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Futoenone, a neolignan from *Magnolia soulangiana*

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Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.085
Data-to-parameter ratio = 7.6

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

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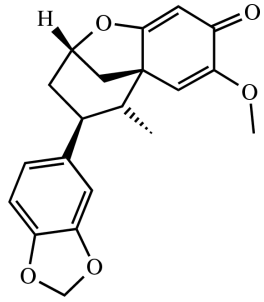
In the title compound, (2*R*,4*R*,5*S*,5a*R*)-*rel*-(−)-4-(1,3-benzodioxol-5-yl)-2,3,4,5-tetrahydro-7-methoxy-5-methyl-8*H*-2,5a-methano-benzoepin-8-one, $C_{20}H_{20}O_5$, the tetrahydrofuran ring has an envelope conformation, and the semiquinone ring is somewhat non-planar, with deviations from coplanarity of up to 0.1603 (13) Å.

Comment

The title neolignan, (I), has been previously reported from *Piper futokadsura* (Ogiso *et al.*, 1968), *Magnolia denudata* (Iida *et al.*, 1982), *Magnolia liliflora* (Iida & Ito, 1983; Talapatra *et al.*, 1982) and other plants (Shizuri & Yamamura, 1983). The crystal structure of an orthorhombic ($P2_12_12_1$) polymorph has been previously reported (Roychowdhury & Ghosh, 1984), but its coordinates are not available. The conformation of the molecule in the orthorhombic polymorph is reported by Roychowdhury & Gosh to be somewhat different from what we find, even in the portion of the molecule with three fused rings, which might be expected to be rather rigid. They report the tetrahydrofuran ring to be a half chair, while in the present trigonal polymorph, it is clearly envelope, with C7′ lying 0.689 (3) Å out of the best plane of the other four atoms. Further, they report that all atoms of the semiquinone ring 'except (the spiro C atom) lie on a plane since the torsion angles associated with this group are within 6° of zero'. We find larger deviations, with torsion angles about C3′—C4′ and C4′—C5′ differing from zero by 12.7 (3) and 19.8 (3)°, respectively. In the present structure, the six atoms of the semiquinone ring deviate from coplanarity by distances ranging from −0.0310 (14) Å for C3′ to 0.1603 (13) Å for C1′. We find the C8—C1′ bond to be somewhat elongated at 1.583 (2) Å, which was also noted by Roychowdhury & Gosh (1.589 Å).



(I)

In the title structure, a short intermolecular C4′...O2(y −x, 1−x, z−1/3) contact exists with a distance of 2.968 (3) Å.

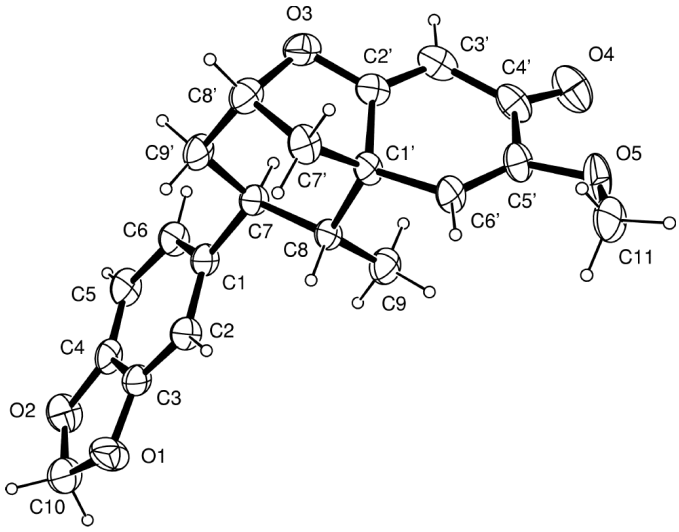


Figure 1
The atom-numbering scheme for (I) with ellipsoids at the 40% probability level.

Experimental

Leaves of *Magnolia soulangiana*, collected in Vancouver, BC, Canada, were air-dried, ground, and extracted with CH_2Cl_2 at room temperature for 24 h. The crude extract was separated by standard vacuum liquid chromatography procedures (Cantrell *et al.*, 1996), using silica gel and *n*-hexane/ethyl acetate mixtures of increasing polarity. Fractions 58–61 (of 66) yielded crystals of futoenone.

Crystal data

$C_{20}H_{20}O_5$
M_r = 340.36
Trigonal, $P3_1$
a = 7.5210 (6) Å
c = 25.2821 (13) Å
V = 1238.50 (15) Å^3
Z = 3
D_x = 1.369 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 23.5–42.6°
μ = 0.81 mm^{-1}
T = 297 K
Trigonal prism, colorless
0.45 × 0.34 × 0.34 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.614, T_{max} = 0.724
4948 measured reflections
1748 independent reflections
1727 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 74.9°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 31$
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.085
S = 1.10
1748 reflections
229 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.0948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0076 (11)

Table 1
Selected geometric parameters (Å, °).

O1—C10	1.435 (3)	O3—C8′	1.473 (3)
O2—C10	1.416 (4)	O4—C4′	1.224 (3)
O3—C2′	1.351 (3)	C8—C1′	1.583 (2)
C3—O1—C10	105.20 (19)	C2′—O3—C8′	107.78 (15)
C4—O2—C10	105.84 (19)		
C2—C1—C7—C8	44.3 (2)	C3′—C4′—C5′—C6′	19.8 (3)
C8′—O3—C2′—C1′	3.3 (2)	C4′—C5′—C6′—C1′	−3.1 (3)
C6′—C1′—C2′—C3′	26.0 (3)	C2′—C1′—C6′—C5′	−18.2 (3)
C7′—C1′—C2′—O3	−29.55 (19)	C2′—C1′—C7′—C8′	42.06 (18)
C1′—C2′—C3′—C4′	−10.4 (3)	C2′—O3—C8′—C7′	25.1 (2)
C2′—C3′—C4′—C5′	−12.7 (3)	C1′—C7′—C8′—O3	−41.81 (18)

The absolute configuration could not be determined. H atoms were placed in calculated positions with C—H distances in the range 0.93–0.98 Å and thereafter treated as riding. A torsional parameter was refined for each methyl group; $U_{iso} = 1.2U_{eq}$ of the attached atom (1.5 for methyl groups).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *MAXUS* (Mackay *et al.*, 1999); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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