Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(4-Nitrophenyl)-2,3,3a,4,5,9b-hexahydrofuro[3,2-*c*]quinoline

K. Ravikumar,[a]* B. Sridhar,[a] M. Mahesh[b] and V. V. Narayana Reddy[b]

[a]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [b]Organic Chemistry Division–II, Indian Institute of Chemical Technology, Hyderabad 500 007, India

Correspondence e-mail: ravikumar_iict@yahoo.co.in

Received 29 March 2005
Accepted 15 April 2005
Online 23 April 2005

Imino Diels–Alder adducts. VI.

Key indicators

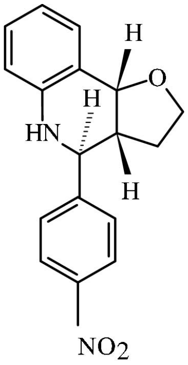
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.163
Data-to-parameter ratio = 16.3

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

The title compound, $C_{17}H_{16}N_2O_3$, is one of the diastereoisomers formed as the result of the Diels–Alder reaction of 1-(4-nitrophenyl)methylidene]-*N*-phenylamine and dihydrofuran catalyzed by zirconium tetrachloride. The furan ring adopts an envelope conformation, while the *N*-heterocyclic ring prefers a sofa conformation. Molecules are linked into centrosymmetric dimers by N—H···O hydrogen-bond interactions.

Comment

Furoquinoline derivatives have been studied extensively for a range of biological applications, such as psychotropic (Nesterova *et al.*, 1995), anti-allergic (Yamada *et al.*, 1992) and anti-inflammatory (Faber *et al.*, 1984) applications. We have recently synthesized and purified pyrano- and furoquinolines using the imino Diels–Alder reaction with $ZrCl_4$ as a green catalyst (Mahesh *et al.*, 2004). The structure of the title compound, (I), is reported here as part of our ongoing structural studies on this series of compounds (Ravikumar *et al.*, 2004, 2005).



(I)

A view of (I) is shown in Fig. 1. In all essential details, the molecular geometry (Table 1) is in good agreement with relevant structures found in the literature (Ma *et al.*, 1999; Batey *et al.*, 2001; Hoemann *et al.*, 2002). Generally, these diastereomers differ according to the stereochemistry (*cis*/*trans*) of the ring junction H atoms and the orientation (*endo*/*exo*) of the furan ring. The H7—C7—C8—H8 torsion angle of −174.4° indicates the *trans* (*exo*) configuration and is in accordance with the coupling constant *J* = 13.4 Hz. Similarly, the H4—C4—C7—H7 torsion angle of 35.5° indicates the *cis* arrangement of the H atoms at the furan ring junction.

The values of the C8—C7—C4—C3 [34.6 (2)°] and C8—N1—C9—C3 [−26.8 (3)°] torsion angles are due to the strain exerted during the cycloaddition process in forming the *N*-

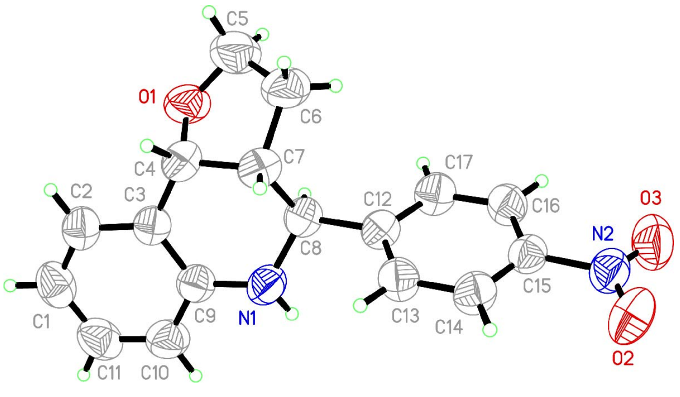


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

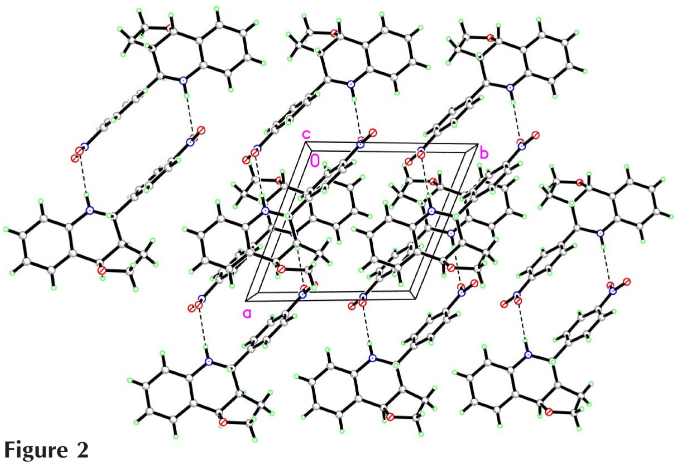


Figure 2
Packing diagram, viewed down the *c* axis, showing the dimers. Dashed lines indicate N—H⋯O hydrogen bonds.

heterocyclic ring. The *N*-heterocyclic ring of the quinoline moiety adopts a sofa conformation [asymmetry parameter ΔC_2(C3—C9) = 0.064 (1); Nardelli, 1983], with atom C8 displaced by 0.626 (2) Å from the mean plane defined by the atoms N1, C9, C3, C4 and C7.

The furan ring is oriented perpendicular to the quinoline ring, with atom O1 displaced by −1.374 (1) Å from the N1/C9/C3/C4/C7 least-squares plane. The furan ring adopts an envelope conformation [asymmetry parameter ΔC_s(C4) = 0.008 (1)].

The absence of steric effects between the NO_2 group and the quinoline moiety accounts for the small dihedral angle of 6.9 (2)° between the nitro group and its attached ring. The phenyl substituent at C8 is rotated about the C8—C12 bond by 74.2 (1)°.

Intermolecular hydrogen bonds between the NO_2 and quinoline groups lead to the formation of dimers between molecules related by an inversion center (Table 2), generating a characteristic $R_2^2(18)$-type motif (Bernstein *et al.*, 1995).

Experimental

To a solution of 1-(4-nitrophenyl)methylidene-*N*-phenylamine (5.5 mmol) in dichloromethane (5 ml) at room temperature were added 2,3-dihydrofuran (5.5 mmol) and ZrCl4 (10 mol%) with stirring for a period of 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 suitable for X-ray study were obtained by recrystallization from a methanol–water (3:1) solution.

Crystal data

$C_{17}H_{16}N_2O_3$
M_r = 296.32
Triclinic, $P\overline{1}$
a = 9.1090 (10) Å
b = 9.2626 (10) Å
c = 9.7553 (11) Å
α = 103.411 (2)°
β = 102.791 (2)°
γ = 107.081 (2)°
V = 727.28 (14) Å^3
Z = 2
D_x = 1.353 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2986 reflections
θ = 2.4–27.8°
μ = 0.09 mm^{-1}
T = 273 (2) K
Block, yellow
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
8354 measured reflections
3308 independent reflections
2549 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017
$\theta_{\rm max}$ = 28.0°
h = −11 → 12
k = −12 → 12
l = −12 → 12

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.163
S = 1.03
3308 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2 + 0.1431P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ < 0.001
$\Delta\rho_{\rm max}$ = 0.39 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—N2	1.221 (2)	N1—C8	1.453 (2)
O3—N2	1.224 (2)	N2—C15	1.472 (2)
N1—C9	1.389 (2)		
C9—N1—C8	118.39 (14)	O2—N2—C15	118.52 (16)
O2—N2—O3	123.25 (17)	O3—N2—C15	118.21 (16)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1*N*⋯O2$^{\rm i}$	0.84 (2)	2.34 (2)	3.164 (2)	171 (2)

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

The H atom attached to the quinoline N atom was located in a difference density map and refined isotropically. All other H atoms were positioned geometrically and treated as riding atoms, 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 1.2$U_{\rm eq}$(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:

o1420 Ravikumar *et al.* • $C_{17}H_{16}N_2O_3$ *Acta Cryst.* (2005). E**61**, o1419–o1421

SHELXTL/PC (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

MM thanks the Council of Scientific and Industrial Research (CSIR), India, for the award of a Senior Research Fellowship.

References

Batey, R. A., Powell, D. A., Acton, A. & Lough, A. J. (2001). *Tetrahedron Lett.* **42**, 7935–7939.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2001). *SMART* (Version 5.625) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.

Faber, K., Stueckler, H. & Kappe, T. J. (1984). *J. Heterocycl. Chem.* **21**, 1177–1181.

Hoemann, M. Z., Xie, R. L., Rossi, R. F., Meyer, S. & Sidhu, A. (2002). *Bioorg. Med. Chem. Lett.* **12**, 129–132.

Ma, Y., Quian, C., Xie, M. H. & Sun, J. (1999). *J. Org. Chem.* **64**, 6462–6467.

Mahesh, M., Venkateshwar Reddy, Ch., Srinivasa Reddy, K., Raju, P. V. K. & Narayana Reddy, V. V. (2004). *Synth. Commun.* **34**, 4089–4104.

Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.

Nesterova, I., Alekseeva, L. M., Andreeva, L. M., Andreeva, N. I., Golovira, S. M. & Granic, V. G. (1995). *Khim. Farm. Zh.* **29**, 31–34.

Ravikumar, K., Mahesh, M. & Narayana Reddy, V. V. (2004). *Acta Cryst.* C**60**, o887–o889.

Ravikumar, K., Sridhar, B., Mahesh, M. & Narayana Reddy, V. V. (2005). *Acta Cryst.* E**61**, o461–o463.

Sheldrick, G. M. (1990). *SHELXTL/PC*. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Yamada, N., Kadowaki, S., Takashi, K. & Umezu, K. (1992). *Biochem. Pharmacol.* **44**, 1211–1215.

Acta Cryst. (2005). E**61**, o1419–o1421 Ravikumar *et al.* • $C_{17}H_{16}N_2O_3$ **o1421**