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8,9-Dimethoxy-6-methyl-6,11,12,14-tetrahydro-6a*H*-1,3-dioxolo[4,5-*h*]isoquino[2,1-*b*]isoquinoline

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.070
wR factor = 0.177
Data-to-parameter ratio = 9.6

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

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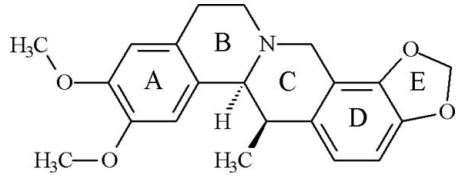
The title compound, $C_{21}H_{23}O_4N$, also known as cavidine, contains four fused six-membered rings and one fused five-membered ring. Both of the rings of the perhydroquinolizine residue adopt a half-chair conformation, while the remaining rings are essentially planar.

Comment

The title compound, (I), also known as cavidine, is an isoquinoline alkaloid isolated from *Colydalis tbalictrifolia* (Yu *et al.*, 1970). As part of an investigation into the conformations of alkaloids, we have synthesized (I) and determined its relative configuration by X-ray crystallographic analysis.



(I)

In (I) (Fig. 1 and Table 1), rings *B* and *C* each adopt a half-chair conformation. The remaining rings (*A*, *D* and *E*) are each essentially planar, with r.m.s deviations of 0.006 (2), 0.002 (2) and 0.004 (2) Å, respectively. The bond lengths and angles are normal (Allen *et al.*, 1987).

Experimental

Compound (I) was prepared according to the procedure of Iwasa *et al.* (1981). A single crystal of (I) was obtained by slow evaporation of an ethanol solution at 283 K over a period of two weeks.

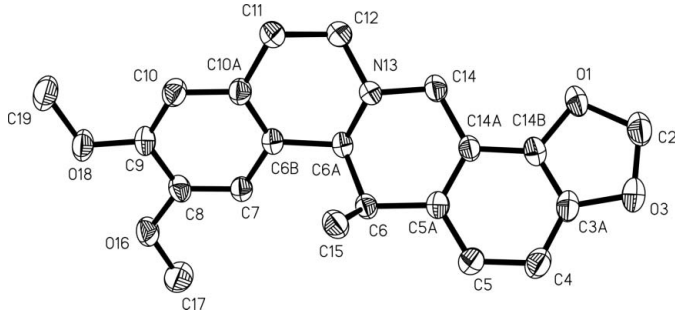


Figure 1
View of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Crystal data

$C_{21}H_{23}NO_4$	$Z = 4$
$M_r = 353.40$	$D_x = 1.293$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.740$ (1) Å	$\mu = 0.09$ mm^{-1}
$b = 8.232$ (1) Å	$T = 295$ (2) K
$c = 28.499$ (2) Å	Block, colourless
$V = 1815.8$ (3) Å^3	0.20 × 0.20 × 0.10 mm

Data collection

MAC DIP 2030K diffractometer	2261 independent reflections
ω scans	1920 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.068$
9552 measured reflections	$\theta_{max} = 27.2°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0652P)^2 + 1.0977P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.070$	$(\Delta/\sigma)_{max} = 0.004$
$wR(F^2) = 0.177$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.26$ e Å^{-3}
2261 reflections	Extinction correction: *SHELXL97*
236 parameters	Extinction coefficient: 0.079 (9)
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C11—C12—N13—C14	−170.2 (4)	C6—C6*A*—N13—C14	72.3 (4)
C11—C12—N13—C6*A*	66.9 (5)	C6*B*—C6*A*—N13—C12	−41.1 (5)
C6*B*—C6*A*—N13—C14	−164.1 (3)	C6—C6*A*—N13—C12	−164.8 (4)

In the absence of significant anomalous scattering, 1241 Friedel pairs were merged. Since there is no chemical evidence to support the assignment of absolute configuration, only the relative configuration was determined. H atoms were included in the riding model approximation, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97*; molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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