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

N1*A*—C1*A*	1.378 (7)	N3*A*—C11*A*	1.382 (6)
N1*A*—C11*A*	1.314 (6)	N3*A*—C19*A*	1.451 (6)
N2*A*—C1*A*	1.435 (6)	C2*A*—C3*A*	1.489 (7)
N2*A*—C2*A*	1.379 (6)	C3*A*—C4*A*	1.368 (7)
N2*A*—C12*A*	1.445 (6)	C3*A*—C11*A*	1.429 (6)
N3*A*—C10*A*	1.389 (6)	C4*A*—C5*A*	1.418 (7)
C1*A*—N1*A*—C11*A*	118.4 (4)	C2*A*—C3*A*—C11*A*	117.6 (4)
C1*A*—N2*A*—C2*A*	123.9 (4)	C3*A*—C4*A*—C5*A*	120.6 (4)
C1*A*—N2*A*—C12*A*	118.4 (4)	C4*A*—C5*A*—C10*A*	117.7 (4)
C10*A*—N3*A*—C11*A*	122.5 (4)	N3*A*—C10*A*—C5*A*	120.4 (4)
C11*A*—N3*A*—C19*A*	119.2 (4)	N1*A*—C11*A*—N3*A*	118.1 (4)
N1*A*—C1*A*—N2*A*	119.5 (4)	N1*A*—C11*A*—C3*A*	125.2 (4)
N2*A*—C2*A*—C3*A*	114.2 (4)	N3*A*—C11*A*—C3*A*	116.7 (4)
C2*A*—C3*A*—C4*A*	120.3 (4)		

The space group was uniquely determined from the systematic absence: $0k0$ when $k = 2n + 1$. Bijvoet pairs were not averaged. The non-H atoms were refined anisotropically and H atoms were placed in calculated positions but not refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: direct methods *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1036). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 79–81

3-[(*Z*)-Piperidin-1-ylmethylidene]-2,3-dihydro-1,4-benzodioxan-2-one

Concepción Bozzo,[a] Maria D. Pujol,[a] Xavier Solans[b] and Mercè Font-Bardia[b]

[a]*Laboratori de Química Farmacèutica, Universitat de Barcelona, Av. Diagonal s/n, E-08028 Barcelona, Spain, and* [b]*Departamento de Cristalografia, Mineralogia i Dipòsits Minerals, Universitat de Barcelona, Martí i Franqués s/n, E-08028 Barcelona, Spain. E-mail: xavier@natura.geo.ub.es*

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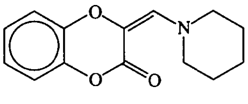
Abstract

The crystal structure of the title compound has been determined in order to obtain the geometry of one isomer of $C_{14}H_{15}NO_3$. The molecule has the unusual feature of a planar dioxane ring and several bond angles are enlarged due to steric hindrance.

Comment

In connection with our investigations into 1,4-benzodioxane chemistry, the reaction between 1,4-benzodioxane-2-carboxylic acid and some amines has been studied (Ruiz *et al.*, 1996), and the title compound, (I), was isolated. Although spectroscopic data show that (I) is clearly different from its isomer 2-piperidinylcarbonyl-1,4-benzodioxane, X-ray diffraction analysis has permitted the determination of the structure and geometry of this new unexpected compound.



(I)

The 1,4-benzodioxo ($C_6H_4O_2$) fragment of the 1,4-benzodioxane moiety has the same geometric values as those observed in a study of 29 structures containing this moiety, obtained from the Cambridge Structural Database (Allen & Kennard, 1993). The O1—C2—C7—O3 torsion angle has a value of −2.2 (3)°; this value varies between 0.0 and 4.6° in the previously mentioned study.

The dioxane ring is planar (Fig. 1), with the largest deviation from the mean plane being 0.018 (2) Å for the C1 atom. This ring is usually not planar, with atoms C1 and C8 being out of the plane defined by the remaining four atoms, but the C_{sp^2} character of C1 in the title structure produces the planarity of the ring.

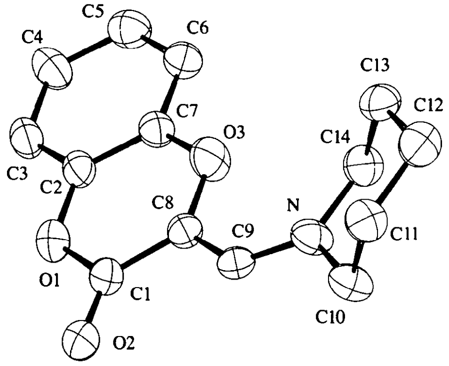


Fig. 1. *ORTEP* (Brueggemann & Schmid, 1990) drawing of (I) showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The largest and smallest bond angles [O3—C8—C1 122.71 (15) and C8—C1—O1 116.92 (15)°, respectively] have also been observed in compounds with the 1,4-benzodioxan-2-yl moiety [average values 122.2 (5) and 114.8 (5)°, respectively; Leger, Colleter & Carpy, 1983; Dzvinchuk *et al.*, 1990].

The piperidine ring has the typical chair form, with atoms C11 and C14 0.655 (3) Å out of the plane defined by the remaining four atoms. Atoms O3, C8, C1, C9 and N are in a plane. The steric hindrance between the piperidine group and O3 produces an increase of the C8—C9—N bond angle to 132.4 (2)° and a shortening of the H9···H10*A* contact distance to 2.07 (4) Å.

Experimental

Single crystals of the title compound were grown from a methylene chloride solution at 269 K.

Crystal data

$C_{14}H_{15}NO_3$
$M_r = 245.27$
Monoclinic
$P2_1/a$
$a = 8.994 (3)$ Å
$b = 16.534 (4)$ Å
$c = 9.428 (2)$ Å
$\beta = 118.11 (3)°$
$V = 1236.6 (6)$ Å^3
$Z = 4$
$D_x = 1.317$ 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
0.4 × 0.2 × 0.2 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: none
3741 measured reflections
3591 independent reflections
2230 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.040$
$\theta_{\rm max} = 30.11°$
$h = -12 \rightarrow 11$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.312$
$S = 0.985$
3591 reflections
202 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1659P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$

$\Delta\rho_{\rm max} = 0.312$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.375$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.000 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O2—C1	1.189 (2)	C7—O3	1.377 (2)
C1—O1	1.391 (2)	O3—C8	1.384 (2)
C1—C8	1.440 (2)	C8—C9	1.372 (2)
O1—C2	1.375 (2)	C9—N	1.339 (2)
O2—C1—O1	116.3 (2)	C7—O3—C8	117.52 (13)
O2—C1—C8	126.8 (2)	C9—C8—O3	119.60 (14)
O1—C1—C8	116.92 (15)	C9—C8—C1	117.70 (15)
C2—O1—C1	119.81 (13)	O3—C8—C1	122.71 (15)
O1—C2—C7	121.93 (15)	N—C9—C8	132.4 (2)
O1—C2—C3	118.5 (2)	C9—N—C10	120.4 (2)
C6—C7—O3	117.87 (15)	C9—N—C14	125.74 (15)
O3—C7—C2	121.02 (15)		

The positions of nine H atoms were determined from a difference map and the positions of the remaining six H atoms (H4, H6, H11, H11*A*, H14 and H14*A*) were computed. The isotropic displacement parameters and coordinates were refined for the first nine H atoms. An overall isotropic *U* and constrained atomic coordinates (riding model) were refined for the remaining six H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. 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* (Brueggemann & Schmid, 1990). Software used to prepare material for publication: *CIFTAB* (Sheldrick, 1994).

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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†

Francesc Viladomat,[a] Carles Codina,[a] Jaume Bastida,[a] Xavier Solans[b] and Mercè Font-Bardia[b]

[a]*Departamento de Productes Naturals, Facultat de Farmacia, Universidad de Barcelona, E-08028 Barcelona, Spain, and* [b]*Departamento de Cristalografía, Mineralogía y Depósitos Minerales, Universidad de Barcelona, Marti i Franqués s/n, E-08028 Barcelona, Spain. E-mail: xavier@natura.geo.ub.es*

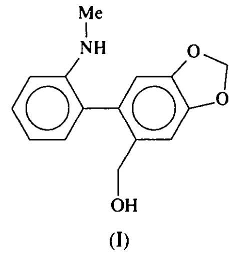
(*Received 4 July 1997; accepted 15 September 1997*)

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).

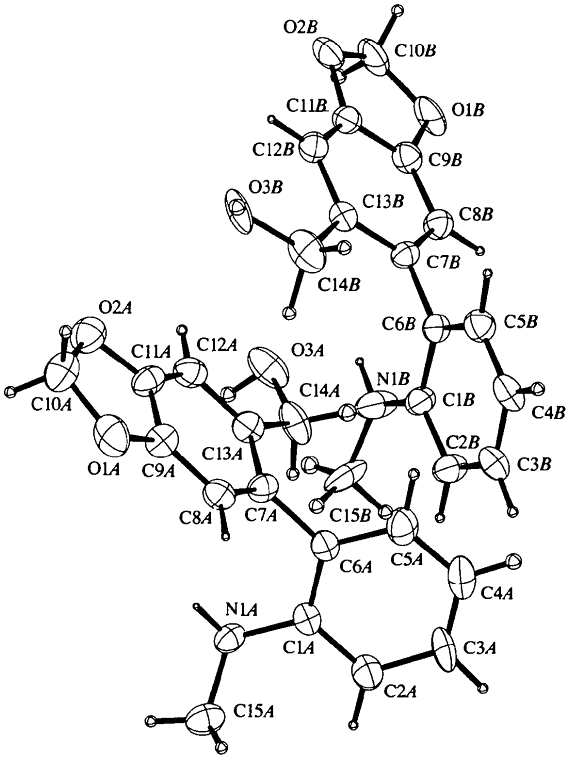


Fig. 1. The molecular structure of (I) showing the two independent molecules with 50% probability displacement ellipsoids.

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

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