A single torsional parameter was refined for each methyl group. Otherwise, H atoms were placed in calculated positions, riding on their parent C atoms.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *WINGX* (Farrugia, 1996). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *ORTEP*3 (Farrugia, 1997). Software used to prepare material for publication: *WRITECIF* (Pauson & Muir, 1996).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1262). Services for accessing these data are described at the back of the journal.

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Gomisin N

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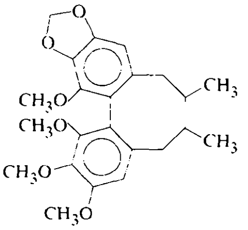
Abstract

The crystal structure of gomisin N (1,2,3,12-tetramethoxy-6,7-dimethyl-10,11-methylenedioxy-5,6,7,8-tetrahydrodibenzo[*a*,*c*]cyclooctene, $C_{23}H_{28}O_6$) has been determined. The cyclooctene ring of the molecule has a twisted boat–chair conformation. The angle between the two phenyl rings is 60.6 (11)°.

Comment

The title compound, (I), was originally isolated from the fruits of *Schisandra chinensis* Baill., a well known oriental medicinal plant (Ikeya *et al.*, 1979). Its structure was established by spectroscopic methods. Intense hepatoprotective action was found with gomisin N (Hikino *et al.*, 1984). In the course of our work on the biochemistry of natural lignans, we reisolated the title compound from the seeds of *Schisandra chinensis* (Lojková *et al.*, 1997; Slanina *et al.*, 1997).



(I)

The main part of the molecule is the cyclooctadiene ring, which possesses a twisted boat–chair conformation. The methyl group bonded to the cyclooctadiene ring at C7 is in a pseudo-axial position, while the methyl group bonded at C8 is in a pseudo-equatorial position. Two phenyl rings are connected to the eight-membered cycle. Both of these rings are relatively planar: the maximum deviations of best planes through atoms C10–C15, and C1–C5 and C16, are 0.01 (5) and 0.03 (2) Å, respectively (or 0.2 and 1.6, respectively, on a δ/σ scale). The angle between these planes is 60.6 (11)°. The angle between the analogous planes in 4,11-dibromogomisin D is 73° (Ikeya *et al.*, 1976).

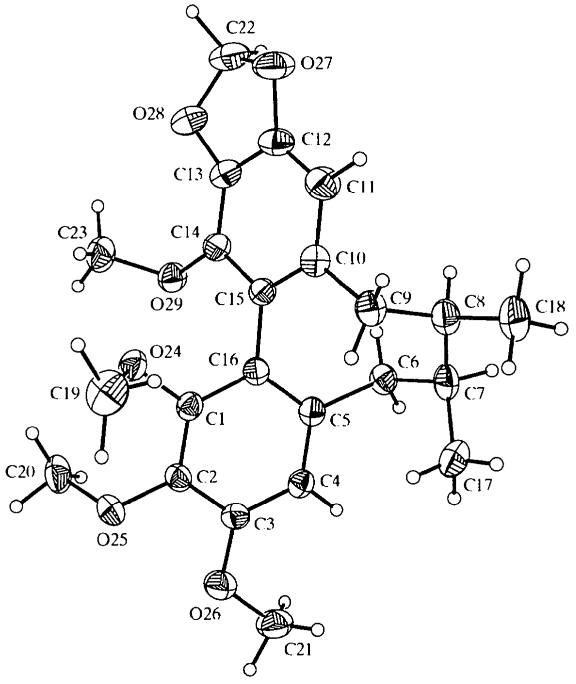


Fig. 1. *ORTEP*III (Johnson & Burnett, 1996) plot of the title compound. Non-H atoms are drawn with 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radii.

The intermolecular contacts in (I) are based mainly on hydrophobic and C—H···O interactions: C19—H19*A*···O25 (H···O = 2.48 Å), C20—H20*B*···O24 (2.39 Å), C22—H22*A*···O26 [2.3 (2) Å], C23—H23*A*···O25 (2.65 Å), C23—H23*B*···O24 (2.63 Å) and C23—H23*C*···O28 (2.47 Å).

Experimental

We reisolated crystals of (I) from the seeds of *Schisandra chinensis* using petroleum ether extraction, silica-gel column chromatography and crystallization from 95% ethanol at room temperature (Slanina *et al.*, 1995).

Crystal data

$C_{23}H_{28}O_6$
$M_r = 400.45$
Orthorhombic
$P2_12_12_1$
$a = 8.7171\ (8)$ Å
$b = 10.9766\ (12)$ Å
$c = 21.946\ (4)$ Å
$V = 2099.9\ (5)$ Å^3
$Z = 4$
$D_x = 1.267$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10.4–14.0°$
$\mu = 0.091$ mm^{-1}
$T = 291\ (2)$ K
Prism
0.8 × 0.8 × 0.4 mm
Colourless

Data collection

Kuma KM-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2208 measured reflections
2184 independent reflections
1684 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.149$

$\theta_{\rm max} = 25.24°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 26$
3 standard reflections every 60 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.100$
$S = 0.955$
2184 reflections
309 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.50P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.159$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.176$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0019 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—C2	1.380 (4)	C10—C15	1.395 (4)
C1—C16	1.406 (4)	C10—C11	1.397 (4)
C2—C3	1.391 (4)	C11—C12	1.366 (5)
C3—C4	1.390 (4)	C12—C13	1.369 (4)
C4—C5	1.399 (4)	C12—O27	1.378 (4)
C5—C16	1.393 (4)	C13—C14	1.373 (4)
C5—C6	1.524 (4)	C13—O28	1.395 (3)
C6—C7	1.551 (4)	C14—C15	1.419 (4)
C7—C8	1.543 (5)	C15—C16	1.487 (4)
C8—C9	1.541 (4)	C22—O27	1.424 (5)
C9—C10	1.518 (4)	C22—O28	1.433 (5)
O26—C3—C4	124.5 (3)	C13—C12—O27	109.9 (3)
C5—C6—C7	117.1 (2)	C12—C13—O28	109.7 (3)
C17—C7—C8	114.3 (3)	C14—C13—O28	128.6 (3)
C18—C8—C7	112.0 (3)	C3—O26—C21	117.7 (3)
C10—C9—C8	112.7 (3)	C12—O27—C22	104.8 (2)
C11—C12—O27	128.0 (3)	C13—O28—C22	103.9 (3)

The absolute structure could not be determined reliably due to a lack of significant anomalous scattering; the Flack (1983) parameter was −2.5 (16).

Data collection: *KM-4 Software* (Kuma, 1992). Cell refinement: *KM-4 Software*. Data reduction: *KM-4 Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*III (Johnson & Burnett, 1996). Software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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