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# 5,6-Dihydroxy-1,2,3,13-tetramethoxy-6,7-dimethyl-5,6,7,8-tetrahydrobenzo[3,4]cyclooctа[1,2-*f*][1,3]-benzodioxol-5-yl benzoate sesquihydrate

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.006 Å
*R* factor = 0.048
*wR* factor = 0.131
Data-to-parameter ratio = 8.8

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



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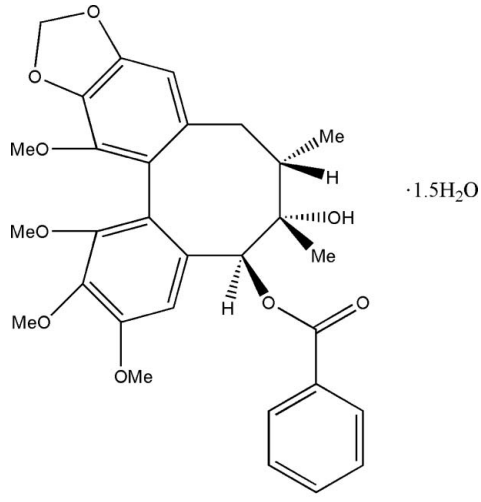
In the title compound, $C_{30}H_{32}O_9\cdot1.5H_2O$, the cyclooctadiene eight-membered ring adopts a twisted boat–chair conformation. O—H···O hydrogen bonding helps to stabilize the crystal structure.

## Comment

The title compound (common name Schisantherin A), (I), was isolated from a Chinese medicine, Fructus Schisandrae sphenantherae (the fruit of *Schisandra sphenanthera* Rehd. et Wils.), and was found to have antihepatotoxic activity (Fang *et al.*, 1975). We report here the crystal structure of (I).




(I)

The molecular structure of (I) is shown in Fig. 1. The cyclooctadiene eight-membered ring adopts a twisted boat–chair conformation. The benzodioxole group is planar and makes a dihedral angle with the C27-containing benzene ring of 67.5 (2)°. The C16-containing ring is nearly perpendicular to the C27-benzene plane, the dihedral angle being 88.0 (2)°.

Adjacent Schisantherin A molecules link together *via* O—H···O hydrogen bonding involving solvent water molecules (Table 1).

## Experimental

The dried fruits of *Schisandra sphenanthera* (1500 g) were extracted with ethanol and fractionated into $CHCl_3$- and $H_2O$-soluble fractions. The $CHCl_3$-soluble fraction was chromatographed on a silica gel column, eluting with $CHCl_3$–EtOAc (2:1) to afford Schisantherin A, (I). Colorless single crystals of (I) were obtained by slow evaporation of an ethanol solution.

*Crystal data*

$C_{30}H_{32}O_9\cdot1.5H_2O$
$M_r$ = 563.58
Orthorhombic, $P2_12_12$
$a$ = 12.818 (3) Å
$b$ = 27.883 (7) Å
$c$ = 7.921 (2) Å
$V$ = 2831.0 (12) Å$^3$
$Z$ = 4
$D_x$ = 1.322 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 (2) K
Block, colorless
0.26 × 0.22 × 0.18 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: none
10679 measured reflections
3264 independent reflections
1932 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
$\theta_{max}$ = 26.4°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.131
$S$ = 1.00
3264 reflections
372 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4$A$···O11 | 0.84 | 2.14 | 2.900 (5) | 150 |
| O10—H10$A$···O4 | 0.90 | 1.90 | 2.797 (6) | 178 |
| O10—H10$B$···O9$^{i}$ | 0.84 | 2.23 | 3.071 (6) | 175 |

Symmetry code: (i) $x, y, z-1$.

H atoms on O atoms were located in a difference Fourier map and refined as riding in their as-found relative positions [O—H = 0.84–0.92 Å, $U_{iso}$(H) = 1.5$U_{eq}$(O)]. Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angles were refined to fit the electron density; $U_{iso}$(H) = 1.5$U_{eq}$(C). Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic), 0.97 (methylene) or 0.98 Å (methine), and refined in riding mode with $U_{iso}$(H) = 1.2$U_{eq}$(C). In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration of (I) was assigned arbitrarily.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

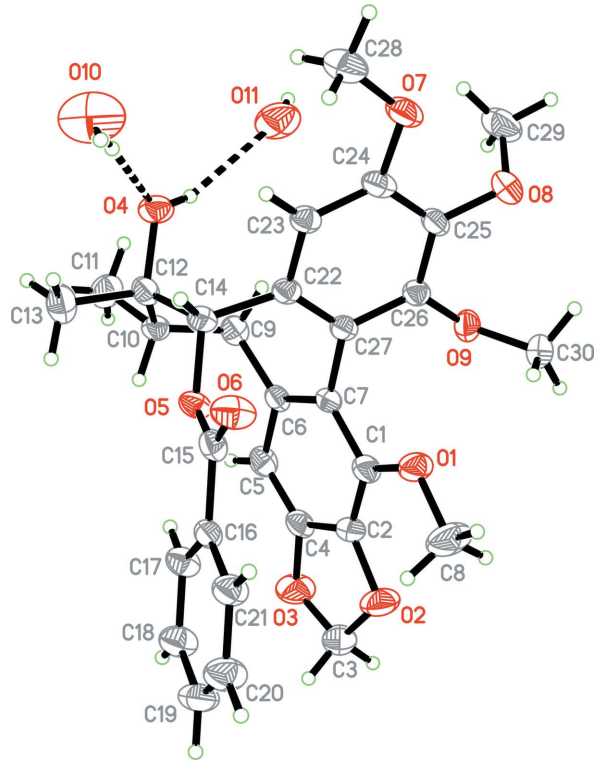



**Figure 1**
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate hydrogen bonds.

## References

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Fang, S.-D., Huang, M.-F., Liu, L.-S., Gao, Y.-L. & Hu, J.-S. (1975). *Acta Chim. Sinica*, **33**, 57–60.
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