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Conformations of imperatorin

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.142
Data-to-parameter ratio = 21.8

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

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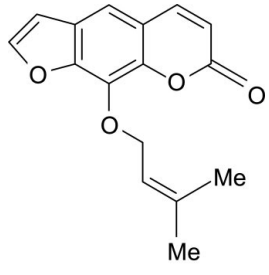
The crystal structure of a pure sample of 9-(3-methylbut-2-enyloxy)-7*H*-furo[3,2-*g*]chromen-7-one or imperatorin, $C_{16}H_{14}O_4$, is composed of two independent molecules with different conformations of the 3-methylbutyl group. The structure of an impure sample of imperatorin has already been reported [Cox, Jaspars, Kumarasamy, Nahar, Sarker & Shoeb (2003). *Acta Cryst.* C**59**, o520–o522.]

Comment

Aegle marmelos Correa (Rutaceae family) is the only species of the genus *Aegle* that is found in Pakistan (Hassanuddin & Ghazanfar, 1980). It is one of the most important medicinal plants of India (Srivastava *et al.*, 1996). Its roots and aerial parts are used in Ayurvedic and Unani systems of medicine for the treatment of various ailments. The alcoholic extracts of its roots possess hypoglycemic (Ohashi *et al.*, 1995), the ripe fruit extracts show antiviral (Mazumdar, 1975), and seed oil shows antibacterial (Banerji & Kumar, 1980) activities. A survey of the chemical literature of *A. marmelos* revealed that its various parts such as roots, bark, leaves, heartwood and fruits have been investigated by several groups and a number of coumarins (Ohashi *et al.*, 1995), alkaloids (Govindachari & Premila, 1983), lignan-glucosides, triterpenoids (Chatterjee & Chaudhary, 1960), sterols (Chatterjee & Bose, 1952), carbohydrates (Parikh *et al.*, 1958), and anthraquinones (Srivastava *et al.*, 1996) have been obtained from this plant. Several bioactive constituents have also been reported from *A. marmelos* (Chatterjee & Bose, 1952). In this paper, we report the isolation and crystal structure of imperatorin, (I). The structure of an impure sample of imperatorin containing phellopterin as an impurity has already been reported (Cox *et al.*, 2003).



(I)

The asymmetric unit of (I) is composed of two independent molecules (Figs. 1 and 2) with different conformations of the 3-methylbutyl moiety. The structure is identical to that reported earlier containing a mixture of (I) and its 4-methoxy derivative phellopterin (Cox *et al.*, 2003). However, the structure

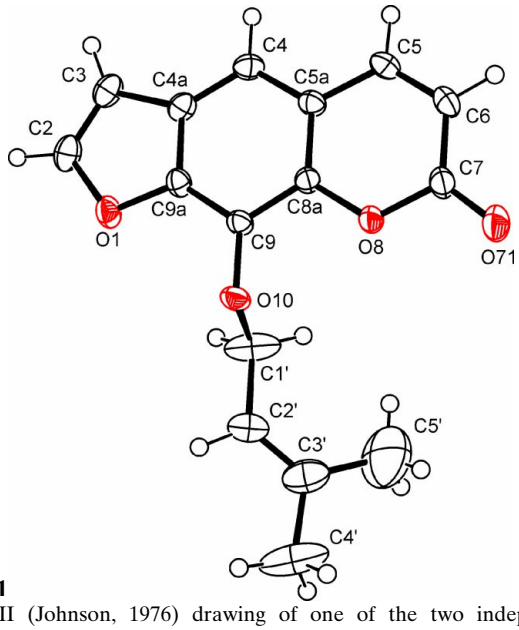


Figure 1
*ORTEP*II (Johnson, 1976) drawing of one of the two independent molecules of (I), with displacement ellipsoids drawn at the 50% probability level. Only three of the six H atoms with occupancies of 0.5 have been included for each methyl group.

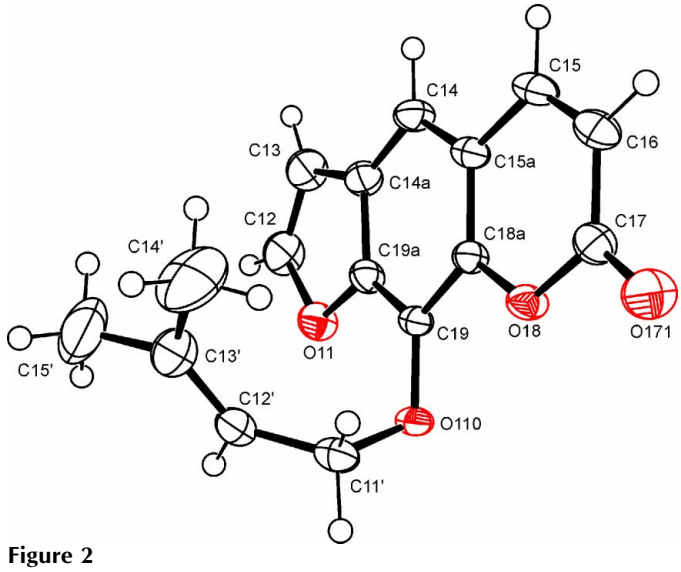


Figure 2
*ORTEP*II (Johnson, 1976) drawing of the second independent molecule of (I), with displacement ellipsoids drawn at the 50% probability level. Only three of the six H atoms with occupancies of 0.5 have been included for each methyl group.

presented here has been obtained from a pure sample containing no impurity and shows marked improvement in the precision of the results.

The molecular dimensions in the two molecules are normal and agree with expected values. Details of intermolecular interactions are similar to those already reported (Cox *et al.*, 2003).

Experimental

Air-dried fruits of *Aegle marmelos Correa* (10.0 kg) were chopped into small pieces and extracted with MeOH three times. The combined extract was evaporated under reduced pressure to obtain a dark-brown residue (500 g). The residue was suspended in distilled water and extracted with petroleum ether, chloroform and ethyl acetate. Each extract was concentrated in vacuo to obtain petroleum ether-, chloroform- and ethyl acetate-soluble fractions, respectively. The petroleum ether extract (32.6 g) was subjected to column chromatography on silica gel using petroleum ether, petroleum ether–ethyl acetate, ethyl acetate, ethyl acetate–methanol and finally, pure methanol as the mobile phase. Fractions obtained with 40% ethyl acetate in petroleum ether were pooled to obtain a strong UV-active semi-pure fraction, which was subjected to repeated column chromatography. On further elution of the column with petroleum ether–ethyl acetate (9:1), compound (I) (12.07 mg) was obtained, and it was recrystallized from petroleum ether–chloroform (8:2).

Crystal data

$C_{16}H_{14}O_4$
M_r = 270.27
Triclinic, $P\overline{1}$
a = 11.041 (6) Å
b = 11.729 (6) Å
c = 11.800 (7) Å
α = 64.72 (4)°
β = 89.47 (3)°
γ = 84.17 (2)°
V = 1373.6 (13) Å^3
Z = 4
D_x = 1.307 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 651 reflections
θ = 2.9–30.0°
μ = 0.09 mm^{-1}
T = 173 (2) K
Block, colorless
0.35 × 0.32 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: none
14 651 measured reflections
7853 independent reflections
6297 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 30.0°
$h = -15 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.00
7853 reflections
361 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2 + 0.4P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9a	1.3662 (14)	O11—C19a	1.3648 (15)
O1—C2	1.3897 (16)	O11—C12	1.3894 (17)
C7—O71	1.2121 (16)	C17—O171	1.2093 (17)
C7—O8	1.3782 (14)	C17—O18	1.3838 (15)
O8—C8a	1.3772 (14)	O18—C18a	1.3766 (15)
C8a—C9	1.3931 (15)	C19—O110	1.3725 (15)
C9—O10	1.3675 (14)	O110—C11′	1.4657 (17)
C9a—O1—C2	105.27 (10)	C19a—O11—C12	105.30 (10)
C8a—O8—C7	121.84 (9)	C18a—O18—C17	121.84 (10)
C9—O10—C1′	112.25 (10)	C19—O110—C11′	111.54 (9)

H atoms were included in the refinement at idealized positions, with C—H = 0.95–0.99 Å and U_{iso} = 1.5 (methyl) and 1.2 (the rest) times U_{eq} of the atoms to which they were bonded. In both idependent molecules, each methyl H atom is disordered over two positions, each of occupancy 0.5. The final difference map was free of any chemically significant features.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure:

*SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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