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Methoxyrutaretin, a bioactive furanocoumarin isolated from *Trachyspermum stictocarpum*

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.086
Data-to-parameter ratio = 10.4

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

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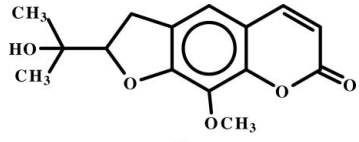
The crystal structure of the title compound, 2,3-dihydro-2-(1-hydroxy-1-methylethyl)-9-methoxy-7*H*-furo[3,2-*g*][1]benzopyran-7-one, also known as methylrutaretin, $C_{15}H_{16}O_5$, is stabilized by intermolecular O—H···O hydrogen-bond interactions, forming zigzag chains parallel to the *c* axis.

Comment

The seed of *Trachyspermum stictocarpum*, popularly known as Ajmoda in Mumbai, India, is widely used as a folklore/herbal medicine in the Indian Ayurved medicinal system (Chopra *et al.*, 1956; Nadkarni, 1954; Gujaral *et al.*, 1953, 1954; Trease, 1949; Maurya *et al.*, 2004). The seeds of this herb are found as a major constituent in many ayurvedic preparations, such as Sahassrayoga (Ravishankar & Sasikala, 1983). Previous phytochemical investigation of *T. stictocarpum* seeds revealed the presence of several coumarins (Bauri & Chattopadhyay, 2005). The title compound, (I), has previously been isolated from different natural sources (Murray, 1978; Purushothaman *et al.*, 1986) and its structure was elucidated by spectroscopic methods. Detailed pharmacological studies of the compound have been described in the literature (Schneider *et al.*, 1967). We present here the crystal structure determination of the compound.



(I)

In the molecule of (I), the furobenzopyranone ring system is approximately planar (Fig. 1), the largest deviation from the least-squares plane being 0.146 (2) Å for atom C11. The furan ring adopts a twist conformation, with $q_2 = 0.091$ (2) Å and $\varphi = 94.3$ (9)° (Cremer & Pople, 1975).

The crystal packing is stabilized by intermolecular O—H···O hydrogen-bond interactions (Table 1), forming zigzag chains of molecules running parallel to the *c* axis (Fig. 2).

Experimental

The parent compound was isolated as a minor product from a methanol extract of *T. stictocarpum* by column chromatography over silica gel with gradient elution, using methanol in chloroform as the solvent system, followed by purification by preparative thin-layer chromatography (PTLC). Attempts to crystallize the parent compound from different solvent systems were unsuccessful. Thus, in order to overcome this difficulty, the compound was methylated by

reaction with diazomethane in diethyl ether (1.2 equiv.) at 273–278 K to obtain the title monomethoxy derivative. The solvent and excess reagent were removed by immersion in a stream of nitrogen, yielding a pale-yellow solid, which was purified by PTLC (yield 90%). Crystals of (I) suitable for X-ray diffraction analysis were obtained by slow evaporation of a hexane–ethyl acetate (4:1) solution at room temperature.

Crystal data

$C_{15}H_{16}O_5$
M_r = 276.28
Orthorhombic, $P2_12_12_1$
a = 7.9166 (9) Å
b = 9.741 (1) Å
c = 17.435 (3) Å
V = 1344.5 (3) Å^3
Z = 4
D_x = 1.365 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 0.86 mm^{-1}
T = 299 (2) K
Block, colourless
0.55 × 0.40 × 0.35 mm

Data collection

Enraf–Nonius CAD4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3131 measured reflections
2397 independent reflections
2335 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 66.9°
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.086
S = 1.05
2397 reflections
230 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.040
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0165 (10)
Absolute structure: Flack (1983), with 994 Friedel pairs
Flack parameter: −0.01 (18)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5*O*···O3^{i}	0.86 (3)	2.12 (3)	2.9537 (19)	164 (2)

Symmetry code: (i) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$.

The H atoms were located in a difference map [refined distances: O—H = 0.86 (3), C—H = 0.90–1.05 (2) Å] and were refined with U_{iso}(H) = 1.2 U_{eq}(C,O).

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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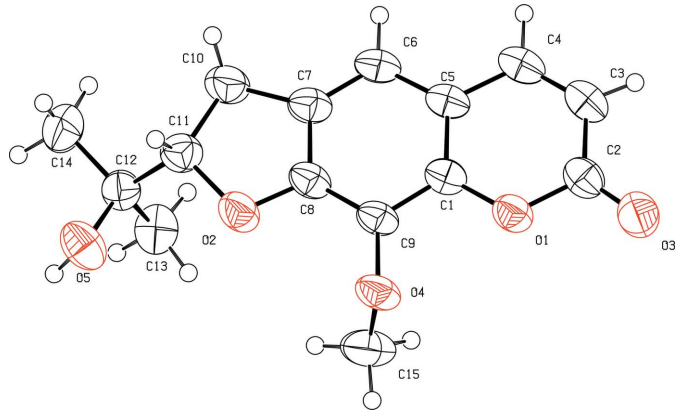

Figure 1
the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

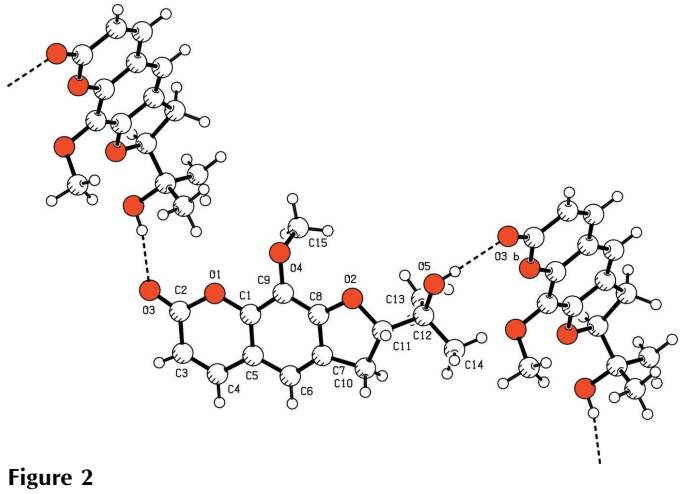

Figure 2
Part of the molecular packing of (I), with hydrogen bonds shown as dashed lines. The O atom labelled with the suffix b is generated by the symmetry operator $(\frac{1}{2}-x, 1-y, -\frac{1}{2}+z)$.

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