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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1023). Services for accessing these data are described at the back of the journal.

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Ismine†

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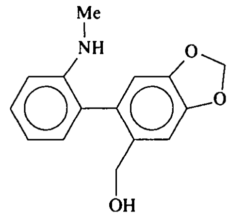
Abstract

The title compound, $C_{15}H_{15}NO_3$, is an alkaloid isolated from several Narcissus species and used to treat a variety of human medical problems. The structure has two molecules in the asymmetric unit which play different roles in the hydrogen-bonding scheme. This produces differences in the geometry of each molecule.

Comment

Ismine, (I), is an alkaloid isolated from whole plants of several Narcissus species (Viladomat *et al.*, 1990, 1992; Codina *et al.*, 1990) and from *Boophane flava* (Viladomat *et al.*, 1995). The Narcissus are Amaryllidacea species endemic to the north of the Iberian Peninsula. Plants of this genus have been used throughout history to treat a variety of human medical problems (Bastida, Viladomat & Codina, 1997).



(I)

The spectroscopic data of ismine are very close to those of 5,6-dihydrobicolorine, also isolated previously from several Narcissus species. In order to confirm its solid-state molecular structure, an X-ray crystallographic study was undertaken.

The X-ray study shows that only the assignment of the aromatic protons H7 and H10 should be interchanged in the ^{1}H NMR taking into account the HMBC (heteronuclear multiple bond correlation) and HMQC (heteronuclear multiple quantum correlation) correlations (Viladomat *et al.*, 1997).

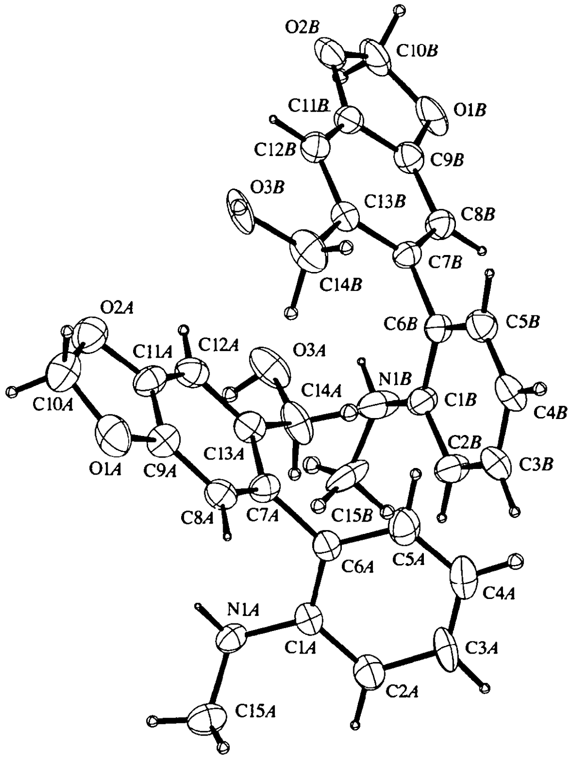


Fig. 1. The molecular structure of (I) showing the two independent molecules with 50% probability displacement ellipsoids.

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† Systematic name: {6-[2-(methylamino)phenyl]-2*H*-1,3-benzodioxol-5-yl}methanol.

There are two non-equivalent molecules in the asymmetric unit, the main difference between them is in the five-membered ring, which is planar in molecule *A*, but in molecule *B*, C10 is the flap of an envelope form. The role of the hydroxyl group in the packing scheme is different for each molecule. The hydroxyl of molecule *A* acts as donor to the hydroxyl group of molecule *B*, while the hydroxyl of molecule *B* acts as donor to the amino group of molecule *A*. This produces differences in the magnitude of the C1—C6—C7—C8 and C12—C13—C14—O3 torsion angles [77.1 (3) and −8.9 (3), respectively, for molecule *A*, and −74.9 (3) and 6.4 (3), respectively, for molecule *B*]. The hydrogen bonds form a complex loop system.

Experimental

Ismine was isolated from Narcissus Asturiensis and crystallized from an *n*-hexane–$CHCl_3$ mixture. The m.p., IR, EIMS, ^1H NMR, ^{13}C NMR are in accordance with reported data (Suau, Gómez & Rico, 1990; Viladomat *et al.*, 1997).

Crystal data

$C_{15}H_{15}NO_3$
$M_r = 257.28$
Triclinic
$P\bar{1}$
$a = 10.232\,(3)$ Å
$b = 10.685\,(6)$ Å
$c = 13.321\,(3)$ Å
$\alpha = 102.06\,(3)^\circ$
$\beta = 103.11\,(2)^\circ$
$\gamma = 108.08\,(3)^\circ$
$V = 1285.5\,(9)$ Å^3
$Z = 4$
$D_x = 1.329$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12$–21°
$\mu = 0.093$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.5 × 0.3 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
7561 measured reflections
7561 independent reflections
4730 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 29.96^\circ$
$h = -14 \rightarrow 13$
$k = -15 \rightarrow 14$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.140$
$S = 0.986$
7561 reflections
464 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0892P)^2 + 0.2160P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.069$ (H30A)
$\Delta\rho_{\max} = 0.257$ e Å^{-3}
$\Delta\rho_{\min} = -0.224$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.017 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1A—C1A	1.405 (2)	N1B—C1B	1.368 (3)
N1A—C15A	1.448 (2)	N1B—C15B	1.438 (3)
O1A—C9A	1.372 (2)	O1B—C9B	1.379 (2)
O1A—C10A	1.424 (3)	O1B—C10B	1.423 (3)
O2A—C11A	1.383 (2)	O2B—C11B	1.384 (2)
O2A—C10A	1.427 (3)	O2B—C10B	1.428 (3)
O3A—C14A	1.407 (3)	O3B—C14B	1.417 (2)
C6A—C7A	1.492 (2)	C6B—C7B	1.502 (2)
C9A—C11A	1.375 (2)	C9B—C11B	1.370 (2)
C1A—N1A—C15A	118.8 (2)	C1B—N1B—C15B	123.7 (2)
C9A—O1A—C10A	106.0 (2)	C9B—O1B—C10B	105.52 (15)
C11A—O2A—C10A	105.94 (15)	C11B—O2B—C10B	104.96 (15)
C2A—C1A—N1A	121.2 (2)	N1B—C1B—C6B	119.53 (15)
N1A—C1A—C6A	119.36 (14)	N1B—C1B—C2B	121.7 (2)
C5A—C6A—C1A	118.5 (2)	C5B—C6B—C1B	118.9 (2)
C5A—C6A—C7A	120.3 (2)	C5B—C6B—C7B	120.37 (15)
O1A—C9A—C11A	110.3 (2)	O1B—C10B—O2B	108.1 (2)
O1A—C10A—O2A	108.1 (2)	C9B—C11B—O2B	110.01 (15)
C9A—C11A—O2A	109.4 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1NA···O2B^{i}	0.889 (19)	2.44 (2)	3.149 (3)	136.9 (16)
N1B—H1NB···O2A^{i}	0.83 (2)	2.39 (2)	3.038 (3)	135.5 (19)
O3B—H3OB···N1A^{ii}	0.94 (4)	1.94 (4)	2.849 (3)	162 (4)
O3A—H3OA···O3B^{i}	0.90 (4)	1.91 (4)	2.799 (3)	168 (4)

Symmetry codes: (i) $-x, -y, 1 - z$; (ii) $1 + x, 1 + y, 1 + z$.

Data collection: *CAD*4-*PC* (Kretschmar, 1996). Cell refinement: *CAD*-4-*PC*. Data reduction: *CFEO* (Solans, 1978). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*3.2/*PC* (Brueggemann & Schmid, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1026). Services for accessing these data are described at the back of the journal.

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