4446 reflections
343 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 1.3800P]$ where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Te1	0.07929 (2)	0.592048 (11)	0.899581 (9)	0.01296 (8)
Br1	0.10054 (3)	0.38493 (2)	0.92671 (2)	0.01919 (9)
S1	0.19666 (7)	0.75449 (5)	0.93479 (4)	0.01799 (15)
S2	0.33051 (7)	0.57034 (5)	0.96452 (4)	0.01944 (15)
S3	−0.08252 (6)	0.72006 (5)	0.83570 (4)	0.01698 (14)
S4	−0.14727 (7)	0.51475 (5)	0.83710 (4)	0.01939 (15)
O1	0.3343 (2)	0.5451 (2)	0.55487 (12)	0.0255 (5)
N1	0.4348 (2)	0.7378 (2)	1.00888 (15)	0.0234 (5)
N2	−0.3118 (2)	0.6481 (2)	0.77751 (15)	0.0210 (5)
C1	0.3334 (3)	0.6917 (2)	0.9738 (2)	0.0179 (5)
C2	0.5496 (3)	0.6837 (2)	1.0442 (2)	0.0256 (6)
C3	0.5234 (3)	0.6495 (2)	1.1297 (2)	0.0275 (6)
C4	0.4412 (4)	0.8424 (2)	1.0148 (2)	0.0347 (8)
C5	0.3908 (5)	0.8795 (3)	1.0937 (3)	0.0472 (9)
C6	−0.1948 (3)	0.6289 (2)	0.8122 (2)	0.0175 (5)
C7	−0.4145 (3)	0.5724 (2)	0.7683 (2)	0.0290 (7)
C8	−0.3981 (4)	0.5128 (3)	0.6925 (2)	0.0373 (8)
C9	−0.3505 (3)	0.7450 (2)	0.7515 (2)	0.0250 (6)
C10	−0.4240 (3)	0.7986 (3)	0.8174 (2)	0.0329 (7)
C11	0.1695 (2)	0.5803 (2)	0.7827 (2)	0.0147 (5)
C12	0.2157 (3)	0.6603 (2)	0.7418 (2)	0.0160 (5)
C13	0.2721 (3)	0.6526 (2)	0.6652 (2)	0.0178 (5)
C14	0.2814 (3)	0.5628 (2)	0.6292 (2)	0.0175 (5)
C15	0.2350 (3)	0.4824 (2)	0.6701 (2)	0.0211 (6)
C16	0.1798 (3)	0.4911 (2)	0.7464 (2)	0.0182 (5)
C17	0.3909 (4)	0.6241 (2)	0.5123 (2)	0.0297 (7)

Table 2. *Selected geometric parameters (Å, °)*

Te1—C11	2.147 (3)	S4—C6	1.715 (3)
Te1—S3	2.6184 (8)	O1—C14	1.366 (3)
Te1—S1	2.6187 (8)	O1—C17	1.433 (4)
Te1—S4	2.6910 (9)	N1—C1	1.323 (4)
Te1—S2	2.7211 (10)	N1—C4	1.470 (4)
Te1—Br1	2.9427 (7)	N1—C2	1.482 (4)
Te1···Br1^i	3.4229 (10)	N2—C6	1.316 (4)
S1—C1	1.735 (3)	N2—C9	1.473 (4)
S2—C1	1.707 (3)	N2—C7	1.480 (4)
S3—C6	1.737 (3)		
C11—Te1—S3	87.67 (7)	Te1—Br1···Te1^i	89.801 (9)
C11—Te1—S1	94.06 (7)	C1—S1—Te1	89.18 (10)
S3—Te1—S1	76.27 (3)	C1—S2—Te1	86.41 (9)
C11—Te1—S4	89.51 (7)	C6—S3—Te1	88.58 (10)
S3—Te1—S4	67.44 (3)	C6—S4—Te1	86.66 (9)
S1—Te1—S4	143.36 (2)	C14—O1—C17	117.5 (2)
C11—Te1—S2	86.75 (7)	C1—N1—C4	123.2 (3)
S3—Te1—S2	142.12 (2)	C1—N1—C2	119.9 (2)
S1—Te1—S2	66.81 (2)	C4—N1—C2	116.8 (2)
S4—Te1—S2	149.83 (2)	C6—N2—C9	122.6 (2)
C11—Te1—Br1	91.73 (7)	C6—N2—C7	120.3 (3)
S3—Te1—Br1	141.09 (2)	C9—N2—C7	117.0 (2)
S1—Te1—Br1	142.45 (2)	N1—C1—S2	122.5 (2)
S4—Te1—Br1	73.65 (2)	N1—C1—S1	120.1 (2)
S2—Te1—Br1	76.55 (2)	S2—C1—S1	117.4 (2)
C11—Te1···Br1^i	173.05 (7)	N2—C6—S4	122.1 (2)
S3—Te1···Br1^i	86.64 (2)	N2—C6—S3	120.6 (2)
S1—Te1···Br1^i	88.46 (2)	S4—C6—S3	117.3 (2)
S4—Te1···Br1^i	84.63 (2)	C16—C11—C12	119.2 (2)
S2—Te1···Br1^i	100.20 (3)	C16—C11—Te1	119.4 (2)
Br1—Te1···Br1^i	90.197 (9)	C12—C11—Te1	121.5 (2)
C11—Te1—Br1···Te1^i	−173.32 (7)	S3—Te1—C11—C16	−126.1 (2)
Te1—S2—C1—S1	−4.08 (14)	S1—Te1—C11—C16	157.8 (2)
Te1—S1—C1—S2	4.24 (15)	S4—Te1—C11—C16	−58.7 (2)
Te1—S4—C6—S3	1.48 (14)	S2—Te1—C11—C16	91.4 (2)
Te1—S3—C6—S4	−1.52 (15)	Br1—Te1—C11—C12	−166.3 (2)

Symmetry code: (i) $-x, 1-y, 2-z$.

Refinement was on F^2 for all reflections except for 10 flagged by us for potential systematic errors.

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

We wish to thank Mr Steffen Kudis, Department of Chemistry, University of Heidelberg, Germany, for assistance during the synthesis of the compound.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1340). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Rhoiptelenyl Acetate, a New Pentacyclic Triterpenoid from *Ficus thunbergii*

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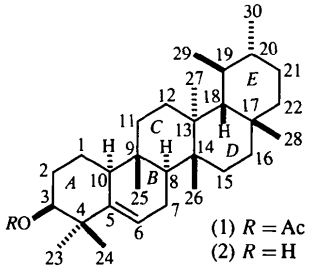
Abstract

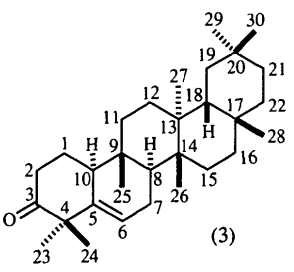
Rhoiptelenol, (2), isolated from *Ficus thunbergii* Maxim. (Moraceae), has been reported as a triterpenoid component. The title compound, (1), whose molecular formula was shown by its high-resolution mass spectrum to be $C_{32}H_{52}O_2$, was obtained from (2) by acetylation with Ac_2O and pyridine. The structure of (1) was

proved by NMR spectroscopy to be one of a rearranged ursane-type triterpenoid with five six-membered rings. We have described the conformation and geometry of (1) by an X-ray crystallographic analysis and molecular-mechanics calculations.

Comment

Ficus thunbergii Maxim. (Moraceae) has been used as a folk medicine against rheumatalgia and arthralgia and as a drug for lower-back pain in China and Japan. Rhoiptelenol, (2), was isolated from the fresh leaves and stems of the plant. The structure of the title compound (1), the acetate of (2), was first elucidated by means of mass spectroscopy and one-dimensional (^{1}H, ^{13}C) and two-dimensional NMR techniques such as heteronuclear multiple-bond correlated (HMBC) spectroscopy and nuclear Overhauser enhancement spectroscopy (NOESY) (Kitajima, Arai & Tanaka, 1994). In this paper, the conformational and geometric studies of (1) are reported. The conformational interdependence of rings *D* and *E* has been characterized by molecular-mechanics force-field calculations (*MM2*; Allinger, 1977) for some canonical forms assumed by the *D*/*E* ring pair.





Rings *A*, *C* and *D* adopt chair forms while ring *B*, owing to a double bond at C5, assumes a slightly distorted half-chair shape as shown by the torsion angles (Table 2). Ring *E* which is *cis*-fused to ring *D* forms a distorted twist boat (Duax, Weeks & Rohrer, 1976).

The results of *MM2* calculations show that the minimum steric energy changes according to the conformation of the *D*/*E* rings as follows: *D* and *E* both chair 87.260; *D* and *E* both boat 90.114; *D* chair, *E* boat 84.187; *D* boat, *E* chair 91.031 kcal mol^{-1}. From this it follows that, in agreement with the X-ray analysis, (1) shows the most stable conformation when *D* is a chair and *E* is a boat. In contrast, glutin-5-en-3-one, (3), which is one of the migrated oleanane-type triterpenoids with a similar skeleton to (1) (five six-membered rings) and has a double bond in the same position as (1), adopts the twist-boat conformation of ring *D* fused to a boat-shaped ring *E* (Ohki, Tachibana, Kuroda, Takenaka & Sasada, 1981). These conformational differences between (1) and (3) can be attributed to the methyl groups (C29 and C30) which are situated at the different (vicinal *versus* geminal) positions of ring *E*.

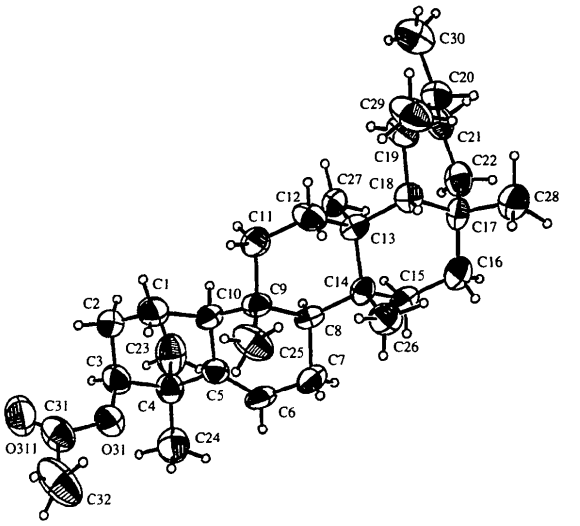


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of (1) with all H atoms. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was obtained from Rhoiptelenol by acetylation with Ac_2O and pyridine.

Crystal data

$C_{32}H_{52}O_2$
M_r = 468.76
Monoclinic
$P2_1$
a = 12.032 (1) Å
b = 7.742 (1) Å
c = 15.148 (1) Å
β = 90.92 (1)°
V = 1410.9 (2) Å^3
Z = 2
D_x = 1.10 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–18°
μ = 0.06 mm^{-1}
T = 296 K
Needle
0.33 × 0.23 × 0.13 mm
Colourless

Data collection

Enraf–Nonius *CAD*-4 *EXPRESS* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical, ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.955, T_{max} = 0.999
3224 measured reflections
3074 independent reflections
1644 observed reflections [$I > 1.0\sigma(I)$]
R_{int} = 0.020
θ_{max} = 26.3°
h = −15 → 0
k = 0 → 9
l = −18 → 18
3 standard reflections
frequency: 120 min
intensity decay: 0.9%

Refinement

Refinement on F
R = 0.056
wR = 0.049
S = 1.38
$(\Delta/\sigma)_{max}$ = 0.02
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.07 e Å^{-3}
Extinction correction: none

1644 reflections
462 parameters
Only H-atom U's refined
$w = 4F_o^2/[\sigma^2(I) + (0.04F_o^2)^2]$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O31	0.8399 (2)	0.6530 (5)	0.5209 (2)	4.70 (8)
O311	0.8422 (3)	0.4282 (6)	0.4285 (2)	6.5 (1)
C1	0.7941 (5)	0.5304 (8)	0.6971 (3)	5.8 (1)
C2	0.7347 (5)	0.4685 (8)	0.6160 (3)	5.8 (1)
C3	0.7279 (4)	0.6088 (7)	0.5475 (3)	4.4 (1)
C4	0.6741 (4)	0.7749 (7)	0.5808 (3)	4.2 (1)
C5	0.7237 (3)	0.8309 (7)	0.6702 (3)	3.5 (1)
C6	0.7398 (4)	0.9945 (6)	0.6908 (3)	3.9 (1)
C7	0.7679 (4)	1.0619 (7)	0.7803 (3)	4.4 (1)
C8	0.7488 (3)	0.9243 (7)	0.8518 (3)	3.06 (9)
C9	0.8063 (4)	0.754	0.8218 (3)	3.4 (1)
C10	0.7440 (4)	0.6884 (7)	0.7391 (3)	4.0 (1)
C11	0.7981 (4)	0.6170 (7)	0.8960 (3)	4.2 (1)
C12	0.8148 (4)	0.6822 (7)	0.9894 (3)	4.2 (1)
C13	0.7390 (3)	0.8333 (6)	1.0126 (3)	3.02 (9)
C14	0.7667 (3)	0.9847 (6)	0.9488 (3)	3.2 (1)
C15	0.6880 (4)	1.1365 (7)	0.9700 (3)	4.6 (1)
C16	0.7023 (4)	1.1970 (7)	1.0655 (3)	5.1 (1)
C17	0.6982 (4)	1.0610 (7)	1.1372 (3)	3.7 (1)
C18	0.7564 (3)	0.8901 (7)	1.1113 (3)	3.5 (1)
C19	0.7371 (4)	0.7390 (8)	1.1783 (3)	4.4 (1)
C20	0.6462 (4)	0.7677 (7)	1.2467 (3)	4.4 (1)
C21	0.5471 (4)	0.8653 (8)	1.2072 (3)	4.6 (1)
C22	0.5741 (4)	1.0323 (7)	1.1632 (3)	4.7 (1)
C23	0.5498 (4)	0.7370 (9)	0.5928 (3)	6.5 (2)
C24	0.6805 (4)	0.9136 (8)	0.5092 (3)	5.7 (1)
C25	0.9292 (4)	0.7851 (9)	0.8019 (3)	5.8 (2)
C26	0.8872 (4)	1.0519 (8)	0.9640 (3)	4.9 (1)
C27	0.6185 (4)	0.7703 (7)	0.9984 (3)	3.8 (1)
C28	0.7590 (4)	1.1392 (8)	1.2200 (3)	5.5 (1)
C29	0.8497 (5)	0.6859 (9)	1.2231 (3)	6.7 (2)
C30	0.6098 (5)	0.601 (1)	1.2867 (3)	7.1 (2)
C31	0.8873 (4)	0.5455 (8)	0.4619 (3)	5.1 (1)
C32	1.0017 (4)	0.617 (1)	0.4432 (3)	8.7 (2)

Table 2. *Geometric parameters (Å, °)*

O31—C3	1.453 (5)	C11—C12	1.513 (6)
O31—C31	1.354 (6)	C12—C13	1.528 (7)
O311—C31	1.168 (7)	C13—C14	1.559 (6)
C1—C2	1.491 (7)	C13—C18	1.570 (6)
C1—C10	1.509 (8)	C13—C27	1.542 (6)
C2—C3	1.504 (7)	C14—C15	1.547 (7)
C3—C4	1.530 (8)	C14—C26	1.554 (6)
C4—C5	1.533 (6)	C15—C16	1.528 (7)
C4—C23	1.537 (7)	C16—C17	1.515 (7)
C4—C24	1.529 (8)	C17—C18	1.551 (7)
C5—C6	1.318 (7)	C17—C22	1.566 (6)
C5—C10	1.536 (7)	C17—C28	1.565 (7)
C6—C7	1.488 (6)	C18—C19	1.569 (7)
C7—C8	1.539 (7)	C19—C20	1.535 (6)
C8—C9	1.564 (5)	C19—C29	1.562 (7)
C8—C14	1.553 (6)	C20—C21	1.525 (7)
C9—C10	1.535 (6)	C20—C30	1.497 (9)
C9—C11	1.547 (6)	C21—C22	1.493 (8)
C9—C25	1.534 (6)	C31—C32	1.516 (8)
C3—O31—C31	116.2 (4)	C12—C13—C27	106.9 (4)
C2—C1—C10	114.8 (5)	C14—C13—C18	110.7 (4)
C1—C2—C3	110.9 (5)	C14—C13—C27	111.2 (3)
O31—C3—C2	108.8 (4)	C18—C13—C27	109.4 (3)
O31—C3—C4	107.1 (4)	C8—C14—C13	109.4 (4)
C2—C3—C4	113.4 (4)	C8—C14—C15	110.4 (3)
C3—C4—C5	111.6 (4)	C8—C14—C26	110.9 (3)
C3—C4—C23	107.3 (4)	C13—C14—C15	107.7 (3)
C3—C4—C24	109.2 (4)	C13—C14—C26	111.6 (3)
C5—C4—C23	108.4 (4)	C15—C14—C26	106.7 (4)
C5—C4—C24	113.9 (4)	C14—C15—C16	111.7 (4)
C23—C4—C24	106.1 (4)	C15—C16—C17	117.5 (4)
C4—C5—C6	122.3 (4)	C16—C17—C18	113.0 (4)
C4—C5—C10	116.9 (4)	C16—C17—C22	108.8 (4)
C6—C5—C10	120.5 (4)	C16—C17—C28	106.6 (4)
C5—C6—C7	125.7 (4)	C18—C17—C22	112.3 (4)
C6—C7—C8	111.4 (4)	C18—C17—C28	109.0 (4)
C7—C8—C9	108.0 (3)	C22—C17—C28	106.8 (4)
C7—C8—C14	115.9 (4)	C13—C18—C17	115.3 (3)
C9—C8—C14	118.3 (3)	C13—C18—C19	112.9 (4)
C8—C9—C10	107.6 (3)	C17—C18—C19	113.5 (3)
C8—C9—C11	109.4 (3)	C18—C19—C20	116.4 (4)
C8—C9—C25	110.8 (3)	C18—C19—C29	109.9 (4)
C10—C9—C11	109.4 (3)	C20—C19—C29	111.6 (4)
C10—C9—C25	110.6 (3)	C19—C20—C21	111.6 (4)
C11—C9—C25	109.0 (4)	C19—C20—C30	111.4 (5)
C1—C10—C5	110.8 (4)	C21—C20—C30	110.8 (4)
C1—C10—C9	114.7 (4)	C20—C21—C22	115.5 (4)
C5—C10—C9	112.9 (4)	C17—C22—C21	116.8 (4)
C9—C11—C12	116.3 (4)	O31—C31—O311	124.5 (4)
C11—C12—C13	113.5 (4)	O31—C31—C32	106.9 (5)
C12—C13—C14	107.3 (3)	O311—C31—C32	128.4 (5)
C12—C13—C18	111.2 (3)		

C10—C1—C2—C3	57.1 (6)	C12—C13—C14—C8	58.4 (4)
C1—C2—C3—C4	−55.6 (6)	C13—C14—C8—C9	−51.9 (4)
C2—C3—C4—C5	48.3 (5)	C14—C8—C9—C11	40.8 (5)
C3—C4—C5—C10	−42.7 (5)	C13—C14—C15—C16	59.7 (5)
C4—C5—C10—C1	43.0 (5)	C14—C15—C16—C17	−50.8 (6)
C5—C10—C1—C2	−49.9 (6)	C15—C16—C17—C18	38.7 (6)
C5—C6—C7—C8	−13.9 (6)	C16—C17—C18—C13	−38.7 (5)
C6—C7—C8—C9	49.2 (4)	C17—C18—C13—C14	51.0 (4)
C7—C8—C9—C10	−66.5 (4)	C18—C13—C14—C15	−60.0 (4)
C8—C9—C10—C5	47.5 (4)	C17—C18—C19—C20	12.6 (5)
C9—C10—C5—C6	−12.5 (6)	C18—C19—C20—C21	37.2 (6)
C10—C5—C6—C7	−5.7 (7)	C19—C20—C21—C22	−53.7 (6)
C8—C9—C11—C12	−40.0 (5)	C20—C21—C22—C17	17.9 (6)
C9—C11—C12—C13	53.7 (5)	C21—C22—C17—C18	32.9 (5)
C11—C12—C13—C14	−61.2 (5)	C22—C17—C18—C19	−47.6 (5)

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIR88* (Burla *et al.*, 1989). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1168). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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