	5078 reflections with $I > 2\sigma(I)$

Monochromator: graphite

Detector resolution: 8.33 pixels mm^{-1}

T = 100.0(1) K

ω scans

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

T_{min} = 0.952, T_{max} = 0.985

37602 measured reflections

R_{int} = 0.037

θ_{max} = 33.2º

θ_{min} = 2.0º

h = −17→18

k = −14→22

l = −22→23

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.039

$wR(F^2)$ = 0.110

S = 1.04

5485 reflections

322 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

$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.3157P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.33 e $Å^{-3}$

$\Delta\rho_{min}$ = −0.43 e $Å^{-3}$

Extinction correction: none

Special details

Experimental. The low-temparture data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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	0.51439 (10)	0.09740 (7)	0.38914 (7)	0.0204 (2)
H1O1	0.4547	0.0845	0.3784	0.031*
O2	0.36546 (11)	0.22455 (9)	0.33128 (8)	0.0268 (2)
O3	0.52258 (9)	0.37362 (9)	1.00414 (8)	0.0238 (2)
O4	0.37219 (9)	0.45694 (8)	1.04344 (8)	0.0229 (2)
H1O4	0.4132	0.4950	1.0647	0.034*
O5	0.24247 (9)	0.10261 (8)	0.94160 (7)	0.0207 (2)
H1O5	0.1826	0.1146	0.9692	0.031*

O1W	0.18414 (14)	−0.03114 (10)	0.83170 (10)	0.0392 (3)
H1W1	0.2135	0.0153	0.8597	0.059*
H2W1	0.2348	−0.0780	0.8284	0.059*
C1	0.48950 (11)	0.17482 (9)	0.53030 (8)	0.0149 (2)
H1A	0.5503	0.1394	0.5565	0.018*
H1B	0.4208	0.1395	0.5383	0.018*
C2	0.51212 (12)	0.18495 (10)	0.43130 (9)	0.0167 (2)
H2A	0.5863	0.2136	0.4241	0.020*
C3	0.42575 (12)	0.24988 (10)	0.39091 (9)	0.0173 (2)
C4	0.41499 (12)	0.34438 (9)	0.43416 (9)	0.0164 (2)
H4A	0.4878	0.3760	0.4306	0.020*
C5	0.38724 (10)	0.32674 (9)	0.53292 (9)	0.0141 (2)
H5A	0.3183	0.2895	0.5338	0.017*
C6	0.36119 (12)	0.41553 (9)	0.58430 (9)	0.0169 (2)
H6A	0.3069	0.4525	0.5520	0.020*
H6B	0.4292	0.4521	0.5908	0.020*
C7	0.31401 (11)	0.39177 (9)	0.67552 (9)	0.0163 (2)
H7A	0.2416	0.3617	0.6682	0.020*
H7B	0.3017	0.4491	0.7077	0.020*
C8	0.39051 (10)	0.32789 (9)	0.73082 (8)	0.0133 (2)
C9	0.43285 (11)	0.24511 (9)	0.67389 (9)	0.0139 (2)
H9A	0.3659	0.2069	0.6636	0.017*
C10	0.47826 (11)	0.26784 (9)	0.57986 (8)	0.0134 (2)
C11	0.51232 (13)	0.18212 (10)	0.72738 (9)	0.0192 (3)
H11A	0.5069	0.1192	0.7047	0.023*
H11B	0.5889	0.2031	0.7184	0.023*
C12	0.48871 (11)	0.18022 (10)	0.82437 (9)	0.0159 (2)
H12A	0.5362	0.1444	0.8592	0.019*
C13	0.40569 (10)	0.22535 (9)	0.86518 (8)	0.0132 (2)
C14	0.32271 (10)	0.28331 (9)	0.81068 (8)	0.0131 (2)
C15	0.26629 (11)	0.36017 (9)	0.86653 (9)	0.0152 (2)
H15A	0.3171	0.4127	0.8707	0.018*
H15B	0.1990	0.3811	0.8364	0.018*
C16	0.23455 (10)	0.32886 (9)	0.95957 (9)	0.0151 (2)
H16A	0.1818	0.2776	0.9561	0.018*
H16B	0.1982	0.3797	0.9904	0.018*
C17	0.33939 (10)	0.29809 (9)	1.01102 (8)	0.0135 (2)
C18	0.39615 (10)	0.21458 (9)	0.96506 (8)	0.0135 (2)
H18A	0.4735	0.2130	0.9872	0.016*
C19	0.34447 (11)	0.11884 (10)	0.98870 (9)	0.0156 (2)
H19A	0.3980	0.0718	0.9682	0.019*
C20	0.32994 (11)	0.10269 (10)	1.08802 (9)	0.0168 (2)
C21	0.25994 (11)	0.18336 (10)	1.12574 (9)	0.0166 (2)
H21A	0.1858	0.1822	1.0993	0.020*
H21B	0.2510	0.1743	1.1887	0.020*
C22	0.31287 (11)	0.27781 (10)	1.10947 (9)	0.0159 (2)
H22A	0.3821	0.2819	1.1430	0.019*
H22B	0.2625	0.3256	1.1313	0.019*
C23	0.32822 (13)	0.40318 (10)	0.38653 (10)	0.0211 (3)

H23A	0.3446	0.4038	0.3246	0.032*
H23B	0.3304	0.4656	0.4089	0.032*
H23C	0.2548	0.3774	0.3959	0.032*
C24	0.59359 (11)	0.31730 (10)	0.57976 (9)	0.0177 (2)
H24A	0.6217	0.3206	0.5206	0.027*
H24B	0.6454	0.2830	0.6157	0.027*
H24C	0.5853	0.3790	0.6030	0.027*
C25	0.48953 (11)	0.38732 (10)	0.76549 (9)	0.0170 (2)
H25A	0.5099	0.4327	0.7220	0.025*
H25B	0.5528	0.3478	0.7773	0.025*
H25C	0.4673	0.4183	0.8187	0.025*
C26	0.22587 (11)	0.21961 (10)	0.77842 (9)	0.0169 (2)
H26A	0.1749	0.2080	0.8262	0.025*
H26B	0.2564	0.1617	0.7579	0.025*
H26C	0.1864	0.2497	0.7312	0.025*
C27	0.42208 (11)	0.37913 (10)	1.01693 (9)	0.0156 (2)
C28	0.44652 (12)	0.09760 (11)	1.13187 (10)	0.0212 (3)
H28A	0.4892	0.0481	1.1058	0.032*
H28B	0.4377	0.0861	1.1938	0.032*
H28C	0.4853	0.1555	1.1234	0.032*
C29	0.26903 (14)	0.01038 (11)	1.10450 (10)	0.0230 (3)
H29A	0.1934	0.0146	1.0829	0.034*
H29B	0.2679	−0.0025	1.1665	0.034*
H29C	0.3077	−0.0388	1.0743	0.034*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0267 (5)	0.0164 (4)	0.0182 (4)	0.0060 (4)	−0.0010 (4)	−0.0018 (4)
O2	0.0336 (6)	0.0244 (5)	0.0225 (5)	0.0055 (5)	−0.0084 (5)	−0.0024 (4)
O3	0.0131 (4)	0.0263 (5)	0.0321 (6)	−0.0038 (4)	0.0029 (4)	−0.0081 (5)
O4	0.0185 (4)	0.0162 (4)	0.0341 (6)	−0.0015 (4)	−0.0005 (4)	−0.0079 (4)
O5	0.0165 (4)	0.0246 (5)	0.0209 (4)	−0.0043 (4)	−0.0010 (4)	−0.0027 (4)
O1W	0.0420 (8)	0.0329 (7)	0.0428 (8)	−0.0038 (6)	−0.0015 (6)	−0.0116 (6)
C1	0.0170 (5)	0.0136 (5)	0.0141 (5)	0.0030 (4)	0.0008 (4)	0.0015 (4)
C2	0.0195 (6)	0.0151 (5)	0.0157 (5)	0.0031 (5)	0.0010 (4)	0.0007 (4)
C3	0.0209 (6)	0.0153 (5)	0.0158 (5)	0.0025 (5)	0.0008 (4)	0.0032 (4)
C4	0.0177 (5)	0.0138 (5)	0.0176 (5)	0.0020 (4)	−0.0016 (4)	0.0025 (4)
C5	0.0133 (5)	0.0133 (5)	0.0157 (5)	0.0027 (4)	−0.0013 (4)	0.0009 (4)
C6	0.0191 (5)	0.0122 (5)	0.0193 (5)	0.0040 (4)	−0.0009 (5)	0.0015 (4)
C7	0.0164 (5)	0.0137 (5)	0.0189 (5)	0.0049 (4)	−0.0015 (4)	−0.0006 (4)
C8	0.0122 (5)	0.0117 (5)	0.0161 (5)	0.0016 (4)	−0.0016 (4)	−0.0013 (4)
C9	0.0145 (5)	0.0121 (5)	0.0152 (5)	0.0029 (4)	−0.0001 (4)	0.0002 (4)
C10	0.0130 (5)	0.0118 (5)	0.0154 (5)	0.0020 (4)	−0.0009 (4)	0.0009 (4)
C11	0.0227 (6)	0.0199 (6)	0.0149 (5)	0.0108 (5)	0.0021 (5)	0.0024 (5)
C12	0.0158 (5)	0.0165 (5)	0.0156 (5)	0.0045 (4)	−0.0002 (4)	0.0006 (4)
C13	0.0117 (5)	0.0134 (5)	0.0146 (5)	0.0015 (4)	−0.0007 (4)	−0.0015 (4)
C14	0.0117 (5)	0.0123 (5)	0.0153 (5)	0.0020 (4)	−0.0005 (4)	−0.0021 (4)

C15	0.0130 (5)	0.0148 (5)	0.0179 (5)	0.0031 (4)	−0.0008 (4)	−0.0027 (4)
C16	0.0110 (5)	0.0161 (5)	0.0183 (5)	0.0012 (4)	0.0006 (4)	−0.0028 (5)
C17	0.0104 (5)	0.0146 (5)	0.0154 (5)	0.0002 (4)	0.0003 (4)	−0.0024 (4)
C18	0.0119 (5)	0.0139 (5)	0.0145 (5)	0.0019 (4)	0.0003 (4)	−0.0016 (4)
C19	0.0135 (5)	0.0154 (5)	0.0178 (5)	−0.0001 (4)	0.0007 (4)	−0.0015 (4)
C20	0.0158 (5)	0.0172 (6)	0.0174 (5)	−0.0011 (5)	0.0004 (4)	0.0009 (5)
C21	0.0142 (5)	0.0196 (6)	0.0161 (5)	−0.0004 (5)	0.0025 (4)	−0.0003 (5)
C22	0.0139 (5)	0.0184 (5)	0.0154 (5)	−0.0006 (5)	0.0011 (4)	−0.0025 (4)
C23	0.0240 (6)	0.0185 (6)	0.0206 (6)	0.0042 (5)	−0.0050 (5)	0.0043 (5)
C24	0.0132 (5)	0.0186 (6)	0.0213 (6)	0.0002 (4)	−0.0018 (4)	0.0015 (5)
C25	0.0147 (5)	0.0170 (5)	0.0192 (5)	−0.0005 (5)	−0.0015 (4)	−0.0011 (5)
C26	0.0149 (5)	0.0178 (6)	0.0181 (5)	−0.0008 (5)	−0.0021 (4)	−0.0026 (5)
C27	0.0157 (5)	0.0161 (5)	0.0149 (5)	−0.0007 (5)	−0.0001 (4)	−0.0024 (4)
C28	0.0185 (6)	0.0242 (6)	0.0211 (6)	0.0032 (5)	−0.0007 (5)	0.0023 (5)
C29	0.0268 (7)	0.0178 (6)	0.0244 (6)	−0.0036 (5)	0.0032 (5)	0.0017 (5)

Geometric parameters (Å, °)

O1—C2	1.4123 (17)	C13—C14	1.5343 (18)
O1—H1O1	0.7517	C14—C15	1.5463 (18)
O2—C3	1.2112 (18)	C14—C26	1.5511 (19)
O3—C27	1.2140 (17)	C15—C16	1.5301 (19)
O4—C27	1.3288 (17)	C15—H15A	0.9700
O4—H1O4	0.8017	C15—H15B	0.9700
O5—C19	1.4279 (17)	C16—C17	1.5369 (18)
O5—H1O5	0.8436	C16—H16A	0.9700
O1W—H1W1	0.8654	C16—H16B	0.9700
O1W—H2W1	0.9063	C17—C27	1.5275 (19)
C1—C2	1.5340 (19)	C17—C18	1.5443 (18)
C1—C10	1.5403 (19)	C17—C22	1.5553 (18)
C1—H1A	0.9700	C18—C19	1.5497 (19)
C1—H1B	0.9700	C18—H18A	0.9800
C2—C3	1.5177 (19)	C19—C20	1.5356 (19)
C2—H2A	0.9800	C19—H19A	0.9800
C3—C4	1.514 (2)	C20—C29	1.533 (2)
C4—C23	1.5178 (19)	C20—C21	1.5385 (19)
C4—C5	1.5561 (18)	C20—C28	1.540 (2)
C4—H4A	0.9800	C21—C22	1.517 (2)
C5—C6	1.5279 (19)	C21—H21A	0.9700
C5—C10	1.5487 (17)	C21—H21B	0.9700
C5—H5A	0.9800	C22—H22A	0.9700
C6—C7	1.5331 (19)	C22—H22B	0.9700
C6—H6A	0.9700	C23—H23A	0.9600
C6—H6B	0.9700	C23—H23B	0.9600
C7—C8	1.5418 (18)	C23—H23C	0.9600
C7—H7A	0.9700	C24—H24A	0.9600
C7—H7B	0.9700	C24—H24B	0.9600
C8—C25	1.5477 (19)	C24—H24C	0.9600
C8—C9	1.5548 (18)	C25—H25A	0.9600

C8—C14	1.5912 (18)	C25—H25B	0.9600
C9—C11	1.5408 (19)	C25—H25C	0.9600
C9—C10	1.5610 (18)	C26—H26A	0.9600
C9—H9A	0.9800	C26—H26B	0.9600
C10—C24	1.5456 (18)	C26—H26C	0.9600
C11—C12	1.4995 (19)	C28—H28A	0.9600
C11—H11A	0.9700	C28—H28B	0.9600
C11—H11B	0.9700	C28—H28C	0.9600
C12—C13	1.3344 (18)	C29—H29A	0.9600
C12—H12A	0.9300	C29—H29B	0.9600
C13—C18	1.5287 (18)	C29—H29C	0.9600
C2—O1—H1O1	107.5	C14—C15—H15B	108.8
C27—O4—H1O4	115.2	H15A—C15—H15B	107.7
C19—O5—H1O5	115.8	C15—C16—C17	110.71 (10)
H1W1—O1W—H2W1	109.4	C15—C16—H16A	109.5
C2—C1—C10	114.31 (11)	C17—C16—H16A	109.5
C2—C1—H1A	108.7	C15—C16—H16B	109.5
C10—C1—H1A	108.7	C17—C16—H16B	109.5
C2—C1—H1B	108.7	H16A—C16—H16B	108.1
C10—C1—H1B	108.7	C27—C17—C16	109.46 (11)
H1A—C1—H1B	107.6	C27—C17—C18	109.75 (10)
O1—C2—C3	112.21 (11)	C16—C17—C18	110.43 (10)
O1—C2—C1	111.28 (11)	C27—C17—C22	102.55 (10)
C3—C2—C1	109.60 (11)	C16—C17—C22	112.20 (10)
O1—C2—H2A	107.9	C18—C17—C22	112.16 (11)
C3—C2—H2A	107.9	C13—C18—C17	113.70 (11)
C1—C2—H2A	107.9	C13—C18—C19	110.44 (11)
O2—C3—C4	122.94 (13)	C17—C18—C19	114.36 (11)
O2—C3—C2	121.21 (13)	C13—C18—H18A	105.8
C4—C3—C2	115.71 (11)	C17—C18—H18A	105.8
C3—C4—C23	110.52 (11)	C19—C18—H18A	105.8
C3—C4—C5	106.83 (10)	O5—C19—C20	111.80 (11)
C23—C4—C5	113.93 (11)	O5—C19—C18	111.49 (11)
C3—C4—H4A	108.5	C20—C19—C18	113.98 (11)
C23—C4—H4A	108.5	O5—C19—H19A	106.3
C5—C4—H4A	108.5	C20—C19—H19A	106.3
C6—C5—C10	111.33 (11)	C18—C19—H19A	106.3
C6—C5—C4	113.51 (11)	C29—C20—C19	110.15 (11)
C10—C5—C4	112.60 (10)	C29—C20—C21	109.64 (11)
C6—C5—H5A	106.3	C19—C20—C21	108.21 (11)
C10—C5—H5A	106.3	C29—C20—C28	108.31 (12)
C4—C5—H5A	106.3	C19—C20—C28	109.28 (11)
C5—C6—C7	110.45 (11)	C21—C20—C28	111.25 (11)
C5—C6—H6A	109.6	C22—C21—C20	112.92 (11)
C7—C6—H6A	109.6	C22—C21—H21A	109.0
C5—C6—H6B	109.6	C20—C21—H21A	109.0
C7—C6—H6B	109.6	C22—C21—H21B	109.0
H6A—C6—H6B	108.1	C20—C21—H21B	109.0
C6—C7—C8	114.11 (11)	H21A—C21—H21B	107.8

C6—C7—H7A	108.7	C21—C22—C17	114.11 (11)
C8—C7—H7A	108.7	C21—C22—H22A	108.7
C6—C7—H7B	108.7	C17—C22—H22A	108.7
C8—C7—H7B	108.7	C21—C22—H22B	108.7
H7A—C7—H7B	107.6	C17—C22—H22B	108.7
C7—C8—C25	107.81 (11)	H22A—C22—H22B	107.6
C7—C8—C9	110.15 (10)	C4—C23—H23A	109.5
C25—C8—C9	111.42 (10)	C4—C23—H23B	109.5
C7—C8—C14	110.83 (10)	H23A—C23—H23B	109.5
C25—C8—C14	110.43 (10)	C4—C23—H23C	109.5
C9—C8—C14	106.24 (10)	H23A—C23—H23C	109.5
C11—C9—C8	110.83 (10)	H23B—C23—H23C	109.5
C11—C9—C10	113.12 (10)	C10—C24—H24A	109.5
C8—C9—C10	117.41 (10)	C10—C24—H24B	109.5
C11—C9—H9A	104.7	H24A—C24—H24B	109.5
C8—C9—H9A	104.7	C10—C24—H24C	109.5
C10—C9—H9A	104.7	H24A—C24—H24C	109.5
C1—C10—C24	108.77 (11)	H24B—C24—H24C	109.5
C1—C10—C5	108.10 (10)	C8—C25—H25A	109.5
C24—C10—C5	111.64 (11)	C8—C25—H25B	109.5
C1—C10—C9	107.17 (10)	H25A—C25—H25B	109.5
C24—C10—C9	113.86 (11)	C8—C25—H25C	109.5
C5—C10—C9	107.06 (10)	H25A—C25—H25C	109.5
C12—C11—C9	114.43 (11)	H25B—C25—H25C	109.5
C12—C11—H11A	108.7	C14—C26—H26A	109.5
C9—C11—H11A	108.7	C14—C26—H26B	109.5
C12—C11—H11B	108.7	H26A—C26—H26B	109.5
C9—C11—H11B	108.7	C14—C26—H26C	109.5
H11A—C11—H11B	107.6	H26A—C26—H26C	109.5
C13—C12—C11	125.86 (12)	H26B—C26—H26C	109.5
C13—C12—H12A	117.1	O3—C27—O4	122.91 (13)
C11—C12—H12A	117.1	O3—C27—C17	125.12 (13)
C12—C13—C18	117.80 (11)	O4—C27—C17	111.85 (11)
C12—C13—C14	119.34 (11)	C20—C28—H28A	109.5
C18—C13—C14	122.84 (11)	C20—C28—H28B	109.5
C13—C14—C15	111.82 (10)	H28A—C28—H28B	109.5
C13—C14—C26	109.13 (11)	C20—C28—H28C	109.5
C15—C14—C26	105.82 (10)	H28A—C28—H28C	109.5
C13—C14—C8	107.67 (10)	H28B—C28—H28C	109.5
C15—C14—C8	110.49 (10)	C20—C29—H29A	109.5
C26—C14—C8	111.95 (10)	C20—C29—H29B	109.5
C16—C15—C14	113.78 (11)	H29A—C29—H29B	109.5
C16—C15—H15A	108.8	C20—C29—H29C	109.5
C14—C15—H15A	108.8	H29A—C29—H29C	109.5
C16—C15—H15B	108.8	H29B—C29—H29C	109.5
C10—C1—C2—O1	−176.29 (11)	C18—C13—C14—C8	146.80 (12)
C10—C1—C2—C3	−51.58 (15)	C7—C8—C14—C13	−176.92 (10)
O1—C2—C3—O2	2.22 (19)	C25—C8—C14—C13	−57.52 (13)
C1—C2—C3—O2	−121.94 (15)	C9—C8—C14—C13	63.43 (12)

O1—C2—C3—C4	178.06 (11)	C7—C8—C14—C15	−54.54 (13)
C1—C2—C3—C4	53.89 (16)	C25—C8—C14—C15	64.86 (13)
O2—C3—C4—C23	−5.55 (19)	C9—C8—C14—C15	−174.19 (10)
C2—C3—C4—C23	178.69 (12)	C7—C8—C14—C26	63.13 (13)
O2—C3—C4—C5	118.89 (15)	C25—C8—C14—C26	−177.47 (11)
C2—C3—C4—C5	−56.86 (15)	C9—C8—C14—C26	−56.51 (13)
C3—C4—C5—C6	−173.84 (11)	C13—C14—C15—C16	−40.20 (15)
C23—C4—C5—C6	−51.50 (15)	C26—C14—C15—C16	78.51 (13)
C3—C4—C5—C10	58.52 (14)	C8—C14—C15—C16	−160.11 (10)
C23—C4—C5—C10	−179.14 (11)	C14—C15—C16—C17	60.46 (14)
C10—C5—C6—C7	−62.08 (14)	C15—C16—C17—C27	59.55 (13)
C4—C5—C6—C7	169.63 (11)	C15—C16—C17—C18	−61.38 (14)
C5—C6—C7—C8	55.68 (15)	C15—C16—C17—C22	172.70 (10)
C6—C7—C8—C25	75.38 (14)	C12—C13—C18—C17	153.38 (12)
C6—C7—C8—C9	−46.38 (15)	C14—C13—C18—C17	−28.20 (17)
C6—C7—C8—C14	−163.66 (11)	C12—C13—C18—C19	−76.50 (15)
C7—C8—C9—C11	178.31 (11)	C14—C13—C18—C19	101.92 (14)
C25—C8—C9—C11	58.72 (14)	C27—C17—C18—C13	−76.40 (13)
C14—C8—C9—C11	−61.60 (13)	C16—C17—C18—C13	44.36 (14)
C7—C8—C9—C10	46.19 (15)	C22—C17—C18—C13	170.31 (10)
C25—C8—C9—C10	−73.40 (14)	C27—C17—C18—C19	155.47 (11)
C14—C8—C9—C10	166.28 (10)	C16—C17—C18—C19	−83.77 (13)
C2—C1—C10—C24	−67.31 (14)	C22—C17—C18—C19	42.18 (14)
C2—C1—C10—C5	54.09 (14)	C13—C18—C19—O5	−51.30 (14)
C2—C1—C10—C9	169.17 (11)	C17—C18—C19—O5	78.47 (14)
C6—C5—C10—C1	173.37 (11)	C13—C18—C19—C20	−179.04 (11)
C4—C5—C10—C1	−57.84 (14)	C17—C18—C19—C20	−49.27 (15)
C6—C5—C10—C24	−67.03 (14)	O5—C19—C20—C29	47.44 (16)
C4—C5—C10—C24	61.76 (14)	C18—C19—C20—C29	175.01 (11)
C6—C5—C10—C9	58.22 (14)	O5—C19—C20—C21	−72.40 (14)
C4—C5—C10—C9	−173.00 (10)	C18—C19—C20—C21	55.18 (14)
C11—C9—C10—C1	61.30 (14)	O5—C19—C20—C28	166.31 (12)
C8—C9—C10—C1	−167.62 (10)	C18—C19—C20—C28	−66.11 (15)
C11—C9—C10—C24	−59.03 (15)	C29—C20—C21—C22	−178.15 (12)
C8—C9—C10—C24	72.05 (14)	C19—C20—C21—C22	−58.00 (14)
C11—C9—C10—C5	177.08 (11)	C28—C20—C21—C22	62.07 (15)
C8—C9—C10—C5	−51.85 (14)	C20—C21—C22—C17	54.74 (15)
C8—C9—C11—C12	29.61 (17)	C27—C17—C22—C21	−162.94 (11)
C10—C9—C11—C12	163.88 (12)	C16—C17—C22—C21	79.71 (14)
C9—C11—C12—C13	1.8 (2)	C18—C17—C22—C21	−45.27 (14)
C11—C12—C13—C18	−179.78 (13)	C16—C17—C27—O3	−134.99 (15)
C11—C12—C13—C14	1.7 (2)	C18—C17—C27—O3	−13.65 (19)
C12—C13—C14—C15	−156.36 (12)	C22—C17—C27—O3	105.72 (16)
C18—C13—C14—C15	25.24 (17)	C16—C17—C27—O4	48.93 (14)
C12—C13—C14—C26	86.91 (15)	C18—C17—C27—O4	170.28 (11)
C18—C13—C14—C26	−91.48 (14)	C22—C17—C27—O4	−70.35 (14)
C12—C13—C14—C8	−34.81 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O1···O2	0.75	2.39	2.6933 (17)	106
O1—H1O1···O1W^{i}	0.75	1.96	2.692 (2)	167
O1W—H1W1···O5	0.87	1.80	2.6389 (19)	163
O4—H1O4···O1^{ii}	0.80	1.84	2.6357 (16)	169
O5—H1O5···O3^{iii}	0.84	1.95	2.7642 (15)	161
O1W—H2W1···O2^{iv}	0.91	2.42	2.8426 (19)	109
C6—H6A···O4^{v}	0.97	2.50	3.3849 (18)	152
C18—H18A···O3	0.98	2.40	2.8006 (18)	104
C26—H26A···O5	0.96	2.45	3.0012 (18)	116

Symmetry codes: (i) −*x*+1/2, −*y*, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2; (iii) *x*−1/2, −*y*+1/2, −*z*+2; (iv) −*x*+1/2, −*y*, *z*+1/2; (v) −*x*+1/2, −*y*+1, *z*−1/2.

Fig. 1

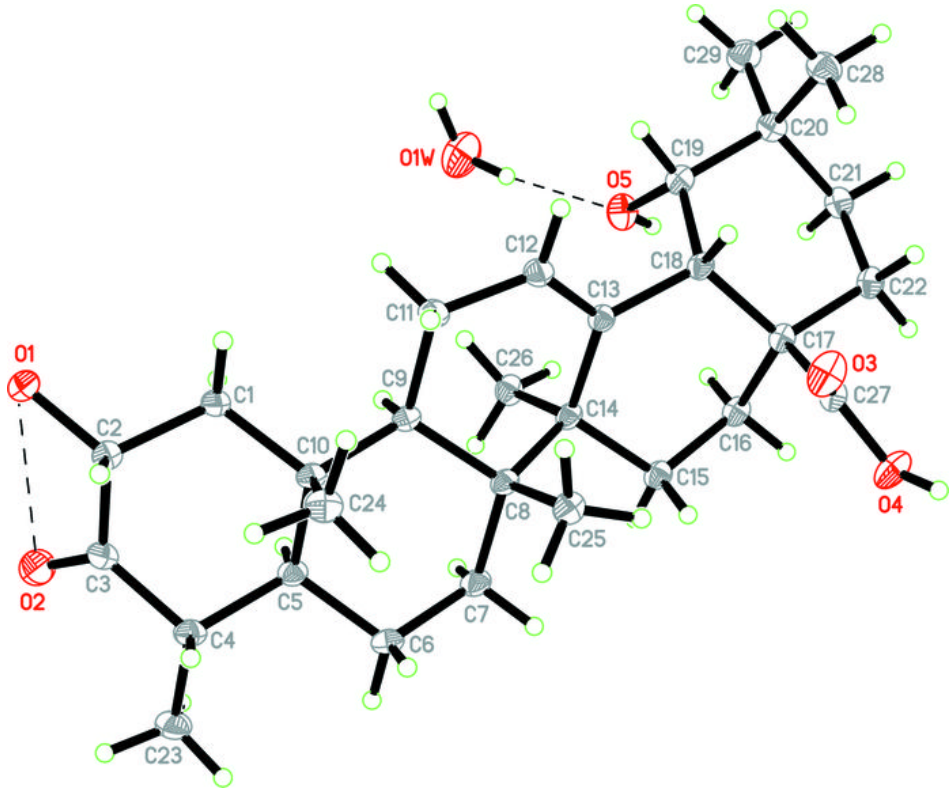

C29
C28
C20
O1W
C19
O5
C21
C12
C18
C22
C13
C11
C17
O3
C26
C9
C27
C14
C1
O1
C2
C10
C16
C15
C24
C8
O4
C25
O2
C3
C5
C7
C4
C6
C23

Fig. 2