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2,3,3a,4,5,9b-Hexahydro-4-phenylfuro-[3,2-*c*]quinoline

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.117
Data-to-parameter ratio = 13.3

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

The title compound, $C_{17}H_{17}NO$, is one of the diastereoisomers formed as result of a Diels–Alder reaction catalysed by zirconium tetrachloride. The compound crystallizes with two crystallographically independent molecules in the asymmetric unit. The *N*-heterocyclic ring adopts a half-chair conformation for both molecules. Only one molecule forms a hydrogen bond; for the other, no acceptor for the NH group is found.

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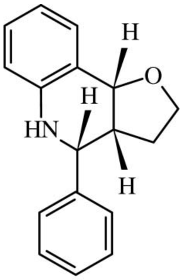
Imino Diels–Alder adducts. XI

Comment

The pharmacological properties of quinoline alkaloids have been investigated to a limited degree, and thus evidence of antibacterial, antifungal and antiviral (HIV) activity has been observed (Grundon, 1998; Michael, 1998). Several quinoline alkaloids have also been found to show cytotoxic, phototoxic and mutagenic activity and to form cycloadducts with DNA (McCormick *et al.*, 1996). Tetrahydroquinoline derivatives are an important class of natural products and exhibit various biological activities (Johnson *et al.*, 1989; Carling *et al.*, 1993), such as psychotropic (Nesterova *et al.*, 1995), anti-allergenic (Yamada *et al.*, 1992) and anti-inflammatory (Khodzhaeva & Bessonova, 1983). In addition, furoquinoline alkaloids, *viz*. dictamnine, *g*-fagarine and skimmianine, exhibit photomutagenic properties (Schimmer & Kuhne, 1990). Recently, research has focused on syntheses of these compounds by different methods (Ma *et al.*, 1999; Mahesh, Makesh & Perumal, 2004; Mahesh, Venkateswar Reddy *et al.*, 2004). For the synthesis of furoquinolines, an imino Diels–Alder reaction using zirconium tetrachloride ($ZrCl_4$) as a catalyst is probably a successful synthetic tool. We report here the structure of a diastereoisomer, namely 2,3,3a,4,5,9b-hexahydro-4-phenylfuro[3,2-*c*]quinoline, (I), as part of our ongoing structural studies of this series of compounds (Ravikumar *et al.*, 2005*a*,*b*,*c*).



(I)

In all essential details, the molecular geometry (Table 1) is in good agreement with comparable structures (Ma *et al.*, 1999; Batey *et al.*, 2001; Hoemann *et al.*, 2002). The asymmetric unit contains two crystallographically independent molecules (*A* and *B*) (Fig. 1). Bond distances and angles are similar for both independent molecules: the largest differences are 0.033 Å for the bond distance C5—O1 and 1.81° for the angle C5—O1—C4. Generally, these diasteromers differ according to the stereochemistry (*cis*/*trans*) of the ring junction H atoms. In the present case, the connection of the two rings is *cis*.

The *N*-heterocyclic ring adopts a half-chair conformation in both molecules, with asymmetry parameters (Nardelli, 1983) ΔC_s(C3—C9) = 0.028 (2) Å in *A* and 0.002 (1) Å in *B*. Atoms C7 and C8 are displaced by 0.237 (2) and −0.414 (2) Å, respectively, in *A*, and 0.314 (2) and −0.332 (2) Å in *B* from the mean plane defined by atoms N1/C3/C4/C9.

The furan ring adopts an envelope conformation [asymmetry parameter ΔC_s(C7*A*) = 0.016 (1) Å] in molecule *A*. In molecule *B*, it also adopts an envelope conformation [asymmetry parameter ΔC_s(C4*A*) = 0.088 (1) Å].

Only one of the two molecules forms an NH hydrogen bond. The NH group of molecule *A* does not act as a donor for an NH hydrogen bond. A similar feature has been reported for the structures of alloxan (Beyer *et al.*, 2001; Coombes *et al.*, 1997), furoquinoline (Ravikumar *et al.*, 2004) and pyranoquinoline (Ravikumar *et al.*, 2005*d*).

Experimental

To a solution of *N*-benzylideneaniline (5.5 mmol) in dichloromethane (5 ml) at room temperature was added 2,3-dihydrofuran (5.5 mmol), $ZrCl_4$ (10 mol%). The solution was stirred for 90 min. The completed reaction was quenched with water and the crude product was purified by column chromatography using 2% ethyl acetate and hexane to yield the title compound. Crystals for X-ray study were obtained by recrystallization from a solution in a mixture of methanol and water (3:1).

Crystal data

$C_{17}H_{17}NO$
$M_r = 251.32$
Triclinic, $P\bar{1}$
$a = 9.3760$ (6) Å
$b = 9.9065$ (6) Å
$c = 16.014$ (1) Å
$\alpha = 96.954$ (1)°
$\beta = 102.005$ (1)°
$\gamma = 110.693$ (1)°
$V = 1329.92$ (14) Å^3

$Z = 4$
$D_x = 1.255$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6873 reflections
$\theta = 2.3$–28.0°
$\mu = 0.08$ mm^{-1}
$T = 273$ (2) K
Block, yellow
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
12802 measured reflections
4659 independent reflections

4015 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\rm max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.117$
$S = 1.04$
4659 reflections
351 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.2842P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}

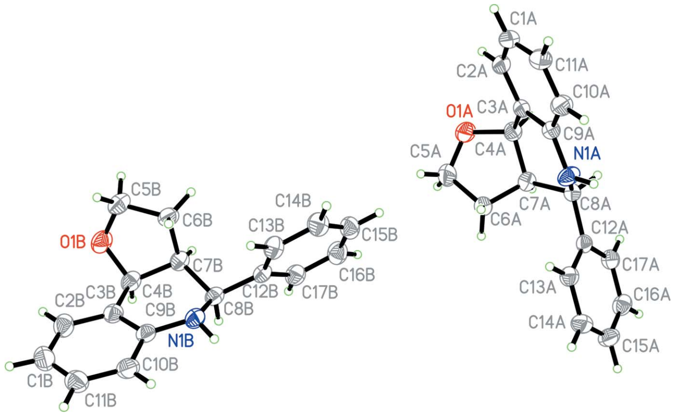


Figure 1
The asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

O1*A*—C5*A*	1.389 (2)	O1*B*—C5*B*	1.425 (2)
O1*A*—C4*A*	1.4324 (19)	O1*B*—C4*B*	1.4294 (19)
N1*A*—C9*A*	1.3860 (19)	N1*B*—C9*B*	1.386 (2)
N1*A*—C8*A*	1.456 (2)	N1*B*—C8*B*	1.457 (2)
C5*A*—O1*A*—C4*A*	109.95 (13)	C5*B*—O1*B*—C4*B*	107.84 (12)
C9*A*—N1*A*—C8*A*	119.50 (13)	C9*B*—N1*B*—C8*B*	120.02 (13)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*B*—H1*BN*···O1*A*$^{\rm i}$	0.844 (19)	2.23 (2)	3.0617 (18)	167.2 (16)

Symmetry code: (i) $x, y+1, z$.

H atoms attached to the quinoline N atoms were located in a difference density map and refined isotropically. All other H atoms were positioned geometrically and treated as riding, with C—H distances in the range 0.93–0.98 Å; $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2Ueq(C) for other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990) and *QMOL* (Gans & Shalloway, 2001); software used to prepare material for publication: *SHELXL97*.

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