

Moschamindole

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Key indicators

Single-crystal X-ray study

$T = 150\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

R factor = 0.046

wR factor = 0.097

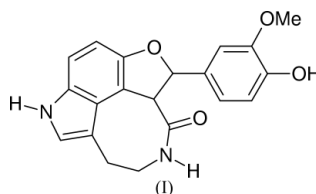
Data-to-parameter ratio = 7.5

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

The solid state structure of moschamindole {systematic name: 10-[2-(4-hydroxy-3-methoxyphenyl)]-1,2,3,4,10,10a-hexahydro-6*H*-furo[2,3,4-*jk*]pyrrolo[4,3,2-*ef*]-3-benzocin-1-one}, $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_4$ is similar to its solution state structure, previously determined by NMR, and evidence of intermolecular hydrogen bonding is now presented.

Comment

Moschamindole, (I), was obtained as one of four indole alkaloids from *Centaurea moschata* and its molecular structure was determined by extensive one-dimensional and two-dimensional NMR spectroscopy (Sarker *et al.*, 1997). We undertook an X-ray study to confirm the structure determined from NMR data and to establish the presence of hydrogen bonding.



Each H atom (H1 and H2) at the donor N-atom positions (N1 and N2) is involved in an intermolecular hydrogen bond (with O4 and O2, respectively). There is also a weak C11—H11...O4 intermolecular hydrogen bond. Three short intramolecular distances are present where the H...A distance 'closes' a five-membered ring. Details of these geometries are shown in Table 2. The absolute stereochemistry of the molecule has not been determined but the two chiral centres, C1 and C2, have the same designation (shown as R in Fig. 1). Here, the torsion angle H1—C1—C2—H2 is $-115(1)^\circ$. The eight-membered ring has a conformation where atoms C2, C4, C5, C6, C7 and C8 lie within 0.05 \AA of a mean plane, with atoms N1 and C3 displaced to the same side of this plane by $1.15(4)$ and $1.28(4)\text{ \AA}$, respectively. The other four rings adopt planar conformations and the dihedral angle between the aryl ring and the dihydrofuran ring is $43.3(1)^\circ$. As a result of ring fusion, valency angle distortions are present around C6, C7 and C8 (Table 1).

Experimental

Moschamindole was obtained from the methanol extract of the seeds of *Centaurea moschata*. Crystals for X-ray work were obtained from a methanol solution.

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Crystal data

$C_{20}H_{18}N_2O_4$
 $M_r = 350.36$
 Monoclinic, $P2_1$
 $a = 4.8724$ (3) Å
 $b = 15.2803$ (9) Å
 $c = 10.6940$ (7) Å
 $\beta = 101.840$ (3)°
 $V = 779.25$ (8) Å³
 $Z = 2$

$D_x = 1.493$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3310
 reflections
 $\theta = 2.9$ – 27.5 °
 $\mu = 0.11$ mm⁻¹
 $T = 150$ (2) K
 Plate, yellow
 $0.40 \times 0.20 \times 0.05$ mm

Data collection

Enraf–Nonius KappaCCD area-
 detector diffractometer
 φ and ω scans to fill Ewald sphere
 5565 measured reflections
 1839 independent reflections
 1272 reflections with $I > 2\sigma(I)$

$R_{int} = 0.080$
 $\theta_{max} = 27.5$ °
 $h = -6 \rightarrow 6$
 $k = -19 \rightarrow 18$
 $l = -12 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.097$
 $S = 0.99$
 1839 reflections
 245 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.007$
 $\Delta\rho_{max} = 0.22$ e Å⁻³
 $\Delta\rho_{min} = -0.28$ e Å⁻³
 Absolute structure: none

Table 1

Selected geometric parameters (Å, °).

O2–C3	1.227 (4)	C7–C12	1.422 (5)
C1–C14	1.509 (5)	C8–C9	1.367 (4)
C6–C13	1.376 (5)		
C13–C6–C7	105.5 (3)	C8–C7–C6	137.5 (3)
C13–C6–C5	122.6 (3)	C9–C8–C7	119.0 (3)
C7–C6–C5	131.9 (3)	C9–C8–C2	108.7 (3)
C12–C7–C8	114.9 (3)	C7–C8–C2	132.2 (3)
C12–C7–C6	107.5 (3)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 ⁱ ···O4 ⁱ	0.79 (4)	2.29 (4)	3.002 (4)	150 (4)
N2–H2 ⁱⁱ ···O2 ⁱⁱ	0.76 (4)	2.15 (4)	2.909 (4)	171 (3)
O4–H4 ⁱⁱⁱ ···O3	1.00 (4)	2.17 (3)	2.631 (5)	106 (2)
C1–H1A ⁱⁱⁱ ···O2	1.00	2.27	2.794 (4)	111
C11–H11 ⁱⁱⁱ ···O4 ⁱⁱⁱ	0.95	2.51	3.367 (4)	150
C15–H15 ⁱⁱⁱ ···O1	0.94	2.37	2.740 (4)	103

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

Friedel pairs were merged in the data set, and the absolute configuration was not determined. H atoms were initially placed in

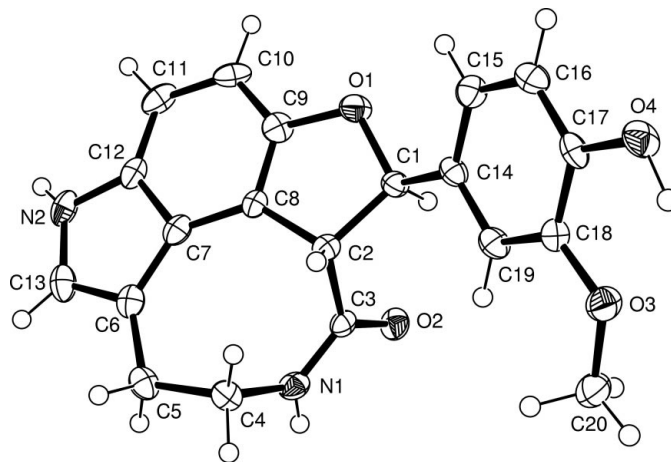


Figure 1

The atomic arrangement in the title molecule. Displacement ellipsoids are shown at the 50% probability level.

calculated positions and thereafter allowed to ride on their attached atoms. In the final cycles of least squares, the coordinates of the H atoms attached to N1, N2 and O4 were freely refined. Each H atom was given an equivalent U_{iso} set at $1.2U_{eq}$ for its attached atom. Hydrogen-bonding geometries were obtained with *PLATON* (Spek, 1998).

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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