Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Kadsuranin: a lignan from the fruit of *Schisandra chinensis*

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.006 Å
R factor = 0.083
wR factor = 0.196
Data-to-parameter ratio = 14.2

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

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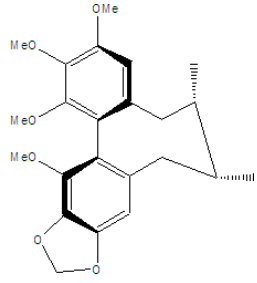
Received 14 January 2004
Accepted 1 March 2004
Online 13 March 2004

The title compound, kadsuranin or 5,6,7,8-tetrahydro-1,2,3,13-tetramethoxy-6,7-dimethylbenzo[3,4]cyclooct[1,2-*f*][1,3]-benzodioxole, $C_{23}H_{28}O_6$, is a dibenzocyclooctadiene-type lignan which was isolated from a Chinese medicine, *Schisandra chinensis* (the fruit of *Schisandra chinensis* Baill.). The molecule contains two six-membered aromatic rings, one five-membered ring, and an eight-membered ring with a twisted-boat–chair conformation.

Comment

Schisandra chinensis is a rich source of dibenzocyclooctadiene lignans, and it has been found to possess some beneficial pharmacological effects, including antihepatitis, antitumour and anti-HIV activities (Hancke *et al.*, 1999; Liu & Li, 1995). To find more about its bioactive constituents, we have investigated the fruit of *Schisandra chinensis*, which led to the isolation of the title compound, kadsuranin, (I). This is the first time this compound has been isolated from the genus *Schisandra*. The structure of (I) was elucidated by spectroscopic methods and confirmed by single-crystal X-ray diffraction analysis.



(I)

Compound (I) was obtained as colourless prisms. A view of the molecule of (I) with the atom-numbering scheme is shown in Fig. 1 and selected bond parameters are listed in Table 1. The molecule contains two six-membered rings, one five-membered ring and one eight-membered ring.

Experimental

Dried powder (4.5 kg) of the fruit of *Schisandra chinensis* was extracted three times with 95% EtOH at room temperature. The solvent was removed by evaporation at reduced pressure, and the residue was successively fractioned with petroleum ether, EtOAc and *n*-BuOH. The residue of the petroleum ether fraction was subjected to column chromatography on silica gel. The column was eluted with a petroleum ether–EtOAc mixture. The crude compound was purified by column chromatography on silica gel with an acetone–chloroform mixture and recrystallized with petroleum ether and

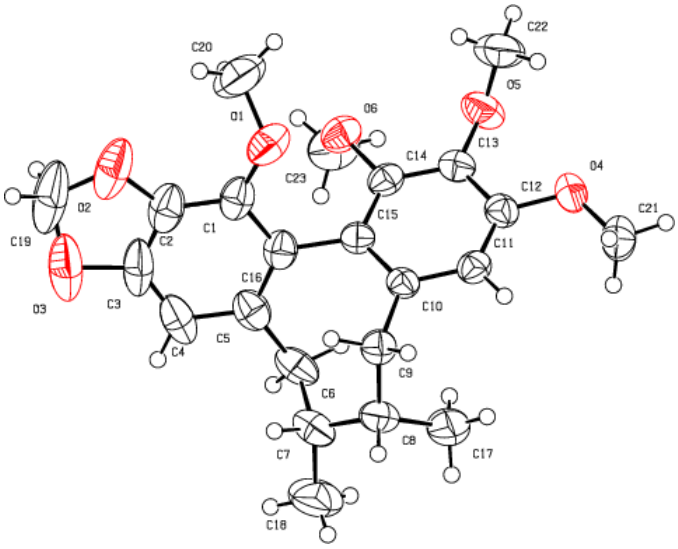


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 35% probability level.

EtOAc (2:1) to afford 2.201 g of the pure title compound, (I) (m.p. 392–394 K). Crystals of (I) suitable for X-ray structure analysis were obtained by slow evaporation of an aqueous solution in petroleum ether and EtOAc (2:1) at room temperature. Spectroscopic analysis: ^{13}C NMR (100 MHz, $CDCl_3$, δ, p.p.m): 151.6 (C3), 151.5 (C1), 148.6 (C12), 141.0 (C14), 140.0 (C2), 137.8 (C10), 134.5 (C13), 134.1 (C5), 123.2 (C16), 110.6 (C4), 102.9 (C11), 100.7 (C19), 61.0 (C22), 60.5 (C23), 59.6 (C20), 55.8 (C21), 40.6 (C8), 39.1 (C6), 35.5 (C9), 33.4 (C7), 21.5 (C18), 12.8 (C17).

Crystal data

$C_{23}H_{28}O_6$
M_r = 400.45
Triclinic, $P\overline{1}$
a = 10.5346 (7) Å
b = 10.9409 (7) Å
c = 11.0568 (7) Å
α = 83.315 (2)°
β = 70.256 (3)°
γ = 63.245 (2)°
V = 1070.09 (12) Å^3
Z = 2
D_x = 1.243 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 642 reflections
θ = 2.4–22.0°
μ = 0.09 mm^{-1}
T = 273 (2) K
Prism, colourless
0.45 × 0.18 × 0.11 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.963, T_{max} = 0.984
5758 measured reflections
3801 independent reflections
2443 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.2°
$h = -9 \rightarrow 12$
$k = -12 \rightarrow 13$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.083
$wR(F^2)$ = 0.196
S = 1.10
3801 reflections
268 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 0.2635P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.363 (4)	O4—C21	1.416 (4)
O1—C20	1.426 (5)	O5—C13	1.382 (4)
O2—C2	1.388 (5)	O5—C22	1.408 (5)
O2—C19	1.431 (6)	O6—C14	1.380 (4)
O3—C3	1.393 (5)	O6—C23	1.411 (4)
O3—C19	1.430 (7)	C1—C16	1.419 (5)
O4—C12	1.372 (4)	C15—C16	1.493 (4)
C1—O1—C20	117.9 (3)	C4—C3—O3	126.8 (5)
C2—O2—C19	104.0 (4)	C2—C3—O3	110.3 (5)
C3—O3—C19	103.3 (4)	C11—C12—O4	125.2 (3)
C12—O4—C21	117.2 (3)	C14—C13—O5	119.6 (3)
C13—O5—C22	114.5 (3)	O5—C13—C12	121.3 (3)
C14—O6—C23	114.3 (3)	O6—C14—C13	119.2 (3)
C2—C1—O1	123.6 (4)	O6—C14—C15	119.3 (3)
C1—C2—O2	129.7 (5)	O3—C19—O2	107.1 (4)
C3—C2—O2	108.9 (4)		

After their location in a difference Fourier map, all H atoms were geometrically positioned and allowed to ride on their attached atoms, with C—H distances in the range 0.93–0.98 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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