Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A diastereoisomer of furo[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

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.150
Data-to-parameter ratio = 18.4

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

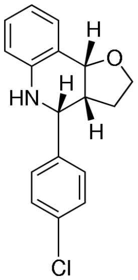
Received 13 December 2004
Accepted 17 December 2004
Online 24 December 2004

Imine Diels–Alder adducts. Part IV.

The title compound, 4-(4-chlorophenyl)-2,3,3a,4,5,9b-hexahydrofuro[3,2-*c*]quinoline, $C_{17}H_{16}ClNO$, was obtained as one of the diastereoisomers from the imino Diels–Alder reaction of an imine and dihydrofuran. The molecule is a *cis* (*endo*) cycloadduct isomer. The tetrahydrofuran ring adopts an envelope conformation, while the *N*-heterocyclic ring is in a sofa conformation. Intermolecular N—H···O hydrogen bonds lead to chains of molecules running along the *a* axis.

Comment

Diels–Alder adducts from the reaction of imines derived from aromatic amines and aldehydes with dihydrofuran/pyran have been used as a central heterocyclic core for a variety of alkaloids, such as skimmianine and balfouridine (Akhmed Khodzhaeva & Bessonova, 1983; Jurd & Wong, 1981). We have recently reported the pyrano- and furoquinolines synthesized using the imino Diels–Alder reaction with $ZrCl_4$ as a potential environmentally benign catalyst (Mahesh *et al.*, 2004). The structure of the title compound, (I), is reported here as an early result in our study of this series of compounds.



(I)

A perspective view of (I) is shown in Fig. 1. In all essential details, the geometry of the molecule (Table 1), in terms of

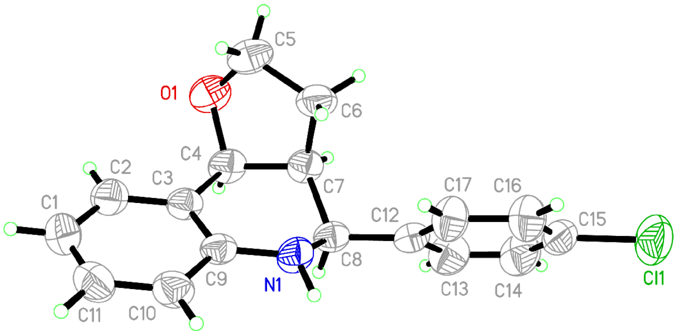


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

interatomic distances and angles, is in good agreement with relevant structures found in the literature (Clegg *et al.*, 1999). The orientations of the junction atoms H7 and H8 differentiate the diastereoisomers for *cis* or *trans* configurations. The H7—C7—C8—H8 torsion angle of −60.6 (2)° indicates the *cis* (*endo*) arrangement of the ring junction H atoms and is in accordance with the coupling constant J = 8.1 Hz. Similarly, the H4—C4—C7—H7 torsion angle of 38.3 (2)° also indicates the *cis* arrangement of the tetrahydrofuran-ring-junction H atoms.

The substitutions of the quinoline ring produce a considerable twist about the C8···C3 vector. This twist can be seen from the C8—C7—C4—C3 and C8—N1—C9—C3 torsion angles of 39.7 (2) and −22.7 (2)°, respectively. The *N*-heterocyclic ring of the quinoline moiety is in a sofa conformation [asymmetry parameter ΔC_2(C9—C3) = 0.030 (1) (Nardelli, 1983)], with atom C8 displaced by 0.600 (2) Å from the N1/C9/C3/C4/C7 basal plane.

The tetrahydrofuran ring adopts an envelope conformation, with an asymmetry parameter ΔC_s(C4) = 0.012 (1). Atom O1 is displaced by 0.503 (2) Å from the N1/C4/C11/C10 least-squares plane. The N1—C8—C12—C17 torsion angle between these two rings is −32.0 (2)°.

Intermolecular N—H···O hydrogen bonding is observed between the N atom of the quinoline ring system and the O atom of the tetrahydrofuran ring, forming chains along the *a* axis (Fig. 2). It is interesting to note that the Cl atom present in the structure is not involved in any interactions. A possible intramolecular C—H···N interaction is also observed in the structure (Table 2).

Experimental

To a solution of *N*-[(1*E*)-(4-chlorophenyl)methylene]-*N*-phenylamine (5.5 mmol) in dichloromethane (5 ml) at room temperature were added 2,3-dihydrofuran (5.5 mmol) and $ZrCl_4$ (10 mol%), and the mixture 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 of (I) for X-ray study were obtained by recrystallization from a methanol–water (3:1) solution.

Crystal data

$C_{17}H_{16}ClNO$
M_r = 285.76
Orthorhombic, *Pbca*
a = 14.1068 (9) Å
b = 9.5526 (6) Å
c = 21.1298 (13) Å
V = 2847.4 (3) Å^3
Z = 8
D_x = 1.333 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 5262 reflections
θ = 2.4–27.9°
μ = 0.26 mm^{-1}
T = 273 (2) K
Block, colourless
0.18 × 0.15 × 0.13 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
30 760 measured reflections
3412 independent reflections

3010 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.023
$\theta_{\rm max}$ = 28.0°
$h = -18 \rightarrow 18$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 27$

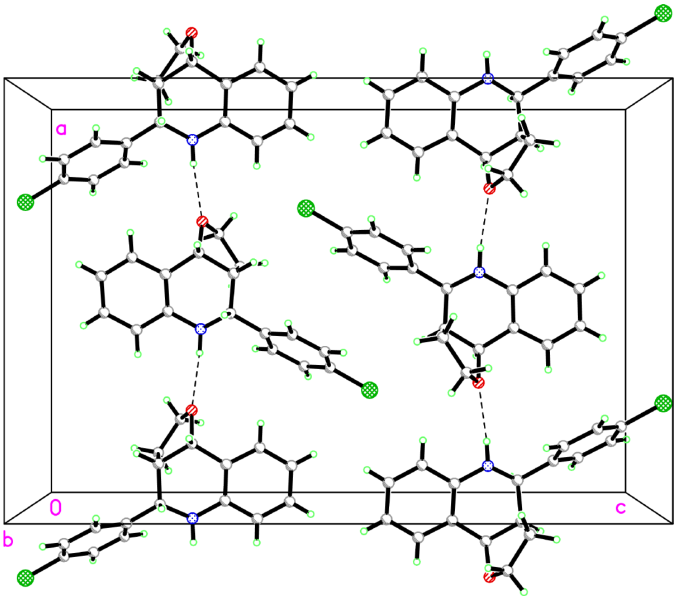


Figure 2
A packing diagram for (I), viewed down the *b* axis. Dashed lines indicate the N—H···O hydrogen bonds.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.150
S = 1.14
3412 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 0.9682P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.44 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.24 e Å^{-3}
Extinction correction: none

Table 1
Selected bond lengths (Å).

Cl1—C15	1.7450 (19)	N1—C9	1.387 (2)
O1—C5	1.426 (3)	N1—C8	1.452 (2)
O1—C4	1.438 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1$^{\rm i}$	0.89 (3)	2.19 (3)	3.066 (2)	168 (2)
C6—H6A···N1	0.97	2.60	2.988 (2)	105

Symmetry code: (i) $x+\frac{1}{2}, y, -z+\frac{3}{2}$.

The H atom on N1 was located in a difference density map and refined freely. All other H atoms were positioned geometrically and treated as riding atoms, with C—H distances in the range 0.93–0.98 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H and 1.2$U_{\rm eq}$(C) for other H.

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); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

o196 Ravikumar *et al.* • $C_{17}H_{16}ClNO$ *Acta Cryst.* (2005). E**61**, o195–o197

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

References

Akhmed Khodzhaeva, K. S. & Bessonova, I. A. (1983). *Chem. Abstr.* **98**, 83727q.

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

Clegg, W., Elsegood, M. R. J., Stanforth, S. P., Hedley, K. A., Stanley, A. L., Raper, E. S. & Creighton, J. R. (1999). *Acta Cryst.* C**55**, 2163–2167.

Jurd, L. & Wong, R. V. (1981). *Aust. J. Chem.* **34**, 1625–1632.

Mahesh, M., Venkateswar 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.

Nardelli, M.(1995). *J. Appl. Cryst.* **28**, 659–1142.

Sheldrick, G. M. (1990). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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

Acta Cryst. (2005). E**61**, o195–o197 Ravikumar *et al.* • $C_{17}H_{16}ClNO$ **o197**