Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.182$

$S = 1.101$

2051 reflections

201 parameters

H atoms: see text

$w = 1/[\sigma^2(F_o^2) + (0.1048P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.158$ e Å^{-3}

$\Delta\rho_{min} = -0.160$ e Å^{-3}

Extinction correction: none

Scattering factors from
International Tables for
Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

N1—C8	1.378 (4)	C5—C20	1.509 (5)
N1—C2	1.392 (3)	C15—C16	1.526 (4)
N1—C15	1.463 (3)	C16—C17	1.456 (4)
C3—C19	1.486 (4)		
C3—C2—C10	130.0 (3)	N18—C17—C16	179.3 (4)
C2—C3—C19	128.4 (3)		

All the non-H atoms were refined using *SHELXL*93 (Sheldrick, 1993) with anisotropic displacement parameters. H atoms were revealed in difference Fourier maps, but only those attached to C13 and C15 were refined, and all others were treated with a riding model.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993). Data reduction: *DATRD2* in *NRCVAX* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Dimethyl oleana-13(18),15(16)-diene-3β,28-diacetate

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Abstract

The title compound, $C_{34}H_{52}O_4$, consists of five six-membered rings. All of the rings except the one with two double bonds exist in chair form. The ring with two double bonds is almost planar with a mean deviation from planarity of only 0.03 Å.

Comment

The title compound, (I), has been isolated from the leaves of the plant *Jacquinia Ruscifolia* which is found in Mexico. The leaves of the plant contains saponins which are generally used as fish poison. The compound has been studied chemically and NMR spectroscopic studies have also been carried out (Mahato & Kundu, 1994). The present X-ray analysis was initiated in order to resolve the details of the molecular structure derived initially from detailed spectral analyses.

(I)
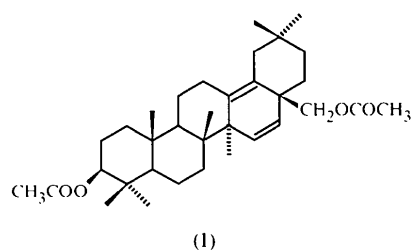
All five rings in the common oleanane-type triterpenes are known to exist mainly in the chair form. In the case of the present compound, rings *A*, *B*, *C* and *E* are in the chair conformation. However, due to the presence of the two double bonds, ring *D* is almost planar. The mean deviation from planarity of ring *D* is 0.0315 Å. The acetate group at C3 is almost planar, with a torsion angle C32—C31—O1—C3 = 178.1 (5)°. The dihedral angle between the mean plane formed by the acetate group and ring *A* is 121.8 (4)° which indicates
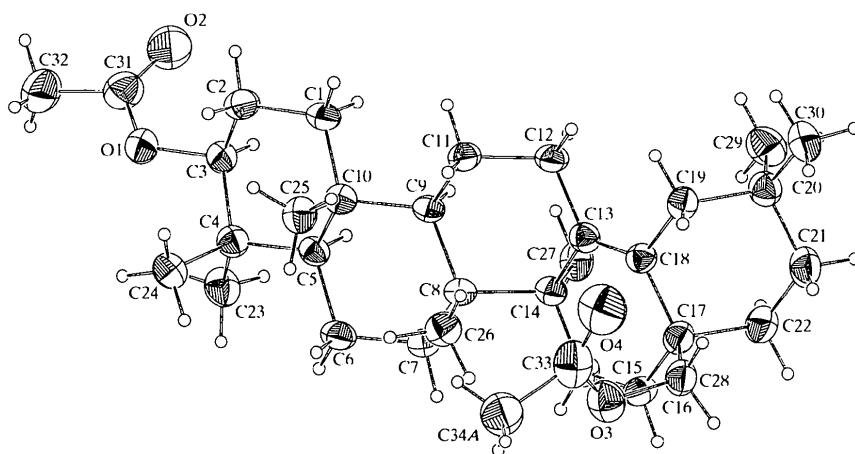
Fig. 1. Structure of the compound showing 30% probability displacement ellipsoids and atom numbering. For clarity, only one of the disorder components of C34 has been shown. H atoms are shown as circles with an arbitrary radius. Note that C27 is part of a methyl group and is not bonded to C18. Similarly, C23 and C24 are bonded only to C4.

that the acetate group is twisted from the plane of ring *A*. The methyl-C atom C34 of the acetate group attached to C28 is disordered and the two alternative positions are designated as C34*A* and C34*B* (see *Experimental*). The acetate group with the C34*A* disorder component is almost planar and it is the C34*A* atom which shows the largest out-of-plane displacement at 0.123 (15) Å. For the C34*B* component, the planarity of the acetate group is more pronounced. This group is twisted from the the plane of the oleanane moiety by 148.9 (4)°. The mean Csp^3—Csp^3 bond length is 1.532 (6) Å with only C8—C14 clearly different with a value of 1.595 (6) Å. This is attributed to the steric effects as these two bonded Csp^3 atoms have no attached H atoms (Cox *et al.*, 1995). The puckering parameters of the six-membered rings *A*, *B*, *C* and *E* have been calculated using the method of Cremer & Pople (1975) and are given in Table 2. There are no strong hydrogen bonds and the structure is stabilized by van der Waals interactions only.

Experimental

Air-dried, crushed leaves of the plant *Jacquinia Ruscifolia* were treated successively with petroleum ether (333–353 K boiling fraction), chloroform and methanol. The residue from the methanol extract was refluxed with 6% methanolic hydrochloric acid for 6 h. The solvent was removed under reduced pressure, water being added during distillation to keep the acid concentration constant. The resulting mixture was extracted with a large volume of diethyl ether, the ethereal solution in turn washed with water, dried and evaporated. The crude material was chromatographed repeatedly over silica gel. Elution with benzene/ether (4:1) afforded a white solid identified as oleana-13(18),15(16)-diene-3β,28-diol. The diol was then acetylated by heating it with pyridine and acetic anhydride over a water bath for 2 h. Work-up resulted in oleana-13(18),15(16)-diene-3β,28-diol diacetate (Dutta, 1972).

Crystals of the title compound were obtained from a methanolic solution by slow evaporation at room temperature.

Crystal data

$C_{34}H_{52}O_4$	Cu *Kα* radiation
$M_r = 524.79$	$\lambda = 1.54178$ Å
Monoclinic	Cell parameters from 24
$P2_1$	reflections
$a = 14.835 (2)$ Å	$\theta = 11.9$–$28.8°$
$b = 7.157 (3)$ Å	$\mu = 0.556$ mm^{-1}
$c = 14.971 (3)$ Å	$T = 293 (2)$ K
$\beta = 103.05 (4)°$	Needle
$V = 1548.5 (7)$ Å^3	$0.5 \times 0.3 \times 0.2$ mm
$Z = 2$	Colourless
$D_x = 1.125$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens AED four-circle diffractometer	1674 reflections with $I > 2\sigma(I)$
ω/θ scans	$R_{int} = 0.022$
Absorption correction:	$\theta_{max} = 70.03°$
ψ scan (North *et al.*, 1968)	$h = -18 \rightarrow 17$
$T_{min} = 0.867$, $T_{max} = 0.895$	$k = -8 \rightarrow 8$
3296 measured reflections	$l = -16 \rightarrow 18$
3189 independent reflections	2 standard reflections
	every 100 reflections
	intensity decay: negligible

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = -0.03$
$R[F^2 > 2\sigma(F^2)] = 0.054$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$wR(F^2) = 0.172$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
$S = 0.822$	Extinction correction: none
3165 reflections	Scattering factors from
346 parameters	*International Tables for*
H atoms: see text	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.076P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Selected geometric parameters (Å, °)*

C3—O1	1.460 (5)	C14—C27	1.556 (6)
C4—C23	1.541 (6)	C15—C16	1.322 (6)
C4—C24	1.563 (6)	C20—C30	1.520 (7)
C8—C26	1.538 (5)	C20—C29	1.529 (6)
C8—C14	1.595 (6)	C31—O2	1.194 (6)
C10—C25	1.544 (6)	C33—O4	1.212 (8)
C13—C18	1.331 (5)		
C2—C3—C4	114.2 (4)	C16—C15—C14	123.6 (5)
C26—C8—C14	106.7 (3)	C15—C16—C17	124.9 (4)
C18—C13—C14	123.7 (4)	C13—C18—C17	123.2 (4)
C27—C14—C8	112.7 (3)		
C1—C2—C3—O1	177.5 (3)	C14—C15—C16—C17	3.6 (7)
O1—C3—C4—C5	−174.5 (3)	C12—C13—C18—C19	−2.5 (6)
C26—C8—C14—C27	175.3 (4)	C32—C31—O1—C3	178.1 (5)

Table 2. *Ring-puckering parameters (Å, °) for four rings*

Ring	q_2	q_3	Q_T	θ
A	0.019 (5)	0.551 (4)	0.551 (4)	2.0 (5)
B	0.089 (4)	0.565 (5)	0.572 (5)	8.9 (4)
C	0.067 (5)	0.572 (4)	0.576 (4)	6.7 (5)
E	0.049 (5)	0.572 (5)	0.574 (5)	4.9 (5)

One of the C atoms (C34) was found to be affected by disorder: this was modelled in terms of two equally occupied sites following refinement of the occupancies to 0.55 (5) and 0.45 (5). The distances between these isotropic components and C33 were restrained to 1.52 (1) Å. All other non-H atoms were refined with anisotropic displacement parameters. All H atoms were included at geometrically calculated positions, except for those of methyl groups attached to sp^2 centres (*i.e.* for C32, C34A and C34B), where the H atoms were located from a circular difference Fourier synthesis. In the refinement, the idealized methyl groups centred on C32, C34A and C34B were allowed to rotate about the local C—C vector, while other H atoms were then allowed to ride on their parent atoms. Hydrogen displacement parameters were also allowed to ride, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H atoms and $x = 1.2$ for all others.

As the absolute structure could not be determined reliably from the Flack parameter [0.2 (5)] and there are no comparable structures where the absolute structure is known, the enantiomer shown cannot be regarded as having been satisfactorily established.

Data collection: local program (Belletti *et al.*, 1993). Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994). Software used to prepare material for publication: *SHELXL*93.

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Uronium sulfate, [OHC(NH₂)₂]₂(SO₄)

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Abstract

The title compound crystallizes in the orthorhombic space group *Pbca*. In the crystal structure, the two H atoms of sulfuric acid protonate the carbonyl groups of two urea molecules. The H—O(urea) distances are 0.83 (3) and 0.87 (2) Å, with O(urea)—O(sulfate) distances of 2.544 (2) and 2.571 (2) Å, respectively.

Comment

The existence of the title compound, uronium sulfate, was deduced, based on a phase-diagram study, about 63 years ago (Dalman, 1934). However, the synthesis of this compound has never been reported; neither crystals nor crystal structure data have been available up to the present. In recent years, we have found that this compound has the important property of activating the phosphorus from phosphatic rock and soil. Uronium sulfate has therefore been applied widely in the manufacture of phosphatic and compound fertilizers (Chen & Xiao, 1989). We report here the synthesis and crystal structure of the title compound, (I). The

(I)