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A diastereoisomer of furo[3,2-*c*]quinoline

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Imine Diels–Alder adducts. III.

Key indicators

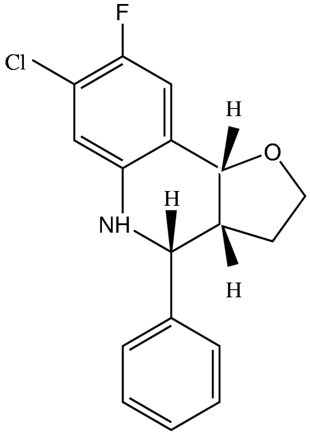
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.005 Å
R factor = 0.066
wR factor = 0.154
Data-to-parameter ratio = 13.7

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

The title compound, 7-chloro-8-fluoro-4-phenyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-*c*]quinoline, $C_{17}H_{15}ClFNO$, was obtained from the imine Diels–Alder reaction of an *N*-benzylideneaniline and 2,3-dihydrofuran catalyzed by $ZrCl_4$. An isomer that crystallizes in the centrosymmetric monoclinic space group *C*2/*c* is presented. The furan ring is in a twist conformation, while the pyridine ring is in a sofa conformation. An intermolecular hydrogen bond between the pyridine NH group and the O atom of the furan ring in a neighboring molecule forms chains along the *b* axis.

Comment

The medicinal importance, such as the anti-allergic (Yamada *et al.*, 1992), psychotropic (Nesterova *et al.*, 1995) and anti-inflammatory (Faber *et al.*, 1984) properties, of pyrano- and furoquinoline compounds have attracted much attention in recent times. We have synthesized and characterized diastereoisomers of disubstituted pyrano- and furoquinolines by the application of the imine Diels–Alder reaction, using $ZrCl_4$ as a new catalyst (Mahesh *et al.*, 2004). Recently, we reported the structures of two diastereoisomers (Ravikumar *et al.*, 2004). In this paper, we report the crystal structure of another diastereoisomer, 7-chloro-8-fluoro-4-phenyl-2,3,3a,4,5,9b-hexahydrofuro[3,2-*c*]quinoline.



(I)

A perspective view of (I) is shown in Fig. 1. Selected interatomic distances are listed in Table 1. In all essential details the geometry of the molecule is similar to other relevant structures found in the literature.

The H7—C7—C8—H8 torsion angle of −61.1° indicates the *cis* arrangement of the H7 and H8 atoms and is in accordance with the coupling constant *J* = 5.2 Hz. The substitution of the

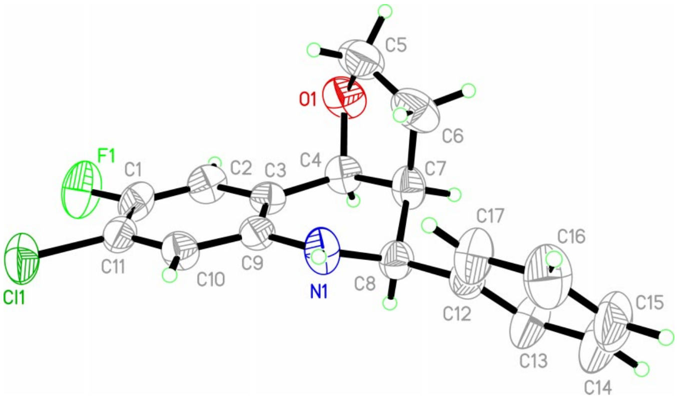


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

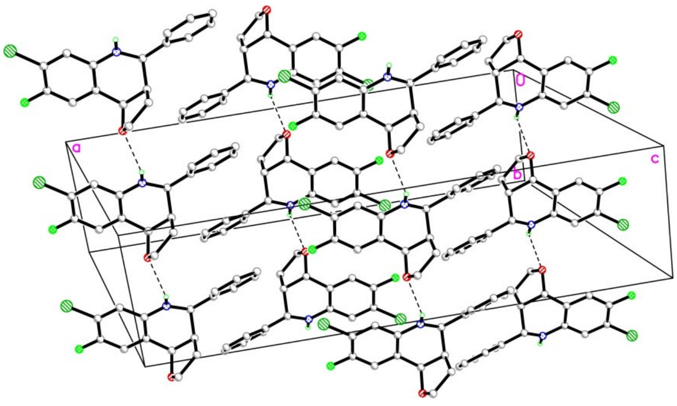


Figure 2
Packing diagram for (I). Dashed lines indicate N—H···O hydrogen bonds. All other H atoms have been omitted for clarity.

quinoline ring system produces a significant twist about the C8···C3 vector. The twist can been seen from the C8—C7—C4—C3 and C8—N1—C9—C3 torsion angles of 32.6 (3) and −24.2 (4)°, respectively.

The phenyl ring is twisted relative to the quinoline ring system, with an interplanar angle of 54.3 (1)°. The torsion angle N1—C8—C12—C17 between these two rings is −27.2 (4)°. The quinoline ring system and the furan rings are nearly perpendicular, as indicated by the dihedral angle of 81.7 (1)° between their least-squares mean planes. Atom O1 is displaced by 1.115 (2) Å from the least-squares plane (N1/C9/C4/C7) of the quinoline ring system. The heterocyclic N1/C8/C7/C4/C3/C9 ring adopts a sofa conformation, with asymmetry parameters (Nardelli, 1983) of ΔC_s(C3) = 0.051 (2) and ΔC_2(C3—C9) = 0.077 (1), and with C8 lying 0.658 (3) Å out of the plane formed by the remaining atoms of the ring. The furan ring adopts the twist conformation with q_2 = 0.35 Å and φ = 14.36° (Cremer & Pople, 1975).

The N atom of the quinoline ring system participates in intermolecular N—H···O hydrogen bonding with the O atom of the furan ring. This hydrogen-bond chain runs along the *b* axis (Fig. 2). Additional weak intra- and intermolecular C—H···O/N hydrogen-bonding interactions are observed in the crystal packing (Table 2).

Experimental

To a solution of the appropriate *N*-benzylideneaniline (5.5 mmol) in dichloromethane (5 ml) at room temperature were added 2,3-dihydrofuran (5.5 mmol) and $ZrCl_4$ (10 mol%). The solution was stirred 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 mixture of methanol and water (3:1).

Crystal data

$C_{17}H_{15}ClFNO$
M_r = 303.75
Monoclinic, $C2/c$
a = 25.826 (2) Å
b = 6.8065 (6) Å
c = 17.2924 (16) Å
β = 102.490 (2)°
V = 2967.8 (4) Å^3
Z = 8
D_x = 1.36 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5098 reflections
θ = 2.4–21.2°
μ = 0.27 mm^{-1}
T = 273 (2) K
Block, colorless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
7178 measured reflections
2607 independent reflections
2074 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25°
$h = -28 \rightarrow 30$
$k = -8 \rightarrow 7$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.155
S = 1.16
2607 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 1.7065P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cl1—C11	1.737 (3)	O1—C4	1.431 (4)
F1—C1	1.356 (3)	N1—C8	1.449 (4)
O1—C5	1.425 (4)	N1—C9	1.381 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.30	2.979 (3)	136
C10—H10···O1^{i}	0.93	2.58	3.336 (4)	138
C6—H6*A*···N1^{ii}	0.97	2.61	2.971 (4)	102

Symmetry codes: (i) $x, y+1, z$; (ii) x, y, z.

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After location of the H atoms in difference density maps, all H atoms were positioned geometrically and treated as riding atoms, with C—H distances in the range 0.93–0.98 Å and the N—H distance fixed at 0.86 Å, and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C,N)$ for the other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997; molecular graphics: *ORTEP3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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