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8-Oxotetrahydropalmatine

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The title compound [systematic name: 2,3,9,10-tetramethoxy-5,6,13,13a-tetrahydroisoquino[3,2-*a*]isoquinolin-8-one], $C_{21}H_{23}NO_5$, a protoberberine-type alkaloid, was isolated from the roots of the plant *Sinomenium acutum*. The piperidine ring adopts an envelope conformation and the pyridinone ring is in a screw–boat conformation.

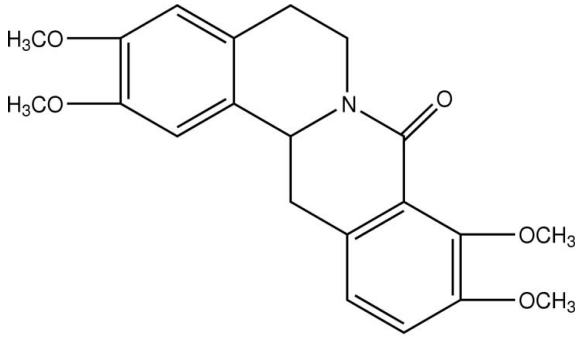
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 9.3

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

Comment

Sinomenium acutum is distributed mainly in the hilly regions of southwest, northwest and southeast China. The roots and stems of the plant are used in 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 traditional Chinese herbal medicines, we have studied the roots of *S. acutum* and obtained several compounds, including the title compound, (I). Compound (I) was first isolated from the stem and roots of *Anamirta cocculus* and identified on the basis of its mass, UV and NMR spectra (Verpoorte *et al.*, 1981). Previously, we have reported the crystal structure of Cheilanthifoline (Wang *et al.*, 2006). We report here the crystal structure of (I).



(I)

The piperidine ring of (I) adopts an envelope conformation, whereas the pyridinone ring is in a screw–boat conformation (Fig. 1). The methoxy groups attached at atoms C2 and C3 are essentially coplanar with the C1–C4/C14/C15 benzene ring (Table 1). The methoxy group attached at atom C10 is almost coplanar with the C9–C12/C17/C18 benzene ring, but that at atom C9 is twisted away from the ring with a torsion angle C21—O4—C9—C10 of 93.0 (3)°.

C—H···O and C—H···π hydrogen bonding interactions are observed in the crystal structure (Table 2).

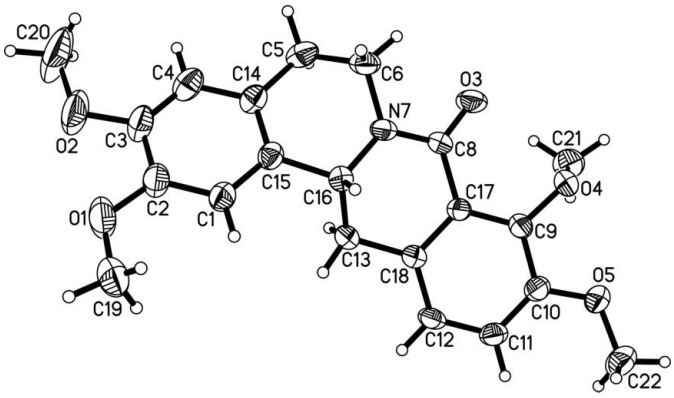


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

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 is obtained from the benzene extract of the powder in vacuum (Chen *et al.*, 1995). The remaining benzene mother liquor (3 kg), after 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 3:1 to 2:1) to afford compound (I) (0.02 g). Single crystals of (I) were obtained after repeated recrystalization from petroleum ether–acetone (1:1).

Crystal data

$C_{21}H_{23}NO_5$
M_r = 369.40
Monoclinic, $P2_1$
a = 7.875 (1) Å
b = 7.870 (1) Å
c = 15.139 (3) Å
β = 93.335 (15)°
V = 936.7 (3) Å^3
Z = 2
D_x = 1.310 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 33 reflections
θ = 3.7–14.8°
μ = 0.09 mm^{-1}
T = 296 (2) K
Block, yellow
0.48 × 0.38 × 0.26 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
2601 measured reflections
2318 independent reflections
1515 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.5°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 10$
$l = -19 \rightarrow 19$
3 standard reflections
every 97 reflections
intensity decay: 1.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 0.92
2318 reflections
249 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997*a*)
Extinction coefficient: 0.008 (2)

Table 1
Selected geometric parameters (Å, °).

O3—C8	1.220 (3)	N7—C6	1.455 (3)
N7—C8	1.361 (3)	N7—C16	1.470 (3)
C19—O1—C2—C1	3.7 (5)	C21—O4—C9—C17	−90.4 (3)
C20—O2—C3—C4	−4.4 (6)	C22—O5—C10—C11	−14.3 (4)
C21—O4—C9—C10	93.0 (3)	C22—O5—C10—C9	165.7 (3)

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 denote the centroids of the C9–C12/C17/C18 and C1–C4/C14/C15 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O3^{i}	0.93	2.33	3.127 (3)	144
C19—H19C···O4^{ii}	0.96	2.56	3.407 (5)	147
C5—H5B···Cg1^{iii}	0.97	2.57	3.519 (3)	165
C6—H6B···Cg1^{iv}	0.97	2.56	3.474 (3)	157
C22—H22A···Cg2^{ii}	0.96	2.83	3.778 (4)	167

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, y-\frac{1}{2}, -z+1$; (iii) $-x+1, y+\frac{1}{2}, -z+1$; (iv) $-x, y+\frac{1}{2}, -z+1$.

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. Owing to the absence of any significant anomalous dispersion in the molecules, Friedel pairs were merged before the final refinement.

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

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References

Chen, C., Sun, L. J. & Xu, H. Q. (1995). Chinese Patent No. 1125724.
Chen, Y. Y., Qiu, C. C. & Shen, L. (1991). *Beijing Yike Daxue Xuebao*, **23**, 235–237. (In Chinese).
Jiangsu New Medical College (1985). *The Dictionary of Chinese Medicine*, pp. 1234–1236. Shanghai: Shanghai Science and Technology Press.
Moriyasu, M., Ichimaru, M. & Nishiyama, Y. (1993). *Bunseki Kagaku*, 42, 659–665. (In Japanese).
Moriyasu, M., Ichimaru, M. & Nishiyama, Y. (1994). *Nat. Med.* **48**, 287–290.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Verpoorte, R., Siwon, J. & Tieken M. E. M. (1981). *J. Nat. Prod.* **44**, 221–223.
Wang, X.-L., Li, Z.-X. & Qin, G.-W. (2006). *Acta Cryst.* E**62**, o81–o82.