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Pachypodol (4,5′-dihydroxy-3,3′,7-trimethoxyflavone)

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Key indicators

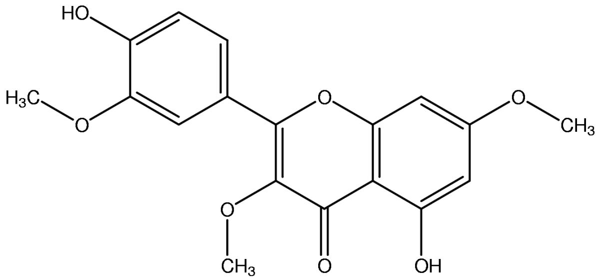
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.155
Data-to-parameter ratio = 12.0

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

One of the products obtained from the fruit of the indigenous Australian tree *Melicope ellyrana* has been confirmed as the flavonoid pachypodol (4,5′-dihydroxy-3,3′,7-trimethoxyflavone), $C_{18}H_{16}O_7$. This compound is similar to a number of the flavonones from the same species with the exception of the reduced side chain. The usual intramolecular hydrogen bonding between the hydroxy and the adjacent carbonyl or methoxy substituent groups is found, together with intermolecular hydrogen bonding between one of these hydroxy groups and a methoxy O atom [O···O 2.875 (4) Å], giving an infinite chain.

Comment

The hexane extract of the fruit of the indigenous Australian tree *Melicope ellyrana* (formerly *Euodia ellyrana* F. Meull) (Hartley, 1981) has yielded, among other products, the flavonoids pachypodol [4′,5-dihydroxy-3,3′,7-trimethoxyflavone, (I)], ternatin [4′,5-dihydroxy-3,3′,7,8-tetramethoxyflavone, (II)], 4′,5-dihydroxy-3,3′,8-trimethoxy-7-(3-methylbut-2-enyloxy)flavone, (III), and 3′,5-dihydroxy-3,4′-dimethoxy-7-(3-methylbut-2-enyloxy)flavone, (IV). Both (III) and (IV) have provided examples of dimorphism [for (III), triclinic and monoclinic (Smith *et al.*, 2001*a*) and (IV), both triclinic representing distinctly different conformational variants (Smith *et al.*, 2001*b*)]. In the case of (III), this was the first report of its isolation from *Melicope* species, although it had previously been isolated from *Boronia coerulescens* (Ashan *et al.*, 1994), while other prenylated flavones similar to (III) and (IV) have been reported, from *M. triphylla* (Higa *et al.*, 1987) and *M. micrococca* (Nasim, 1999). Pachypodol has also been isolated from other plant types, *e.g. Larrea cuneifolia* (Valesi *et al.*, 1972) and *L. tridenta* (Chirikdjian, 1974) as well as *Pachypodanthium confine* (Annonaceae) (Cave *et al.*, 1973), but not previously from *Melicope* species.



(I)

The structure has revealed the basic flavone core similar to other reported flavonoids, *e.g.* 5-hydroxy-3,4′,7-trimethoxyflavone (Gajhede *et al.*, 1989), and to the other flavonone

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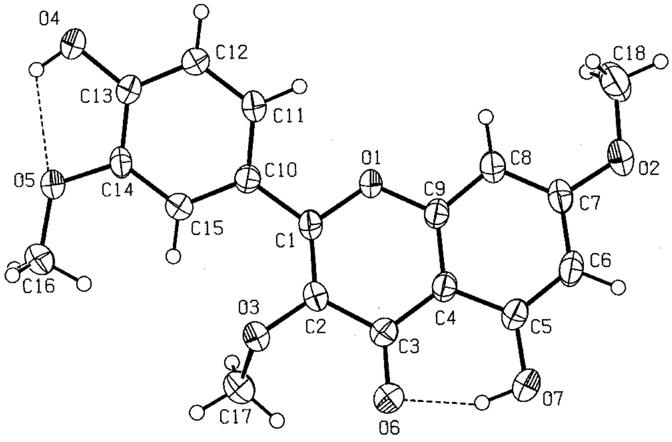

Figure 1
The molecular configuration and atom-naming scheme for (I). Non-H atoms are shown as 40% probability ellipsoids.

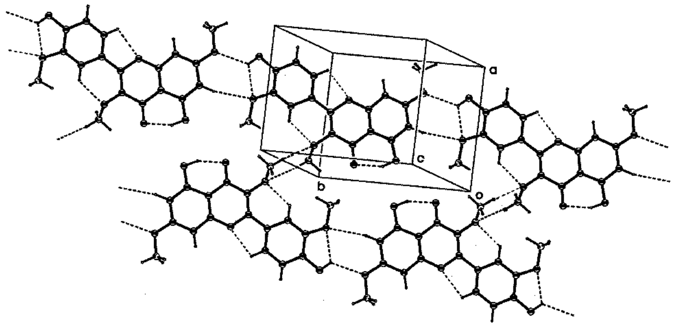

Figure 2
A packing diagram for (I).

components from the same species. The greatest similarity is with the structure of the dimorphic pair (IV) but with the 3-methylbut-2-enyl side chain at C7 reduced. The usual intramolecular C—H⋯O (methoxy) and C—H⋯O (ether) interaction is significant in maintaining near coplanarity in the two ring systems [C11—H3⋯O1 2.664 (4) Å and C15—H5⋯O3 2.898 (4) Å; torsion angle C2—C1—C10—C11 161.3 (4)°]. There is an intramolecular hydrogen-bonding interaction involving the hydroxyl group at C5 and the adjacent ketone oxygen (at C3) [O7—H15⋯O6 2.618 (4) Å] and between the hydroxyl group at C13 and the adjacent methoxy oxygen (at C14) [O4—H1⋯O5 2.688 (4) Å]. A single intermolecular hydrogen-bonding interaction is found between the H atom on this hydroxyl group and a methoxy O atom of an adjacent neighbour [O4—H1⋯O2 2.875 (4) Å; symmetry code: x, $1 + y$, $1 + z$]. This completes a three-centered hydrogen-bonding association about H1 and, together with a weak C6—H2⋯O5 contact [3.494 (5) Å; symmetry code: x, $-1 + y$, $-1 + z$], gives an infinite chain parallel to the b cell direction (Fig. 2). Unusual weak centrosymmetric cyclic hydrogen-bonding intermolecular associations between the oxygen and a methyl H atom of the methoxy substituent group at C2 link the primary chains peripherally [C17—H9⋯O3 3.433 (5) Å; symmetry code: $-x$, $2 - y$, $1 - z$].

Experimental

The title compound was obtained from one of the 23 fractions from the *n*-hexane extract of the fresh fruit of *Melicope ellyrana* (Smith *et al.*, 2001*a*).

Crystal data

$C_{18}H_{16}O_7$
$M_r = 344.31$
Triclinic, $P\overline{1}$
$a = 9.399\ (6)$ Å
$b = 11.836\ (9)$ Å
$c = 8.041\ (3)$ Å
$\alpha = 97.64\ (5)°$
$\beta = 111.08\ (3)°$
$\gamma = 71.83\ (7)°$
$V = 792.8\ (8)$ Å^3
$Z = 2$
$D_x = 1.442$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.0–19.5°$
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Prismatic, yellow
$0.60 \times 0.20 \times 0.20$ mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
2983 measured reflections
2798 independent reflections
1383 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.073$
$\theta_{\rm max} = 25.1°$
$h = 0 \rightarrow 11$
$k = -13 \rightarrow 14$
$l = -9 \rightarrow 8$
3 standard reflections every 150 reflections intensity decay: 0.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.155$
$S = 1.00$
2798 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.0882P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.29$ e Å^{-3}

Table 1
Hydrogen-bonding and short contact geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O4—H1⋯O2$^{\rm i}$	0.81 (4)	2.33 (4)	2.875 (4)	125 (3)
O4—H1⋯O5	0.81 (4)	2.20 (4)	2.688 (4)	120 (3)
O7—H15⋯O6	0.91 (4)	1.76 (3)	2.618 (4)	156 (4)
C6—H2⋯O5$^{\rm ii}$	0.95	2.56	3.494 (5)	167
C11—H3⋯O1	0.95	2.32	2.664 (4)	100
C15—H5⋯O3	0.95	2.30	2.898 (4)	121
C17—H9⋯O3$^{\rm iii}$	0.95	2.53	3.433 (5)	160
C17—H11⋯O6	0.95	2.43	3.020 (5)	120

Symmetry codes: (i) $x, 1 + y, 1 + z$; (ii) $x, -1 + y, -1 + z$; (iii) $-x, 2 - y, 1 - z$.

The H atoms of both hydroxyl groups were located from a difference map and their positional parameters only were refined while the remainder were included at calculated positions and allowed to ride on the attached atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999*a*); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999*b*); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON for Windows* (Spek, 1999); software used to prepare material for publication: *TEXSAN for Windows*.

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