

Cheilanthifoline

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Key indicators

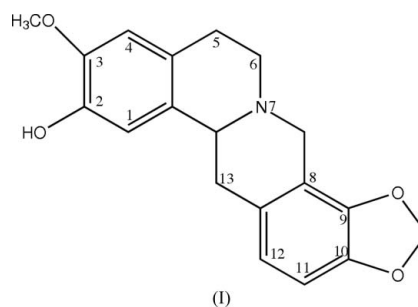
Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.036
 wR factor = 0.089
Data-to-parameter ratio = 7.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, 6,6a,11,14-tetrahydro-8-hydroxy-9-methoxy-12*H*-benzo[*a*]-1,3-benzodioxolo[4,5-*g*]quinolizine, $\text{C}_{19}\text{H}_{19}\text{NO}_4$, a protoberberine-type alkaloid, was isolated from the roots of the plant *Sinomenium acutum*. Both the piperidine rings adopt half-chair conformations. The molecules exist in a dimeric form through $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding between the hydroxy group and methoxy O atom.

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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 folk medicine to cure rheumatism, dropsy and dermatophytosis. A number of alkaloids with different kinds of 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 our systematic search for bioactive substances from Chinese traditional herb medicines, we have studied the roots of *S. acutum* and obtained the title compound, (I). Compound (I) was first isolated in some species of *Corydalis* and identified on the basis of its mass, NMR, IR and UV spectra (Blask *et al.*, 1981; Haisov & Slavk, 1973). We report here the crystal structure of (I).



The benzo[1,3]dioxole ring system is essentially planar (Fig. 1). Both the piperidine rings adopt half-chair conformations. The methoxy group attached at atom C3 is twisted away from the benzene ring with a torsion angle $\text{C}20-\text{O}4-\text{C}3-\text{C}4$ of $25.2(4)^\circ$. In the crystal packing, intermolecular hydrogen bonding between the hydroxy group and the methoxy O atom of an adjacent molecule leads to the formation of dimers (Fig. 2). In addition, a $\text{C}-\text{H}\cdots\pi$ interaction is observed (Table 1).

Experimental

Sinomenine is produced from the powder of the roots of *S. acutum* by the Baoji Yongjia Plant Medicine Extracting Limited Company,

Baoji, People's Republic of China. It is obtained from the benzene extract of the powder in a vacuum (Chen *et al.*, 1995). The remaining 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 20:1 to 3:1) to afford compound (I) (0.2 g). Single crystals of (I) were obtained after repeated recrystallization from acetone.

Crystal data

C₁₉H₁₉NO₄ $D_x = 1.358 \text{ Mg m}^{-3}$
 M_r = 325.35 Mo K α radiation
 Monoclinic, C2 Cell parameters from 39 reflections
 a = 13.314 (2) Å $\theta = 2.6\text{--}11.5^\circ$
 b = 5.151 (1) Å $\mu = 0.10 \text{ mm}^{-1}$
 c = 23.585 (3) Å T = 296 (2) K
 $\beta = 100.39 (1)^\circ$ Rhombohedron, yellow
 V = 1590.9 (4) Å³ 0.48 × 0.34 × 0.14 mm
 Z = 4

Data collection

Siemens P4 diffractometer $\theta_{\text{max}} = 25.5^\circ$
 ω scans $h = 0 \rightarrow 16$
 Absorption correction: none $k = 0 \rightarrow 6$
 1816 measured reflections $l = -28 \rightarrow 28$
 1669 independent reflections 3 standard reflections
 1331 reflections with $I > 2\sigma(I)$ every 97 reflections
 R_{int} = 0.013 intensity decay: 3.9%

Refinement

Refinement on F² $w = 1/[\sigma^2(F_o^2) + (0.051P)^2]$
 R[F² > 2σ(F²)] = 0.036 where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.089$ (Δ/σ)_{max} = 0.001
 S = 0.99 $\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{Å}^{-3}$
 1669 reflections $\Delta\rho_{\text{min}} = -0.14 \text{ e } \text{Å}^{-3}$
 223 parameters Extinction correction: SHELXTL
 H atoms treated by a mixture of independent and constrained refinement Extinction coefficient: 0.0144 (14)

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

D—H...A	D—H	H...A	D...A	D—H...A
O3—H3O...O4	0.91 (4)	2.29 (3)	2.688 (3)	106 (2)
O3—H3O...O4 ⁱ	0.91 (4)	1.98 (4)	2.870 (3)	163 (3)
C20—H20C...Cg1 ⁱⁱ	0.96	2.62	3.460 (3)	146

Symmetry codes: (i) $-x + 1, y, -z$; (ii) $x, y - 1, z$. Cg1 is the centroid of the C1–C4/C14 ring.

The hydroxy H atom was located in a difference Fourier map and refined freely. The other 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, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. 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.

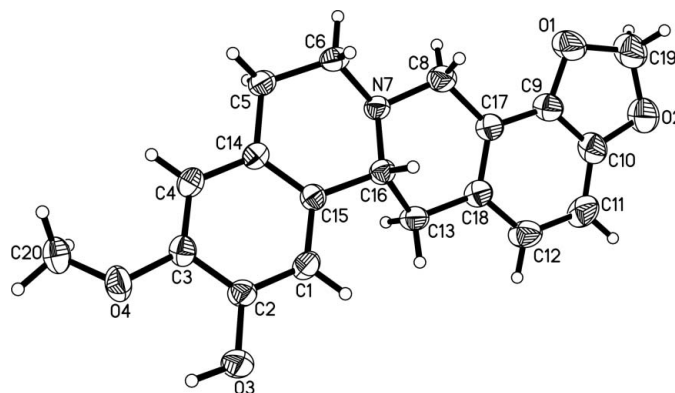


Figure 1 The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

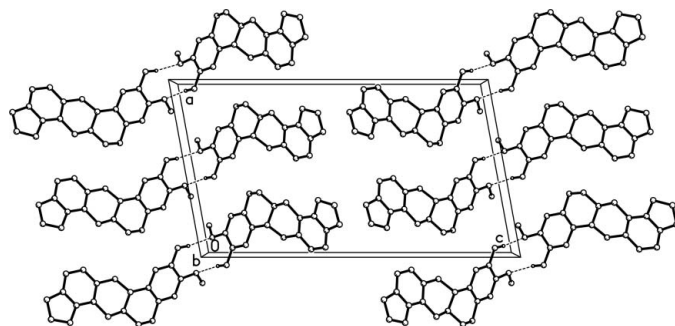


Figure 2 The crystal packing of (I), showing O—H...O hydrogen-bonded (dashed lines) dimers. H atoms not involved in the interactions shown have been omitted.

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