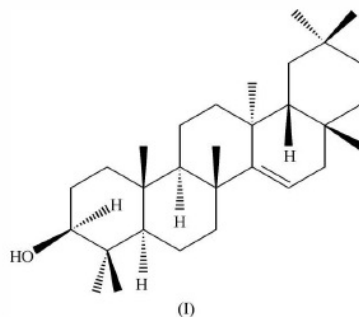
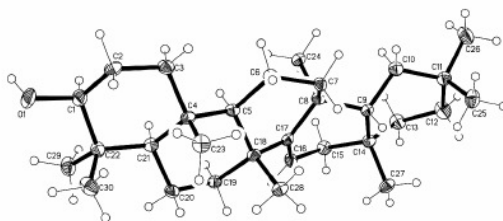
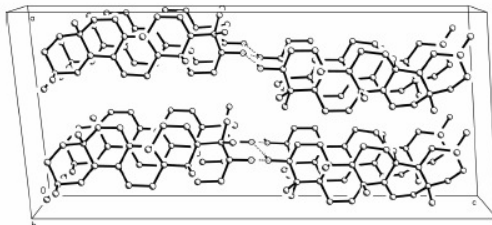


**3 $\beta$ -Taraxerol from *Bridelia micrantha***Simeon F. Kouam,<sup>a</sup> Ulrich Flörke,<sup>b\*</sup> Karsten Krohn,<sup>b</sup> M. Nadeem Akhtar,<sup>b</sup> Bonaventure T. Ngadjui<sup>c</sup> and Berhanu M. Abegaz<sup>d</sup><sup>a</sup>Department of Chemistry, Teachers Training High School, University of Yaounde 1, BP 46, Yaounde, Cameroon, <sup>b</sup>Department Chemie, Fakultät für Naturwissenschaften, Universität Paderborn, Warburgerstraße 100, D-33098 Paderborn, Germany, <sup>c</sup>Department of Organic Chemistry, Faculty of Science, University of Yaounde 1, BP 812, Yaounde, Cameroon, and <sup>d</sup>Department of Organic Chemistry, Faculty of Science, University of Botswana, Private Bag, 0022 Gaborone, Botswana

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Isolation of the title compound, C<sub>30</sub>H<sub>50</sub>O, from *Bridelia micrantha* yielded suitable crystals for an X-ray structure determination, showing it to be in the  $\beta$ -form. The crystal packing is determined by infinite zigzag O—H...O hydrogen-bonded chains.Received 1 February 2005  
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Online 12 February 2005**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 [3 $\beta$ -(*trans*-feruloyl)oxy- $\Delta$ -friedoolean-14-ene], careaborin, *n*-tricontyl ferulate, kaempferol, quercetin, betulinic acid,  $\beta$ -sitosterol, 3-*O*- $\beta$ -sitosterol glucopyranoside and ergosterol were elucidated by <sup>1</sup>H and <sup>2</sup>D NMR techniques.**Key indicators**Single-crystal X-ray study  
T = 120 K  
Mean  $\sigma$ (C—C) = 0.004 Å  
R factor = 0.052  
wR factor = 0.110  
Data-to-parameter ratio = 10.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The molecular structure of taraxerol (Fig. 1) is similar to that of taraxerol acetate (Billodeaux *et al.*, 1999), with a  $\beta$ -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, 3 $\alpha$ -feruloyltaraxerol dichloromethane solvate (Chantrapomma *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 $\cdots$ O1(– $x + \frac{3}{2}$ ,  $y - \frac{1}{2}$ , – $z + 1$ ) with H1 $\cdots$ O1 = 2.41 Å and O–H $\cdots$ O = 173°, giving rise to the formation of infinite zigzag chains along [010], with H–O $\cdots$ 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<sub>2</sub>Cl<sub>2</sub>–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<sub>2</sub>Cl<sub>2</sub>, CH<sub>2</sub>Cl<sub>2</sub>–Et<sub>2</sub>O, Et<sub>2</sub>O–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<sub>2</sub>Cl<sub>2</sub>. Subfraction A (2.8 g) yielded taraxerol (15.5 mg). Recrystallization gave colorless prismatic crystals of the title compound.

### Crystal data

C <sub>30</sub> H <sub>50</sub> O	$D_x = 1.145 \text{ Mg m}^{-3}$
$M_r = 426.70$	Mo $K\alpha$ radiation
Monoclinic, C2	Cell parameters from 1966 reflections
$a = 13.4048 (11) \text{ \AA}$	$\theta = 2.7\text{--}20.6^\circ$
$b = 6.1275 (4) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 30.239 (2) \text{ \AA}$	$T = 120 (2) \text{ K}$
$\beta = 94.810 (3)^\circ$	Prism, colorless
$V = 2475.0 (3) \text{ \AA}^3$	$0.45 \times 0.20 \times 0.15 \text{ mm}$
$Z = 4$	

### Data collection

Bruker SMART CCD area-detector diffractometer	3355 independent reflections
$\varphi$ and $\omega$ scans	2506 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$R_{int} = 0.066$
$T_{min} = 0.971$ , $T_{max} = 0.990$	$\theta_{max} = 28.4^\circ$
15848 measured reflections	$h = -17 \rightarrow 17$
	$k = -8 \rightarrow 8$
	$l = -40 \rightarrow 40$

### Refinement

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

**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  $1.5U_{eq}(C_{methyl} \text{ and } OH)$ . All methyl and hydroxy H atoms were allowed to rotate, but not to tip. The title compound crystallizes in the non-centrosymmetric space group C2; 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 COBKJ (Billodeaux *et al.*, 1999) and IPUXAS (Chantrapomma *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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