

Tetrahydroepiberberine

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Key indicators

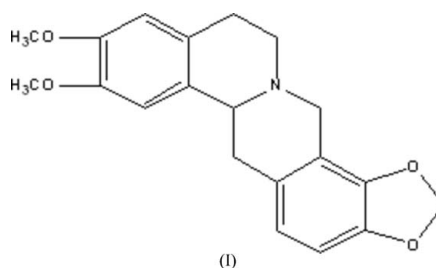
Single-crystal X-ray study
 $T = 288\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.038
 wR factor = 0.086
Data-to-parameter ratio = 9.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound [systematic name: 6,6a,11,12-tetrahydro-8,9-dimethoxy-14*H*-dioxolo[4',5':7,8]isoquinolino[3,2-*a*]isoquinoline], also known as sinactine, $\text{C}_{20}\text{H}_{21}\text{NO}_4$, a protoberberine-type alkaloid, was isolated from the roots of the plant *Sinomenium acutum*. Both piperidine rings adopt half-chair conformations.

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Comment

Sinomenium acutum is distributed mainly in hilly regions of southwest, northwest and southeast China. The roots and stems of the plant are used as a folk medicine to cure rheumatism, dropsy and dermatophytosis. A number of alkaloids with different skeletons have been isolated from the plant (Jiangsu New Medical College, 1985; Chen *et al.*, 1991; Moriyasu *et al.*, 1993, 1994). In the course of a systematic search for bioactive substances from traditional Chinese herbal medicines, we have studied the roots of *S. acutum* and obtained several compounds, including the title compound, (I). Compound (I) has been isolated from several plant species (Dewan *et al.*, 1983). Previously, we have reported the crystal structures of cheilanthifoline (Wang *et al.*, 2006) and 8-oxotetrahydropalmatine (Wang, 2006). We report here the crystal structure of (I).



The 1,3-benzodioxole ring system is essentially planar (Fig. 1). Both piperidine rings adopt half-chair conformations. The methoxy group attached at atom C3 is coplanar with the C1–C4/C4A/C14 benzene ring, but that at atom C2 is twisted away from the ring, with a C17–O4–C2–C1 torsion angle of $-17.5(4)^\circ$.

C–H \cdots O and C–H $\cdots\pi$ hydrogen-bonding interactions are observed in the crystal structure (Table 1).

Experimental

Sinomenine is produced from the powder of the roots of *S. acutum* by the Baoji Yongjia Plant Medicine Extracting Company Limited, Baoji, People's Republic of China. It was obtained from the benzene extract of the powder in a vacuum (Chen *et al.*, 1995). The remaining

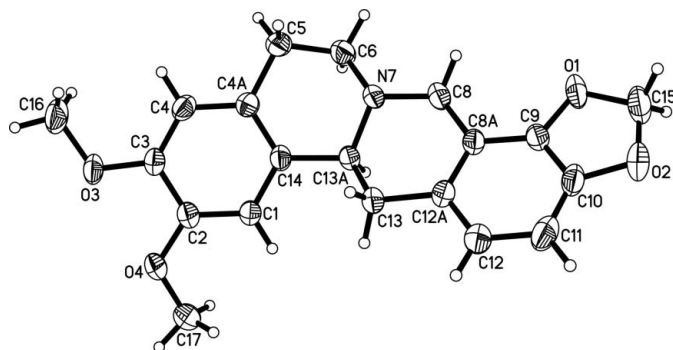


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

benzene mother liquor (3 kg), after the extraction of sinomenine, was obtained from the company. It was subjected to repeated chromatography on a silica-gel column, and eluted with petroleum ether/acetone (from 10:1 to 3:1) to afford compound (I) (0.01 g). Single crystals of (I) were obtained after repeated recrystallization from acetone.

Crystal data

$C_{20}H_{21}NO_4$
 $M_r = 339.38$
 Orthorhombic, $P2_12_12_1$
 $a = 7.367$ (1) Å
 $b = 8.377$ (2) Å
 $c = 27.676$ (5) Å
 $V = 1708.0$ (6) Å³
 $Z = 4$
 $D_x = 1.320$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 32 reflections
 $\theta = 2.5$ – 16.8°
 $\mu = 0.09$ mm⁻¹
 $T = 288$ (2) K
 Block, colourless
 $0.58 \times 0.46 \times 0.38$ mm

Data collection

Siemens P4 diffractometer
 ω scans
 Absorption correction: none
 2472 measured reflections
 2277 independent reflections
 1530 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.012$

$\theta_{max} = 27.5^\circ$
 $h = 0 \rightarrow 9$
 $k = 0 \rightarrow 10$
 $l = -1 \rightarrow 35$
 3 standard reflections every 97 reflections
 intensity decay: 1.6%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.086$
 $S = 0.95$
 2277 reflections
 229 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.16$ e Å⁻³
 $\Delta\rho_{min} = -0.13$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0166 (16)

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C5-H5B \cdots O3^i$	0.97	2.58	3.504 (3)	160
$C17-H17C \cdots O2^{ii}$	0.96	2.59	3.527 (4)	166
$C16-H16C \cdots Cg1^i$	0.96	2.65	3.489 (3)	146
$C6-H6B \cdots Cg2^{iii}$	0.97	2.81	3.697 (3)	153

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$; (ii) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$. $Cg1$ and $Cg2$ denote the centroids of the C1–C4/C4A/C14 and C9–C12/C12A/C8A rings, respectively.

H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.98 Å. The $U_{iso}(H)$ values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. In the absence of significant anomalous scattering, Friedel pairs were merged prior to the final refinement.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997b); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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