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3β-Taraxerol from *Bridelia micrantha*

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.110
Data-to-parameter ratio = 10.7

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

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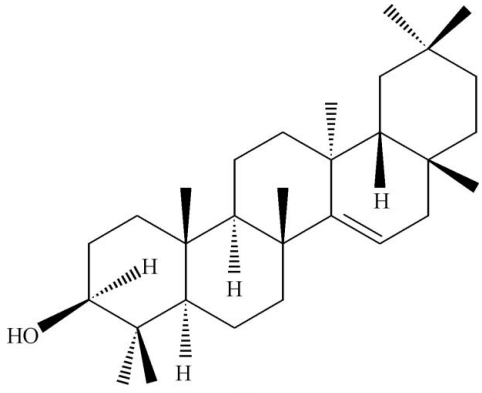
Isolation of the title compound, $C_{30}H_{50}O$, from *Bridelia micrantha* yielded suitable crystals for an X-ray structure determination, showing it to be in the β-form. The crystal packing is determined by infinite zigzag O—H···O hydrogen-bonded chains.

Comment

Eleven chemical constituents were isolated from the chloroform/methanol extract of the stem barks of *Bridelia micrantha*, a plant used in folk medicine in Africa. Although known for long time, the title compound, (I), has only now been characterized crystallographically. The structures of other compounds such as taraxerone [3β-(*trans*-feruloyl)oxy-D-friedoolean-14-ene], careaborin, *n*-tricontyl ferulate, kaempferol, quercetin, betulinic acid, β-sitosterol, 3-*O*-β-sitosterol glucopyranoside and ergosterol were elucidated by ^{1}H and ^{2}D NMR techniques.



(I)

The molecular structure of taraxerol (Fig. 1) is similar to that of taraxerol acetate (Billodeaux *et al.*, 1999), with a β-oriented hydroxyl ligand in (I) instead of the acetate group.

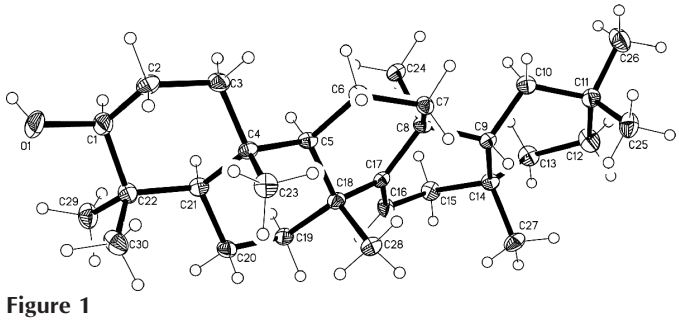


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

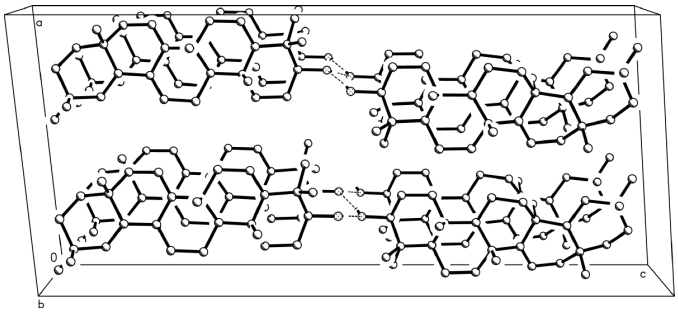


Figure 2
The crystal packing of (I), viewed along [010], with the hydrogen-bonding pattern indicated as dashed lines. H atoms have been omitted.

Other related triterpene skeletons are, for example, 3α-feruloyltaraxerol dichloromethane solvate (Chantrapromma *et al.*, 2003) and taraxerone (Parvez *et al.*, 1999). Geometric bond parameters of these molecules show no significant differences from those of taraxerol. In (I), the C1—O1 bond length is 1.438 (3) Å and the C16═C17 double-bond length is 1.334 (4) Å. The crystal packing shows a strong intermolecular hydrogen-bonding pattern O1—H1···O1($-x+\frac{3}{2}$, $y-\frac{1}{2}$, $-z+1$) with H1···O1 = 2.41 Å and O—H···O = 173°, giving rise to the formation of infinite zigzag chains along [010], with H—O···H angles of about 109°. All these values are normalized for O—H = 0.94 Å.

Experimental

The air-dried barks of *B. micrantha* Baill. (4.5 kg) were macerated in a mixture of CH_2Cl_2–MeOH (1:1) and then pure MeOH. After filtration, the solvent was removed from the extract on a rotary evaporator under reduced pressure. The total extract (180.0 g) was chromatographed on silica gel (230–400 mesh). The column was eluted with the gradient solvent systems: hexane–CH_2Cl_2, CH_2Cl_2–Et_2O, Et_2O–2-propanol, and finally with 100% 2-propanol. The column eluates were monitored by thin-layer chromatography and similar fractions were combined. From that column chromatographic (CC) operation, six subfractions A–F were obtained. Each subfraction was subjected to further CC using gradient solvent hexane/CH_2Cl_2. Subfraction A (2.8 g) yielded taraxerol (15.5 mg). Recrystallization gave colorless prismatic crystals of the title compound.

Crystal data

$C_{30}H_{50}O$
M_r = 426.70
Monoclinic, *C*2
a = 13.4048 (11) Å
b = 6.1275 (4) Å
c = 30.239 (2) Å
β = 94.810 (3)°
V = 2475.0 (3) Å^3
Z = 4
D_x = 1.145 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1966 reflections
θ = 2.7–20.6°
μ = 0.07 mm^{-1}
T = 120 (2) K
Prism, colorless
0.45 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.971, T_{max} = 0.990
15848 measured reflections
3355 independent reflections
2506 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 28.4°
h = −17 → 17
k = −8 → 8
l = −40 → 40

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.110
S = 0.98
3355 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.438 (3)	C15—C16	1.493 (4)
C1—C2	1.514 (4)	C16—C17	1.334 (4)
C1—C22	1.545 (4)	C17—C18	1.538 (4)
C8—C17	1.529 (4)		
O1—C1—C2	110.0 (2)	C16—C17—C8	117.5 (2)
O1—C1—C22	111.4 (2)	C16—C17—C18	122.2 (3)
C2—C1—C22	113.8 (3)	C8—C17—C18	120.2 (2)
C17—C16—C15	121.0 (3)		

H atoms were positioned geometrically (O—H = 0.84, C—H = 0.95–1.00 Å) and refined as riding on their C or O atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl} and OH). All methyl and hydroxy H atoms were allowed to rotate, but not to tip. The title compound crystallizes in the non-centrosymmetric space group *C*2; however, in the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Accordingly, Friedel pairs were merged. The present configuration was chosen to match that of COBKEJ (Billodeaux *et al.*, 1999) and IPUXAS (Chantrapromma *et al.*, 2003).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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