Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(4-Fluorophenyl)-5,6-dihydrobenzimidazo[1,2-c]quinazoline

John Nicolson Low,[a]*† Braulio Insuasty,[b] Harlen Torres[b] and Justo Cobo[c]

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, [b]Grupo de Investigación de Compuestos Heterocíclicos, Departamento de Química, Universidad de Valle, AA 25360, Cali, Colombia, and [c]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain

† Postal address:, Department of Electrical Engineering and Physics, University of Dundee, Dundee DD1 4HN, Scotland

Correspondence e-mail: j.n.low@abdn.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.057
wR factor = 0.162
Data-to-parameter ratio = 17.3

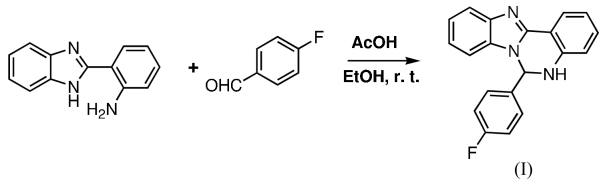
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 October 2003
Accepted 17 October 2003
Online 23 October 2003

The title compound, $C_{20}H_{14}FN_3$, contains one conventional N—H···N hydrogen bond which links the molecules into spiral chains running parallel to the *c* axis; the conformation of this chain appears to be stabilized by an antiparallel C—H···F contact. The structure also contains non-localized solvent in isolated cavities which lie along the crystallographic threefold axis.

Comment

Due to the increasing number of immunocompromized individuals, fungal infections have increased steadily in the last two decades, affecting millions of people worldwide. Since compounds containing the quinazoline skeleton have shown interesting antifungal properties (Chan *et al.*, 1995; Castaldo *et al.*, 1979; Bartroli *et al.*, 1998) this current project is aimed at developing synthetic strategies for preparing heterocyclic compounds containing the quinazoline ring system. The title compound was prepared from 2-(1*H*-benzimidazol-2-yl)-aniline and 4-fluorobenzaldehyde.



(I)

The title compound, (I), contains no unusual bond lengths or angles (Fig. 1). The supramolecular structure is determined by two direction-specific interactions which link the molecules together into spirals running parallel to the *c* axis. These interactions are a conventional N—H···N hydrogen bond and a short C—H···F contact (Table 1). Atom N1 in the molecule at (*x*, *y*, *z*) forms, *via* H1, a hydrogen bond to N7 in the molecule at $(\frac{2}{3} - x + y, \frac{1}{3} - x, \frac{1}{3} + z)$, so forming a *C*(6) (Bernstein *et al.*, 1995) spiral chain which runs parallel to the *c* axis. Atom C8 in this latter molecule forms a contact *via* H8 to atom F24 in the molecule at $(\frac{1}{3} - y, x - y - \frac{1}{3}, z - \frac{1}{3})$, forming a *C*(11) chain. Atom N1 in this molecule, *via* H1, links to N7 in the molecule at (*x*, *y*, *z*), forming an $R_3^3(19)$ ring. Fig. 2 shows a stereoview of the ring structure running along the *c* axis.

Experimental

A solution of 2-(1*H*-benzimidazol-2-yl)aniline and 4-fluorobenzaldehyde (16 mmol) in ethanol (10 ml) was treated with acetic acid (1 ml). The mixture was stirred at room temperature for 10 min and then allowed to crystallize in a refrigerator overnight. The resulting solid was collected and recrystallized from ethanol, affording yellow crystals suitable for X-ray analysis (m.p. 477 K, yield 85%). Analysis

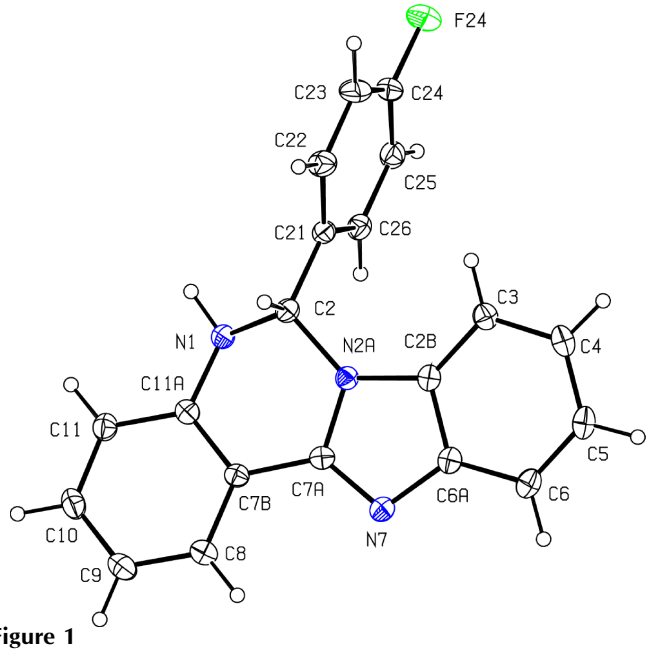


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

calculated for $C_{20}H_{14}FN_3$: C 76.18, H 4.47, N 13.33%; found: C 76.31, H 4.41, N 13.21%.

Crystal data

$C_{20}H_{14}FN_3$
M_r = 315.34
Trigonal (hexagonal axes), $R\overline{3}$
a = 22.3211 (8) Å
c = 17.2220 (9) Å
V = 7431.0 (5) Å^3
Z = 18
D_x = 1.268 Mg m^{-3}
Mo Kα radiation
Cell parameters from 3780 reflections
θ = 3.0–27.5°
μ = 0.09 mm^{-1}
T = 120.0 (2) K
Plate, colourless
0.26 × 0.20 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.978, T_{max} = 0.995
28120 measured reflections
3780 independent reflections
2552 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
θ_{max} = 27.5°
$h = -28 \rightarrow 28$
$k = -28 \rightarrow 28$
$l = -19 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.162
S = 1.04
3780 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0894P)^2 + 1.7404P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N7^{i}	0.93	2.09	3.016 (3)	173
C8—H8···F24^{ii}	0.95	2.49	3.274 (2)	140

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

The structure was refined using hexagonal axes. H atoms were treated as riding atoms, with C—H = 0.95 Å (aromatic), 0.98 Å (methyl) and 0.99 Å (CH_2). The H atom attached to N1 was allowed to ride at a position based on its location in a difference map. The difference map also revealed several peaks lying in voids on and around the threefold axis. These could not be resolved in terms of sensible molecules and the SQUEEZE option in *PLATON* (Spek, 2003) was used to remove the contribution of electron density from these solvent-accessible voids.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work. BI and HT thank COLCIENCIAS and the Universidad de Valle for financial support of this work.

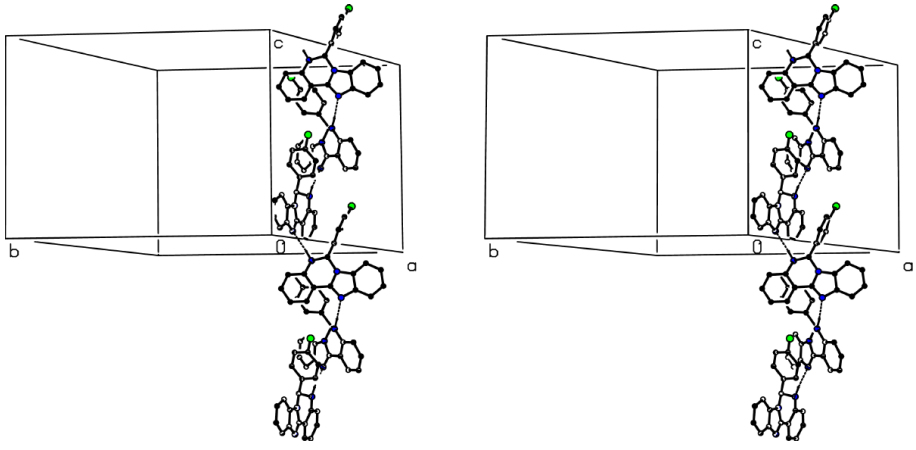


Figure 2
A stereoview of the $R_3^3(19)$ ring formed by the combination of the $C(6)$ and $C(11)$ chains.

References

Bartroli, J., Turmo, E., Algueró, M., Boncompte, E., Vericat, M., Conte, L., Ramis, J., Merlos, M., García-Rafanell, J. & Forn, J. (1998). *J. Med. Chem.* **41**, 1869–1882.

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

Castaldo, R. A., Gump, D. W. & McCormack, J. J. (1979). *Antimicrob. Agents Chemother.* **15**, 81–86.

Chan, J. H., Hong, J. S., Kuyper, L. F., Baccanari, D. P., Joyner, S. S., Tansik, R. L., Boytos, C. M. & Rudolph, S. K. (1995). *J. Med. Chem.* **38**, 3608–3616.

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.