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Denudatin *A*, a neolignan from *Magnolia soulangiana*

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In the title compound, (2*S*,3*R*,3a*R*)-2-(1,3-benzodioxol-5-yl)-3,3a-dihydro-3a-methoxy-3-methyl-5-(2-propenyl)-6(2*H*)-benzofuranone, $C_{20}H_{20}O_5$, the furan ring has a half-chair conformation with its O atom on the twist axis. The semiquinone ring is slightly non-planar, with a maximum deviation of 0.065 (3) Å.

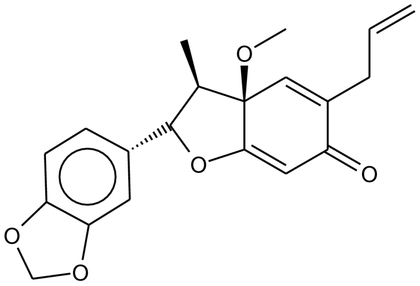
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.006 Å
R factor = 0.061
wR factor = 0.166
Data-to-parameter ratio = 11.5

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

Comment

The title neolignan, (I), has previously been reported from *Magnolia denudata* (Iida *et al.*, 1982; Kuroyanagi *et al.*, 2000), *Magnolia liliflora* (Iida & Ito, 1983) and *Magnolia soulangiana* (Abdallah, 1993).



(I)

The furan ring has a half-chair conformation with its O atom on the twist axis, as shown by the torsion angles in Table 1. The semiquinone ring is only slightly non-planar, with its six C atoms exhibiting an r.m.s. deviation of 0.039 Å from coplanarity, with a maximum deviation of 0.065 (3) Å. The propenyl group is twisted out of the semiquinone-ring plane primarily by rotation about the C7′—C8′ bond, as indicated by the C1′—C7′—C8′—C9′ torsion angle of −125.4 (4)°.

The most closely related neolignan for which the crystal structure has been previously reported is mirandin-*A* (Tomita, *et al.*, 1977), which differs from the title compound, (I), in having the opposite configuration at C5′ and by having a 3,4,5-trimethoxyphenyl substituent rather than the 3,4-methylenedioxyphenyl group of denudatin *A*. In mirandin-*A*, the furan ring also has a twist conformation, but with C7 on the twist axis. Its propenyl group also has a different conformation, with a C6′—C1′—C7′—C8′ torsion angle of −92.7° and a C1′—C7′—C8′—C9′ torsion angle of 2.0°.

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),

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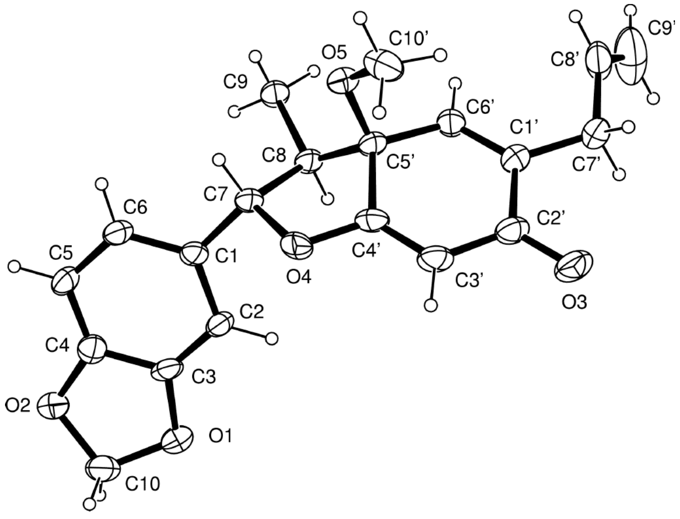

Figure 1

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

using silica gel and *n*-hexane/ethyl acetate mixtures of increasing polarity. Fractions 63–66 (of 66) yielded crystals of denudatin *A*.

Crystal data

$C_{20}H_{20}O_5$
$M_r = 340.36$
Monoclinic, $C2$
$a = 17.8678$ (17) Å
$b = 6.7905$ (18) Å
$c = 16.382$ (2) Å
$\beta = 120.114$ (10)°
$V = 1719.4$ (5) Å^3
$Z = 4$
$D_x = 1.315$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.2$–$23.4°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Needle, colorless
0.45 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3590 measured reflections
2619 independent reflections
1813 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\rm max} = 30.0°$
$h = -24 \rightarrow 21$
$k = 0 \rightarrow 9$
$l = -19 \rightarrow 21$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.166$
$S = 1.04$
2619 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0922P)^2 + 0.03P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.42$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

O3—C2′	1.233 (5)	C3′—C4′	1.322 (5)
C1′—C6′	1.333 (5)	C8′—C9′	1.312 (7)
C4′—O4—C7	108.7 (3)	C9′—C8′—C7′	123.7 (5)
C4′—O4—C7—C8	14.1 (4)	O4—C4′—C5′—C8	−36.9 (4)
C2—C1—C7—O4	−45.0 (4)	C7—C8—C5′—C4′	42.3 (3)
O4—C7—C8—C5′	−35.7 (3)	C6′—C1′—C7′—C8′	4.3 (6)
C7—O4—C4′—C5′	14.5 (4)	C1′—C7′—C8′—C9′	−125.4 (4)
C10′—O5—C5′—C4′	57.6 (4)		

The absolute configuration could not be determined. H atoms were placed in calculated positions with C—H bond distances in the range 0.95–1.00 Å and thereafter treated as riding. A torsional parameter was refined for each methyl group. $U_{\rm iso} = 1.2U_{\rm eq}$ 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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