Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-Oxocanadine

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Received 18 March 2006
Accepted 21 March 2006

Key indicators

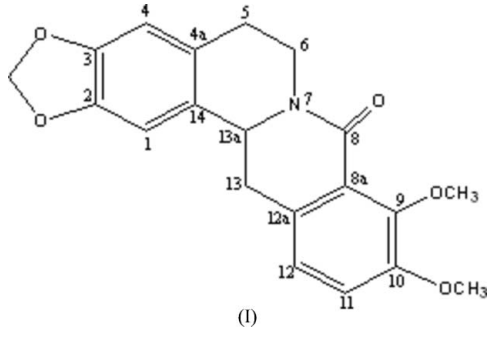
Single-crystal X-ray study
T = 286 K
Mean σ(C–C) = 0.005 Å
R factor = 0.040
wR factor = 0.076
Data-to-parameter ratio = 6.9

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

The title compound, $C_{20}H_{19}NO_5$, a protoberberine-type alkaloid, was isolated from the roots of the plant *Sinomenium acutum*. The piperidine ring adopts a screw-boat conformation and the pyridinone ring is in an envelope conformation.

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 herbal medicines, we have studied the roots of *S. acutum* and obtained several compounds, including the title compound, (I), which is reported here. 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 (Zhang *et al.*, 1991). Previously, we have reported the crystal structures of cheilanthifoline (Wang *et al.*, 2006), 8-oxotetrahydropalmatine (Wang, 2006*a*) and tetrahydroepiberberine (Wang, 2006*b*).



(I)

The piperidine ring adopts a screw-boat conformation whereas the pyridinone ring is in an envelope conformation (Fig. 1). The methoxy group attached at atom C10 is essentially coplanar with the C9–C12/C8*A*/C12*A* benzene ring with a torsion angle C20—O5—C10—C9 of 164.9 (3)°, but that at atom C9 is twisted away from the benzene ring with a torsion angle C19—O4—C9—C10 of −84.5 (4)°. The 1,3-benzodioxole ring system is essentially planar with a C18—O2—C3—C4 torsion angle of 170.6 (4)°.

Experimental

Sinomenine was 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 was obtained from a benzene extract of the powder *in vacuo* (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 3:1 to 2:1) to afford compound (I) (0.01 g). Single crystals of (I) were obtained after repeated recrystalization from methanol.

Crystal data

$C_{20}H_{19}NO_5$	D_x = 1.401 Mg m^{-3}
M_r = 353.36	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 33 reflections
a = 5.132 (2) Å	θ = 3.9–15.5°
b = 7.203 (2) Å	μ = 0.10 mm^{-1}
c = 22.767 (8) Å	T = 286 (2) K
β = 95.59 (3)°	Needle, yellow
V = 837.5 (5) Å^3	0.56 × 0.24 × 0.20 mm
Z = 2	

Data collection

Siemens P4 diffractometer	θ_{max} = 25.2°
ω scans	$h = 0 \rightarrow 6$
Absorption correction: none	$k = 0 \rightarrow 8$
2412 measured reflections	$l = -27 \rightarrow 27$
1651 independent reflections	3 standard reflections
1165 reflections with $I > 2\sigma(I)$	every 97 reflections
R_{int} = 0.016	intensity decay: 3.5%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.076	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 1.00	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1651 reflections	Extinction correction: *SHELXL*
238 parameters	Extinction coefficient: 0.0134 (19)
H-atom parameters constrained	

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} 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 any significant anomalous dispersion, Friedel pairs were merged before the final refinement.

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

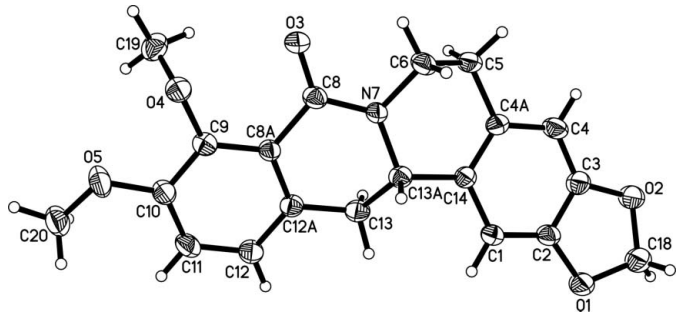


Figure 1
The structure of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

The author thanks the Phytochemistry Key Laboratory of Shaanxi province for the research grant (No. 05js43) and Professor Kai-Bei Yu, Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, for diffraction measurements.

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 5.0). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Wang, X. L. (2006a). *Acta Cryst.* E**62**, o498–o499.
Wang, X. L. (2006b). *Acta Cryst.* E**62**, o1271–o1272.
Wang, X. L., Li, Z. X. & Qin, G. W. (2006). *Acta Cryst.* E**62**, o81–o82.
Zhang, J. S. & Chen, Z. L. (1991). *Planta Med.* **57**, 457–459.