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rac-Eudesm-7(11)-en-4-ol

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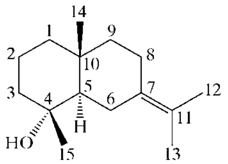
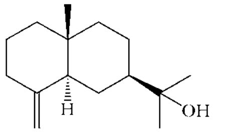
The isolation and structural determination of *rac*-eudesm-7(11)-en-4-ol, $C_{15}H_{26}O$, from the steam distillate of the flowers of *Dipterocarpus cornutus* Dyer (Dipterocarpaceae) is described. The structure was determined from spectroscopic data and a single-crystal X-ray study. Two similar independent molecules comprise the asymmetric unit of the structure.

Comment

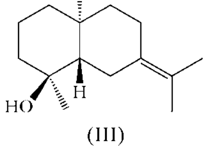
In continuation of our studies of the chemical constituents of the flora of Sumatra (Wahyuni *et al.*, 2004), we have investigated the flowers of *Dipterocarpus cornutus* Dyer (Dipterocarpacaea). This species, known in West Sumatra as kruing, is a resinous tree attaining a height of 20 m and its wood is used in joinery. No chemical work has been performed previously on this species.



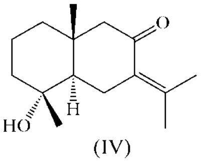
(I)



(II)



(III)



(IV)

Steam distillation of the flowers yielded a crystalline compound (m.p. 421–422 K), which was devoid of optical rotation. The spectroscopic properties of this racemic compound, which are recorded in the *Experimental* section, suggested that it was eudesm-7(11)-en-4-ol. An enantiomer, (I), of this compound was isolated as an oil from *Acritopappus prunifolius* (Bohlmann *et al.*, 1982). Although the authors recorded the ^{1}H NMR spectrum, they omitted to record an optical rotation. These authors were apparently unaware that this eudesmenol had been synthesized previously (Chetty *et al.*, 1968) in three steps from β-eudesmol, (II). More recently (Toyota *et al.*, 1999), this laevorotatory compound has been isolated from the liverwort *Chiloscyphus polyanthos* and its synthesis from (II) was repeated, the absolute stereochemistry being confirmed by X-ray methods on a heavy-atom derivative. However, few spectral data have been recorded.

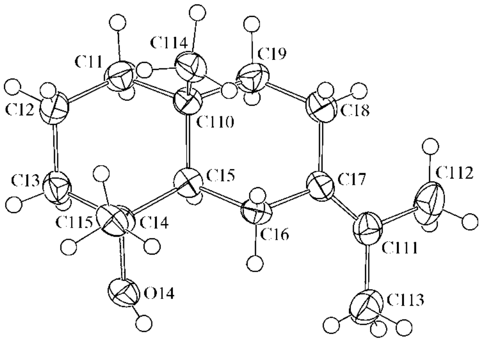


Figure 1
A projection of molecule 1 (molecule 2 is similar) of (I). (The first digit in the atom numbering denotes the number of the molecule.) Displacement ellipsoids at the 50% probability level are shown for C and O atoms. H atoms have arbitrary radii of 0.1 Å.

The dextrorotatory enantiomer, (III) (m.p. 349–359 K), of (I) has been isolated from *Laggera pterodonta* (Zhao *et al.*, 1997). The ^{1}H and ^{13}C NMR spectra recorded for (III) by these workers are similar to those recorded by us, except for one carbon signal.

None of the aforementioned authors made reference to the much earlier studies (Motl *et al.*, 1958) on ‘juniper camphor’ (m.p. 439.5 K), which was isolated from the juniper oil obtained as a by-product during the fermentation of juniper berries in the manufacture of gin. On the basis of degradative evidence, Motl *et al.* (1958) proposed structure (I), without specification of stereochemistry, for this compound, which was devoid of optical rotation. Chetty *et al.* (1968) claimed that the synthetic compound (I) was identical to Motl *et al.*'s juniper camphor in all respects except optical rotation. The evidence adduced for the structure of the new compound suggests that it is the racemate of (I) and (III). Because of the large difference in melting point between the present compound and juniper camphor, we have confirmed the structural assignment by the determination of the crystal structure.

The results of the single-crystal X-ray study are consistent with the stoichiometry and connectivity proposed, the compound being a racemate, with two similar independent molecules comprising the asymmetric unit of the structure. The chirality of the latter is arbitrarily set, in accordance with that assigned to the earlier determination of the structure of cuauhtemone, (IV) (Ivie *et al.*, 1974; Nakanishi *et al.*, 1974), the conformations of all rings being (quasi-)chair (Fig. 1). In the present structure, the molecules pack with weak hydrogen bonding. The geometric parameters are essentially as expected for the structure of (I/III).

Experimental

General directions have been reported previously (Baker *et al.*, 2000). *Dipterocarpus cornutus* Dyer (Dipterocarpaceae) flowers were collected at Rimbo Panti, *ca* 300 m above sea level, 30 km north of

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Lubuk Sikaping, West Sumatra, Indonesia, in April 2001. The herbarium specimen (DR-150) was identified by Dr Rusjdi Tamin and is deposited at the herbarium of Andalas University. Air-dried flowers (250 g) were steam distilled and the oily distillate was crystallized from ethanol, yielding needles (500 mg) of the eudesmenol (m.p. 421–422 K, not raised on further recrystallization). High-resolution EIMS $[M+\mathrm{H}]^{+\cdot}$ found: 222.1985; $^{12}\mathrm{C}_{15}{}^{1}\mathrm{H}_{26}{}^{16}\mathrm{O}$ requires *m*/*z*: 222.1938. IR (cm^{-1}, KBr): 3430, 2997, 2930, 2855, 1457, 1377, 1225, 1172, 1143, 1106, 926, 908; ^{1}H NMR (500 MHz, $\mathrm{CDC1_3}$): δ 0.96 (3H, *s*, 14-H), 1.05 (1H, *m*, 1-H), 1.10 (1H, *dd*, *J* = 14.9 and 5.5 Hz, 9-H), 1.13 (3H, *d*, *J* = 0.8 Hz, 15-H), 1.14 (1*H*, *dd*, *J* = 13.0 and 3.0 Hz, 5-H), 1.31 (1H, *m*, 3-H), 1.39 (1H, *m*, 1-H), 1.42 (1H, *m*, 9-H), 1.55 (2H, *m*, 2-H), 1.64 (1H, *m*, 6-H), 1.66 (3H, *q*, *J* = 1.1 Hz, 12-H or 13-H), 1.69 (3H, *q*, *J* = 1.1 Hz, 12-H or 13-H), 1.80 (1H, *dddd*, *J* = 12.5, 3.3, 3.3 and 1.7 Hz, 3-H), 1.89 (1H, *dd*, *J* = 14.2 and 14.2 Hz, 8-H), 2.49 (1H, *ddddd*, *J* = 14.5, 2.5, 2.5, 2.5 and 2.5 Hz, 8-H), 2.81 (1H, *ddd*, *J* = 13.4, 2.2 and 2.2 Hz, 6-H). ^{13}C NMR (125 MHz, $\mathrm{CDC1_3}$): δ 18.07 (C-14), 20.02 (C-12 or C-13), 20.07 (C-12 or C-13), 20.19 (C-2), 22.04 (C-15), 24.61 (C-6), 25.44 (C-8), 34.81 (C-10), 40.97 (C-1), 43.56 (C-3), 45.23 (C-9), 55.73 (C-5), 72.31 (C-4), 120.96 (C-11), 131.89 (C-7). EIMS: *m*/*z* 222 ($M^{+\cdot}$, 100%), 205 (16), 204 (81), 190 (15), 189 (100), 162 (10), 161 (55), 149 (20), 148 (20), 147 (19), 137 (23), 136 (11), 135 (56), 134 (25), 133 (35), 123 (28), 122 (35), 121 (45), 119 (19), 109 (35), 108 (19), 107 (37), 105 (38).

Crystal data

$\mathrm{C_{15}H_{26}O}$
M_r = 222.37
Triclinic, $P\overline{1}$
a = 9.967 (4) Å
b = 9.973 (3) Å
c = 14.130 (5) Å
α = 96.412 (6)°
β = 110.196 (6)°
γ = 95.100 (6)°
V = 1297.6 (8) Å^3

Z = 4
D_x = 1.138 Mg m^{-3}
Mo Kα radiation
Cell parameters from 1561 reflections
θ = 2.4–26.2°
μ = 0.07 mm^{-1}
T = 150 (2) K
Bar, colourless
0.22 × 0.10 × 0.08 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996
$T_{\min}$ = 0.710, $T_{\max}$ = 0.97
9815 measured reflections

4327 independent reflections
2639 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
$\theta_{\max}$ = 25.0°
$h = -11 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R(F)$ = 0.067
$wR(F^2)$ = 0.168
S = 1.06
4327 reflections
289 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.37F^2]$
$(\Delta/\sigma)_{\max}$ = 0.004
$\Delta\rho_{\max}$ = 0.41 e Å^{-3}
$\Delta\rho_{\min}$ = −0.49 e Å^{-3}

H atoms were located from difference Fourier maps and C-bound H atoms were then placed at idealized positions [C—H = 0.95 Å, and $U_{\mathrm{iso}}(\mathrm{H}) = 1.25U_{\mathrm{eq}}(\mathrm{C})$ (CH and $\mathrm{CH_2}$) and $1.5U_{\mathrm{eq}}(\mathrm{C,O})$ ($\mathrm{CH_3}$ and OH)]. The material was difficult to obtain in a suitably crystalline form and the best available specimen was lost late in the data collection, resulting in 95% completeness after merging equivalents. *SADABS* (Sheldrick, 1996) was also instrumental (note the unreasonable $T_{\min}$ and $T_{\max}$ range) in smoothing irregularities arising from the non-optimal nature of the sample. The subsequent refinement, albeit on weak and limited data, was otherwise smooth and non-idiosyncratic.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *Xtal*3.5 (Hall *et al.*, 1995); program(s) used to refine structure: *CRYLSQ* in *Xtal*3.5; molecular graphics: *Xtal*3.5; software used to prepare material for publication: *BONDLA* and *CIFIO* in *Xtal*3.5.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1451). Services for accessing these data are described at the back of the journal.

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