Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Mersinine A from *Kopsia fruticosa*

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Received 20 February 2003
Accepted 20 March 2003
Online 31 March 2003

The pentacyclic quinoline alkaloid mersinine A, $C_{25}H_{28}N_2O_9$, was isolated from the leaves of *Kopsia fruticosa* (Ker.) A DC. The molecule has a hydroxyl group that forms a hydrogen bond with the carbonyl O atom of an adjacent molecule, which gives rise to infinite chains running along the *c* axis of the crystal.

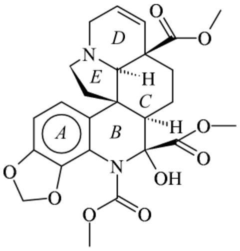
Key indicators

Single-crystal X-ray study
T = 168 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.084
Data-to-parameter ratio = 7.6

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

Comment

Mersinines A and B, and mersiloscine are pentacyclic quinoline alkaloids that are found within the genus *Kopsia*. These alkaloids contain a meloscine skeleton whose five-membered *C* ring has an additional C atom. The title alkaloid, mersinine A, (I), isolated from the leaves of *Kopsia fruticosa* (Ker.) A DC, possesses a tetrahydroquinoline chromophore and its structure has been determined by solution NMR spectroscopy (Kam *et al.*, 2001). The present study reports the solid-state structure, which confirms the NMR results (Fig. 1). The hydroxyl group forms a hydrogen bond to the carbonyl O atom of an adjacent carboxyl unit [$O9\cdots O6^{i}$ 2.873 (2) Å and $O9—H9O\cdots O6^{i}$ 133 (3)°; translation code: (i) $x, y, z-1$], which gives rise to linear chains running along the *c* axis of the crystal.



(I)

Experimental

The title alkaloid was isolated from the leaves of *Kopsia fruticosa* (Ker.) A DC (Kam *et al.*, 2001), and crystals were grown from a solution in ether.

Crystal data

$C_{25}H_{28}N_2O_9$
M_r = 500.49
Monoclinic, $C2$
a = 29.005 (2) Å
b = 8.5430 (6) Å
c = 9.3081 (7) Å
β = 96.163 (1)°
V = 2293.1 (3) Å^3
Z = 4
D_x = 1.450 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5856 reflections
θ = 2.2–26.5°
μ = 0.11 mm^{-1}
T = 168 (2) K
Plate, colorless
0.50 × 0.16 × 0.14 mm

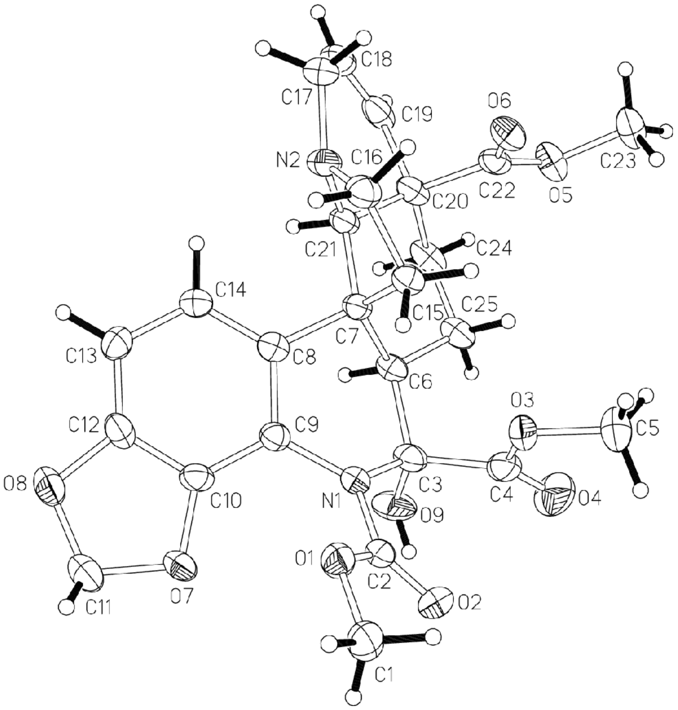


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

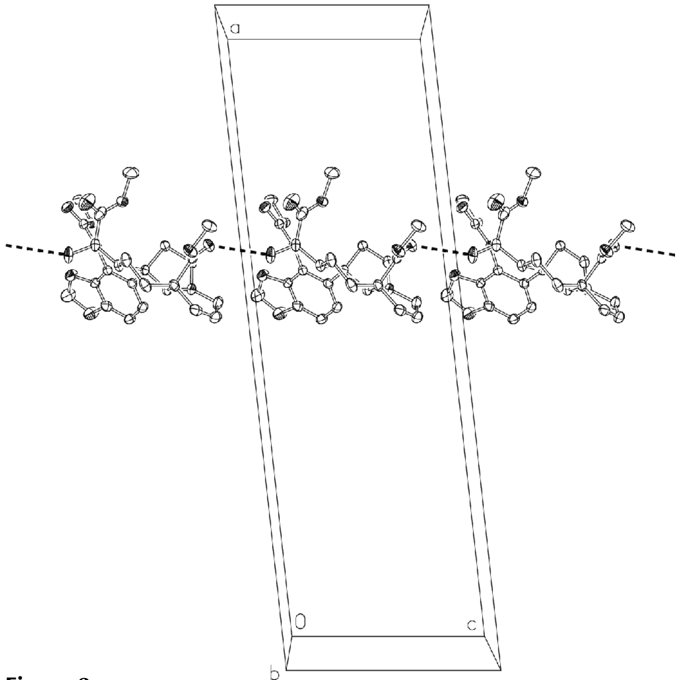


Figure 2
*ORTEP*II (Johnson, 1976) plot, showing the infinite chains of molecules linked by O—H···O bonds.

Data collection

Bruker AXS CCD area-detector diffractometer
ω scans
Absorption correction: none
15060 measured reflections
2493 independent reflections

2173 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\rm max} = 26.5°$
$h = -35 \rightarrow 36$
$k = -10 \rightarrow 5$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.084$
$S = 1.02$
2493 reflections
329 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.449 (3)	C3—C6	1.549 (3)
O1—C2	1.317 (3)	C6—C7	1.548 (3)
O2—C2	1.206 (3)	C7—C8	1.508 (3)
O3—C4	1.337 (4)	C6—C25	1.517 (3)
O3—C5	1.447 (3)	C7—C15	1.549 (3)
O4—C4	1.182 (3)	C7—C21	1.537 (3)
O5—C22	1.325 (4)	C8—C9	1.410 (3)
O5—C23	1.448 (3)	C8—C14	1.382 (3)
O6—C22	1.202 (3)	C9—C10	1.370 (3)
O7—C10	1.370 (3)	C10—C12	1.377 (4)
O7—C11	1.432 (3)	C12—C13	1.362 (3)
O8—C11	1.425 (3)	C13—C14	1.388 (4)
O8—C12	1.369 (3)	C15—C16	1.537 (3)
O9—C3	1.396 (3)	C17—C18	1.476 (4)
N1—C2	1.381 (3)	C18—C19	1.319 (4)
N1—C3	1.453 (3)	C19—C20	1.520 (3)
N1—C9	1.423 (3)	C20—C21	1.545 (3)
N2—C16	1.459 (3)	C20—C22	1.536 (3)
N2—C17	1.467 (3)	C20—C24	1.523 (4)
N2—C21	1.433 (3)	C24—C25	1.526 (4)
C3—C4	1.541 (4)		
C1—O1—C2	114.2 (2)	C7—C8—C14	123.8 (2)
C4—O3—C5	116.3 (2)	C9—C8—C14	119.9 (2)
C22—O5—C23	115.8 (2)	C8—C9—C10	117.4 (2)
C10—O7—C11	104.8 (2)	C8—C9—N1	118.6 (2)
C11—O8—C12	105.6 (2)	C10—C9—N1	123.9 (2)
C2—N1—C3	116.0 (2)	O7—C10—C9	128.5 (2)
C2—N1—C9	124.2 (2)	O7—C10—C12	110.7 (2)
C3—N1—C9	119.3 (2)	C9—C10—C12	120.8 (2)
C16—N2—C17	121.3 (2)	O7—C11—O8	108.9 (2)
C16—N2—C21	110.1 (2)	C13—C12—O8	126.9 (2)
C17—N2—C21	117.4 (2)	C10—C12—C13	123.4 (2)
O1—C2—O2	125.2 (2)	O8—C12—C10	109.7 (2)
O1—C2—N1	112.3 (2)	C12—C13—C14	116.0 (2)
O2—C2—N1	122.4 (2)	C8—C14—C13	122.4 (2)
O9—C3—N1	111.8 (2)	C7—C15—C16	102.4 (2)
O9—C3—C4	109.8 (2)	N2—C16—C15	105.7 (2)
O9—C3—C6	105.5 (2)	N2—C17—C18	113.0 (2)
N1—C3—C4	110.1 (2)	C17—C18—C19	124.1 (2)
N1—C3—C6	109.4 (2)	C18—C19—C20	119.5 (3)
C4—C3—C6	110.2 (2)	C19—C20—C22	102.2 (2)
O3—C4—O4	123.9 (3)	C19—C20—C24	116.7 (2)
O3—C4—C3	111.8 (2)	C19—C20—C21	103.3 (2)
O4—C4—C3	124.1 (3)	C21—C20—C22	117.0 (2)
C3—C6—C25	113.6 (2)	C21—C20—C24	107.8 (2)
C7—C6—C25	115.2 (2)	C22—C20—C24	110.1 (2)
C3—C6—C7	114.7 (2)	N2—C21—C7	104.1 (2)
C6—C7—C8	103.7 (2)	N2—C21—C20	113.6 (2)
C6—C7—C21	109.1 (2)	C7—C21—C20	117.7 (2)
C8—C7—C15	108.6 (2)	O5—C22—O6	121.9 (2)
C8—C7—C21	111.7 (2)	O5—C22—C20	110.7 (2)
C6—C7—C15	123.2 (2)	O6—C22—C20	127.2 (2)
C15—C7—C21	100.6 (2)	C20—C24—C25	109.1 (2)
C7—C8—C9	116.2 (2)	C6—C25—C24	110.1 (2)

Refinement of the Flack parameter (Flack & Schwarzenbach, 1988) was suppressed by the MERG 4 command in *SHELXL*97 (Sheldrick, 1997), as the lack of anomalous scatterers did not allow the determination of the absolute configuration from the X-ray measurements. The absolute configuration was taken as reported in the solution NMR study of the compound (Kam *et al.*, 2001). That study had assumed the configuration to be that of a structurally related compound, meloscine (Bernauer *et al.*, 1969), whose absolute configuration was determined from the X-ray structure of the brominated derivative (Oberhänsli, 1969). The C-bound H atoms were positioned geometrically, and were allowed to ride on their parent atoms [$U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for the aromatic, methylene and methine C atoms, and 1.5$U_{\rm eq}$(C) for the methyl C atoms]. The hydroxyl H atom was located and refined freely [O9—H1O = 0.80 (4) Å].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

The authors thank Dr Jan Wikaira of the University of Canterbury for the diffraction measurements and the University of Malaya (F0717/2002A) for supporting this work.

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