Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(6a*R*)-1-Hydroxy-2,2,6a,6b,9,9,12a-heptamethyl-10-(methylcarbonyloxy)perhydro-4-picenylmethyl acetate

Oxana B. Flekhter,[a] Natalya I. Medvedeva[a] and Kyrill Yu. Suponitsky[b]*

[a]Institute of Organic Chemistry, Ufa Research Center of the Russian Academy of Sciences, 71 prosp. Oktyabrya, 450054 Ufa, Russian Federation, and [b]A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, Vavilov St. 28, Moscow 119991, Russian Federation

Correspondence e-mail: kirshik@yahoo.com

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.071
Data-to-parameter ratio = 11.5

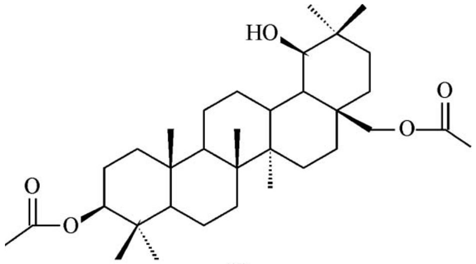
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 March 2007
Accepted 28 March 2007

The title compound, $C_{34}H_{56}O_5$, an oleanane triterpene, is a key intermediate in the synthetic route from betulin to 18(19)-oleanene triterpenoids. In the molecule, all bond lengths and angles are normal. The hydroxy groups are involved in intermolecular O—H···O hydrogen bonds, which link the molecules into zigzag chains running along the *a* axis. The crystal packing is further stabilized by weak intermolecular C—H···O hydrogen bonds.

Comment

Moronic acid derivatives constitute a promising class of new antiviral agents (Kurokawa *et al.*, 1999; Yu *et al.*, 2006; Rios *et al.*, 2001). The seven-step synthesis of moronic acid from the lupane triterpenoid betulin, which is available in high yields from birch bark, was described earlier (Davy *et al.*, 1951; Barton & Brooks, 1951). Here we report the crystal structure of the title compound, (I), which is an important key intermediate in the synthetic route from betulin to 18(19)-oleanene triterpenoids.



(I)

In (I) (Fig. 1), all bond lengths and angles show normal values (Allen *et al.*, 1987; CSD, Version 5.27, Allen, 2002). The cyclohexane rings adopt chair conformations. The acetoxy substituent at C3 is in the equatorial position, while the OH group attached to C19 is in the axial position.

The hydroxy groups are involved in intermolecular O—H···O hydrogen bonds (Table 1), which link the molecules into zigzag chains running along the *a* axis (Fig. 2). The crystal packing is further stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

Experimental

A solution of olean-3,19,28-triol (1 mmol, 0.46 g) [obtained as described by Davy *et al.* (1951)] in dry pyridine (20 ml) and Ac_2O (10 ml) was heated on a steam bath for 3 h. The solution was poured into ice–water (100 ml) and the residue filtered off, washed with water and dried. The crude material was subjected to column chro-

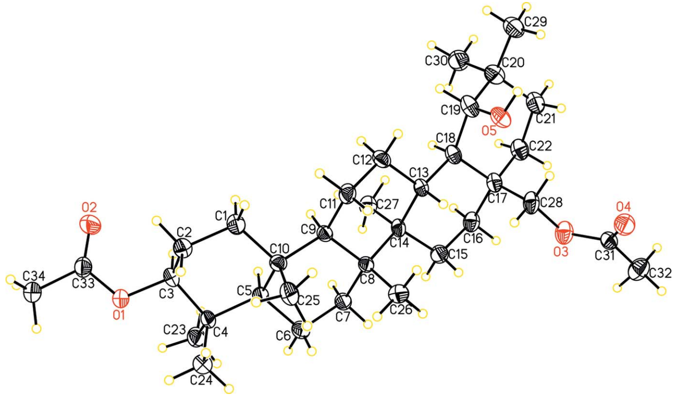


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

matography on silica gel and eluted with $CHCl_3$ to yield 0.43 g (93%) of the pure product, which was crystallized from $CHCl_3$–MeOH, yielding colourless single crystals of (I) suitable for the X-ray analysis after a few days.

Crystal data

$C_{34}H_{56}O_5$	$V = 3052.7$ (4) Å^3
$M_r = 544.79$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 8.4190$ (6) Å	$\mu = 0.08$ mm^{-1}
$b = 11.4960$ (8) Å	$T = 120$ (2) K
$c = 31.541$ (2) Å	0.20 × 0.20 × 0.15 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	4154 independent reflections
Absorption correction: none	2421 reflections with $I > 2\sigma(I)$
25843 measured reflections	$R_{\rm int} = 0.080$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	361 parameters
$wR(F^2) = 0.071$	H-atom parameters constrained
$S = 0.98$	$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
4154 reflections	$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O4$^{\rm i}$	0.85	2.25	3.014 (2)	149
C26—H26B···O2$^{\rm ii}$	0.98	2.41	3.322 (3)	155
C32—H32B···O4$^{\rm iii}$	0.98	2.43	3.323 (3)	152

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

The H atom of the OH group was found in a difference Fourier map and fixed at a distance of 0.85 Å from the O5 atom. The C-bound H atoms were positioned geometrically, with C—H distances of 0.98 Å for the methyl groups and 0.99 Å for the other H atoms. All H atoms were refined in the riding model, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}$(methyl C) or $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C) for all other H atoms. In the absence of any significant anomalous scatterers in the molecule, the 3199 Friedel pairs were merged before the final refinement. The absolute configuration was assigned based on the configuration of the initial reagent used in the synthesis.

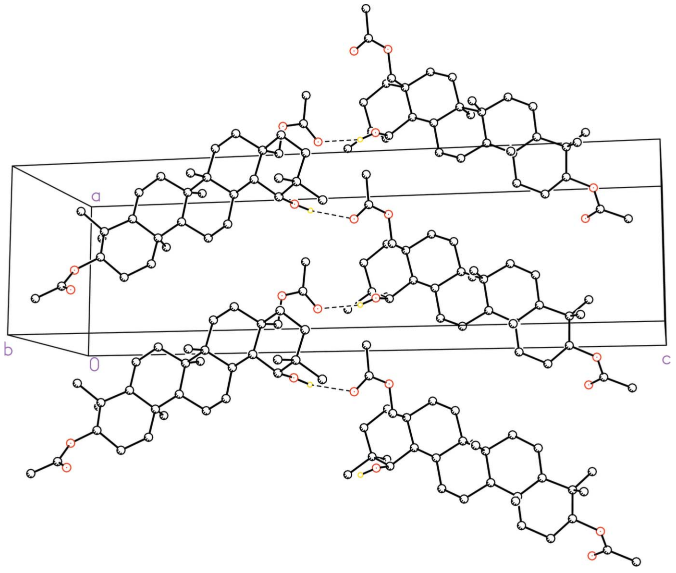


Figure 2
A portion of the crystal packing, showing the zigzag chain of molecules linked by intermolecular O—H···O hydrogen bonds (dashed lines). All H atoms, except those of the hydroxy groups, have been omitted for clarity.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus* (Bruker, 1998); 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*.

This work was supported by the Russian Foundation for Basic Research (grant No. 05–03-82832) and the Federal Program on Support of Leading Scientific Schools (grant No. SS-1153.2006.3).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

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.

Barton, D. H. R. & Brooks, C. J. W. (1951). *J. Chem. Soc.* pp. 257–277.

Bruker (1998). *SAINT-Plus* (Version 6.01) and *SMART* (Version 5.059). Bruker AXS Inc., Madison, Wisconsin, USA.

Davy, G. S., Halsall, T. G. & Jones, E. R. (1951). *J. Chem. Soc.* pp. 2696–2702.

Kurokawa, M., Basnet, P., Ohsugi, M., Hozumi, T., Kadota, S., Namba, T., Kawana, T. & Shiraki, K. (1999). *J. Pharm. Exp. Ther.* **289**, 72–78.

Rios, M. Y., Salina, D. & Villarreal, M. L. (2001). *Planta Med.* **67**, 443–446.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Yu, D., Sakurai, Y., Chen, C.-H., Chang, F.-R., Huang, L., Kashiwada, Y. & Lee, K.-H. (2006). *J. Med. Chem.* **49**, 5462–5469.

o2290 Flekhter *et al.* • $C_{34}H_{56}O_5$ *Acta Cryst.* (2007). E**63**, o2289–o2290