Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6,7-Dimethoxy-2-methyl-3,4-dihydroisoquinolin-1(2*H*)-one

Ikram M. Said, Norizan A. A. Hamid, Jalifah Latif, Laily B. Din and Bohari M. Yamin*

School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail: bohari_bob@yahoo.com

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.145
Data-to-parameter ratio = 15.8

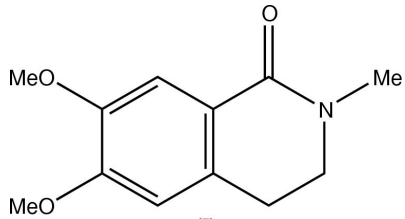
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{12}H_{15}NO_3$, the isoquinoline moiety is essentially planar, except for the N and adjacent Csp^3 atoms, which deviate from the mean plane by 0.198 (2) and 0.686 (2) Å, respectively. The bond length between the N and carbonyl C atoms [1.353 (3) Å] is close to a double-bond value, indicating electron delocalization. No significant intermolecular interactions are observed.

Received 10 February 2005
Accepted 22 February 2005
Online 4 March 2005

Comment

Tetrahydroisoquinoline derivatives, obtained either from plants or by synthesis, are of great interest due to their biological and pharmacological properties (Brzezinska, 1994; Anderson *et al.*, 1998; Loesel *et al.*, 1987).



(I)

The title compound, (I) (Fig. 1), is an isoquinoline analogue of 6,7-dimethoxy-1-phenyl-1,2,3,4-tetrahydroisoqinoline, (II) (Olszak *et al.*, 1996), and 2-chlorobenzoxazolo[3,2-*b*]isoquinoline-6-one, (III) (Ravishankar *et al.*, 2001). Structural studies have shown that the degree of planarity of the isoquinoline moiety is affected by the presence of various substituents. For example, the molecule of (III) is essentially

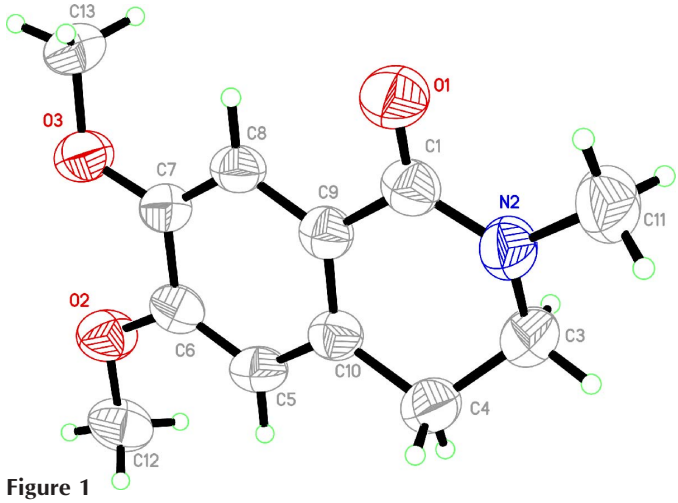


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids and H atoms drawn as spheres of abitrary radius.

planar (Ravishankar *et al.*, 2001), whereas in a dione system such as *N*-(2-chlorobenzyl)-1,2,3,4-tetrahydroisoquinoline-1,3-dione, the isoquinoline moiety is slightly folded about the fused C9—C10 bond (Pandi *et al.*, 2002). In contrast to (II), where the saturated fragment has a twist conformation, in (I) it has a half-chair or envelope conformation [Q = 0.430 (2) Å, θ = 116.8 (3) and φ_2 = 100.1 (3); Cremer & Pople, 1975], with C1—N2—C3—C4 and N2—C3—C4—C10 torsion angles of −42.8 (3) and 49.5 (2)°, respectively. The C1/C4/C5/C6/C7/C8/C9/C10 fragment of the isoquinoline moiety is essentially planar, with a maximum deviation from the mean plane of 0.026 (2) Å for atom C6. Atoms N2 and C3 are displaced from that plane by 0.198 (2) and 0.686 (2) Å, respectively. As a result, the N2—C1 bond length [1.353 (3) Å] is shorter than those in (II) [1.461 (3) Å] and in (III) [1.408 (4) Å], indicating electron delocalization in (I) involving the O1—C1 and C1—N2 bonds. All other bond lengths and angles in (I) (Table 1) are in normal ranges (Allen *et al.*, 1987) and agree with those in other isoquinoline compounds. In contrast to the crystal structures of (II) and (III), no significant intermolecular interactions are observed in (I).

Experimental

The roots of *Alseodaphne perakensis* were collected from Sungkai, Perak, and a voucher specimen (ALM 5735) was deposited at the UKMB herbarium of Universtiti Kebangsaan Malaysia. The roots were air-dried, ground and soaked in MeOH for 48 h. The resulting extract was filtered and concentrated under reduced pressure to give 108.3 g of crude extract. The crude extract was acidified with 5% H_2SO_4 and filtered. The acid extract was then basified with 5% Na_2CO_3 until pH 8–9 was attained and extracted with $CHCl_3$ to obtain the crude alkaloid. The crude alkaloid (15.8 g) was chromatographed using liquid chromatography (VLC) and eluted with $CHCl_3$ and increasing amounts of MeOH. The VLC of the crude alkaloid yielded four fractions, denoted 01–04. Fraction 03 was further chromatographed using VLC with $CHCl_3$–MeOH (7:3 *v*/*v*) as eluents to yield *N*-methylcorydaldine. Colourless crystals were obtained from the chromatographed solution after 3 d upon evaporation at room temperature (m.p. 398–400 K). EIMS *m*/*z* (% int.): [M^+] 221 (77), 178 (82), 150 (100), 135 (15), 107 (10), 92 (13). ^{1}H NMR: δ 2.88 (2H, *t*, *J* = 6.9 Hz, H-4), 3.08 (3H, *s*, NCH_3), 3.49 (2H, *t*, *J* = 6.9 Hz, H-3), 3.86 (3H, *s*, OCH_3-7), 3.87 (3H, *s*, OCH_3-6), 6.58 (1H, *s*, H-5),7.55 (1H, *s*, H-8), ^{13}C NMR: δ 27.5 (C4), 35.2 (C12), 48.4 (C3), *56.07 (C12), *56.14 (C13), 109.3 (C5), 110.5 (C8), 121.9 (C9), 131.6 (C10), 148.0 (C7), 152.0 (C6), 165.0 (C1) (* values may be interchanged).

Crystal data

$C_{12}H_{15}NO_3$
M_r = 221.25
Monoclinic, $P2_1/c$
a = 9.7846 (17) Å
b = 16.865 (3) Å
c = 6.8914 (12) Å
β = 103.934 (3)°
V = 1103.7 (3) Å^3
Z = 4
D_x = 1.331 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 912 reflections
θ = 2.1–26.5°
μ = 0.10 mm^{-1}
T = 273 (2) K
Plate, colourless
0.31 × 0.11 × 0.06 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.971, T_{max} = 0.994
6118 measured reflections
2285 independent reflections
1673 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 26.5°
$h = -10 \rightarrow 12$
$k = -21 \rightarrow 20$
$l = -6 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.145
S = 1.09
2285 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.2375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.227 (2)	N2—C11	1.451 (3)
O2—C6	1.359 (2)	N2—C3	1.458 (3)
O3—C7	1.364 (2)	C1—C9	1.486 (3)
N2—C1	1.353 (3)	C3—C4	1.497 (3)
C11—N2—C1—O1	−4.7 (3)	C3—N2—C1—C9	11.0 (3)
C3—N2—C1—O1	−170.6 (2)		

After the location of the H atoms in a difference map, their positions were idealized. The H atoms were allowed to ride on the parent C atoms, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 and 1.2U_{eq}(C) for CH groups.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government for the research grant IRPA No. 09-02-02-0163.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Anderson, W. K., Heider, A. R., Raju, N. & Yutch, J. A. (1998). *J. Med. Chem.* **31**, 2097–2102.
Brzezinska, E. (1994). *Acta Pol. Pharm.* (*Drug Res*), **51**, 137–141.
Cremer, D. & Pople, I. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Loesel, W., Roos, O. & Schnorrenberger, G. (1987). US Patent No. 4 694 085.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Olszak, T. A., Stepien, A., Grabowski, M. J. & Brzezinska, E. (1996). *Acta Cryst.* C**52**, 1038–1040.
Pandi, A. S., Rajakannan, V., Velmurugan, D., Parvez, M., Kim, M.-J., Senthilvelan, A. & Rao, S. N. (2002). *Acta Cryst.* C**58**, o164–o167.
Ravishankar, T., Chinnakali, K., Senthilvelan, A., Fun, H.-K., Ramakrishnan, V. T., Chantrapromma, S., Razak, I. A. & Usman, A. (2001). *Acta Cryst.* E**57**, o1209–o1212.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.