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Cubebin, a lignan isolated from *Aristolochia odoratissima* L.

Julia Bruno-Colmenárez,[a] Alfredo Usubillaga,[b] Nancy Khouri[b] and Graciela Díaz de Delgado[a]*

[a]Laboratorio de Cristalografía, LNDRX, Facultad de Ciencias, Universidad de Los Andes, Mérida 5101, Venezuela, and [b]Grupo de Productos Naturales y Química Medicinal, Facultad de Farmacia, Universidad de Los Andes, Mérida 5101, Venezuela

Correspondence e-mail: diaz@ula.ve

Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.006 Å
R factor = 0.053
wR factor = 0.121
Data-to-parameter ratio = 9.4

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

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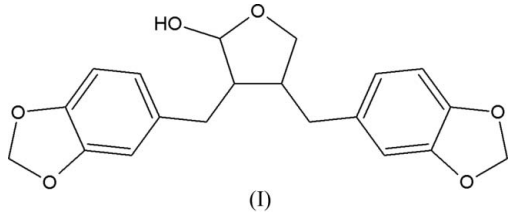
The structure of cubebin [systematic name: 3,4-bis(1,3-benzodioxol-5-ylmethyl)tetrahydrofuran-2-ol], $C_{20}H_{20}O_6$, is stabilized by O—H···O and C—H···O hydrogen bonds.

Comment

Cubebin is a dibenzylbutyrolactone lignan, commonly found in coniferae and in the wood, roots and resin of many plants. It has been isolated from hinokiol (Ishiguro, 1936) and obtained by synthesis (Battersbee *et al.* 1969). Cubebin and its derivatives show a range of biological activities; recent studies report their analgesic, anti-inflammatory and trypanocidal activity (Bastos *et al.*, 2001; Souza *et al.*, 2004; de Souza *et al.*, 2005; da Silva *et al.*, 2005). Our material was isolated from *Aristolochia odoratissima L.* (Usubillaga *et al.*, 2005), which grows in the humid lowlands of the Maracaibo Lake (Venezuela). This plant contains aristolochic acid and is used as an anti-ophidian remedy.



(I)

Fig. 1 shows the molecular structure of the title compound, (I), with the atom- and ring-labelling scheme. The five-membered ring *C* adopts an envelope conformation, with C2 as the flap atom. The substituents at C2 and C3 on ring *C* are *trans* to each other.

A search of the Cambridge Structural Database (Version 5.28; Allen, 2002) resulted in only one similar structure, a lignan with the same molecular formula isolated from *Daphne tangutica* Maxium, called (−)-dihydrosesamin, (II) (Lin-Gen *et al.*, 1983).

Experimental

Cubebin was extracted from the ground roots of *Aristolochia odoratissima* L. by treatment with *n*-pentane. Upon concentration of the pentane extract to half its volume, a precipitate was obtained which was filtered and crystallized from $CHCl_3$–hexane (1:1). Recrystallization from pentane yielded colourless needles (140 mg; m.p. 403–406 K). Mass spectrum: M^+ 356 ($C_{20}H_{20}O_6$, 32%), 338 ($M^+ - H_2O$, 12%), 203 (15%), 135 (100%). ^{1}H NMR (400 MHz, $CDCl_3$, δ, p.p.m.): 6.68 (*d*, 2H, H11 and H19), 6.62 (*m*, 2H, H12 and H20), 6.47 (*s*, 2H, H7 and H15), 5.88 (*s*, 4H, $OCH_2O \times 2$), 5.12 (*d*, 1H, H1), 3.90 (*t*, 1H, H4*A*), 3.72 (*t*, 1H, H4*B*), 2.64 (*m*, 4H, H13*A*,*B* and H5*A*,*B*), 2.09 (*m*, 1H, H3) 2.06 (*m*, 1H, H2).

Crystal data

$C_{20}H_{20}O_6$
M_r = 356.36
Monoclinic, $P2_1$
a = 11.631 (2) Å
b = 5.5969 (10) Å
c = 13.577 (2) Å
β = 99.548 (3)°
V = 871.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 291 K
0.3 × 0.2 × 0.1 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.944, T_{max} = 0.989
5459 measured reflections
2212 independent reflections
962 reflections with $I > 2\sigma(I)$
R_{int} = 0.070

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.121
S = 0.99
2212 reflections
235 parameters
1 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1^{i}	0.82	1.95	2.744 (4)	164
C9—H9A···O4^{ii}	0.97	2.59	3.182 (6)	120
C13—H13A···O2	0.97	2.51	2.838 (5)	100

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

H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C,O). In the absence of appreciable anomalous scattering, Friedel equivalents were merged before the final refinement cycles. The configuration reported in the literature was used for the refinement.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *PLATON* (Spek, 2003), *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2007).

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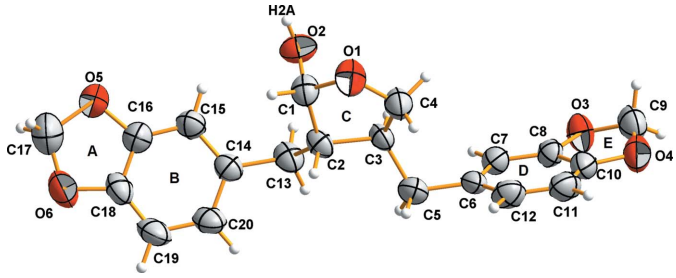

Figure 1
The molecular structure of the title compound, with the atom- and ring labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

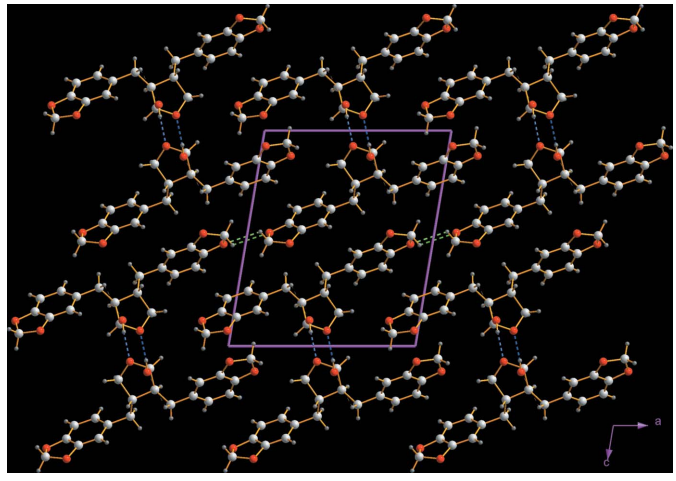

Figure 2
View along the *b* axis of the packing arrangement and intermolecular hydrogen bonds for the title compound. Blue dashed lines indicate O2—H2A···O1 hydrogen bonds and green dashed lines C9—H9A···O4.

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