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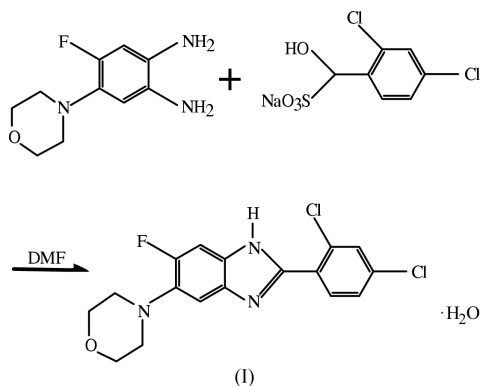
Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(C-C)$ = 0.003 Å
R factor = 0.038
wR factor = 0.106
Data-to-parameter ratio = 14.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-(2,4-Dichlorophenyl)-5-fluoro-6-morpholin-4-yl-1H-benzimidazole monohydrate

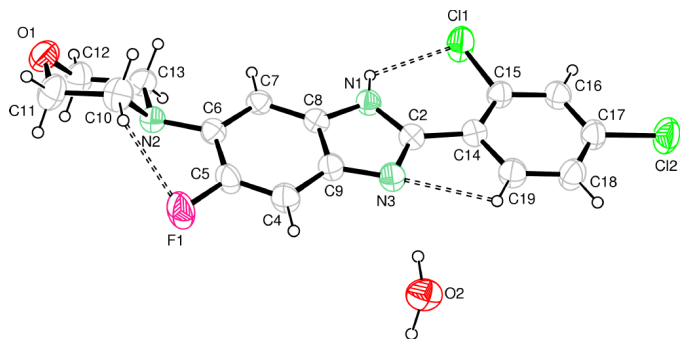
The crystal structure of the title compound, $C_{17}H_{14}Cl_2FN_3O \cdot H_2O$, is stabilized by hydrogen bonds. The morpholine ring exhibits a typical chair conformation, while the benzimidazole system is planar.Received 14 August 2002
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Comment

The benzimidazole nucleus, which is a useful structure for further molecular exploration and for the development of new pharmaceutical compounds, has received much attention in recent years (Mann *et al.*, 2001; Nakano *et al.*, 2000). Owing to the versatility of this heterocycle, we are focusing our research efforts on new and potent benzimidazole derivatives. In our previous studies we described the synthesis and antimicrobial activities of some benzimidazole derivatives (Göker *et al.*, 2001; Kuş *et al.*, 2001). 2-(Substituted phenyl)-benzimidazoles have various pharmaceutical activities such as antibacterial, antiviral, antitumour and anti-inflammatory (Coburn *et al.*, 1987; Roth *et al.*, 1997; Chen *et al.*, 1993; Denny *et al.*, 1993; Evans *et al.*, 1996).

To evaluate their antimicrobial activities, we synthesized 2-(2,4-dichlorophenyl)-5-fluoro-6-morpholin-4-yl-1H-benzimidazole and its analogues. The synthesis and physical and spectroscopic data for this compound, which has a substituted phenyl ring at the 2 position of the benzimidazole nucleus, has been reported by us (Kuş, 2002). In this research, the X-ray structure of the monohydrate, (I), was determined in order to establish its conformation.

The intramolecular bond distances and angles (Table 1) for the benzimidazole moiety of (I) are in good agreement with those observed in other benzimidazole derivatives (Göker *et al.*, 1995; Özbey *et al.*, 1998; Kendi *et al.*, 1999; Vasudevan *et al.*, 1994; Balasubramanian *et al.*, 1996). The bond lengths C15—

**Figure 1**

ORTEP (Johnson, 1965) drawing of the asymmetric unit of the title compound, with the atomic numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

Cl1 and C17—Cl2 (Table 1) are within the expected range (Allen *et al.*, 1987).

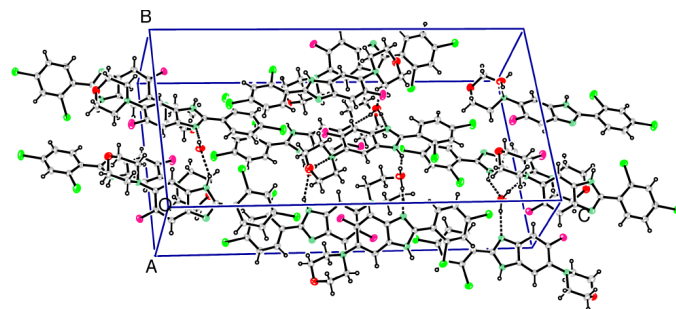
As expected, the benzimidazole ring system is essentially planar [maximum deviation 0.017 (3) Å for C9], with a dihedral angle of 1.54 (9) Å between the imidazole and the benzene ring planes. The F atom lies -0.077 (2) Å from the least-squares plane defined by all the atoms of the two fused rings. In the molecule, the benzimidazole system is coplanar with the phenyl substituent, owing to the hydrogen bonding between the imidazole N atoms and the chlorine and one of the C atoms of the phenyl moiety (Table 2). The dihedral angle between the mean planes of these rings is 0.15 (7)°, so the structure is almost planar except for the morpholine ring, as shown in Fig. 1. The dichlorophenyl ring is planar and the atom Cl1 is situated 0.0240 (9) Å above this plane.

The morpholine ring deviates from planarity, with atoms N2 and O1 displaced from the C10/C11/C12/C13 mean plane by 0.689 (2) and -0.645 (2) Å, respectively. The dihedral angle between the mean planes of the morpholine ring and the benzimidazole ring is 40.8 (1)°. The puckering parameters (Cremer & Pople, 1975) of this ring are $Q = 0.581$ (2) Å, $\theta = 177.1$ (3)° and $\varphi = -43$ (5)°; thus the morpholine ring adopts a chair conformation.

The crystal structure of the title compound is stabilized by intra- and intermolecular hydrogen bonds (Table 2, Fig. 2).

Experimental

To obtain 2-(2,4-dichlorophenyl)-5-fluoro-6-morpholin-4-yl-1*H*-benzimidazole (Kuş, 2002), to a suspension of 4-fluoro-5-morpholin-4-yl-benzene-1,2-diamine (Sanna *et al.*, 1998) (0.211 g, 1 mmol) in DMF (5 ml), the sodium metabisulfite adduct of 2,4-dichlorobenzaldehyde (0.191 g, 1 mmol) was added and the mixture was heated under a nitrogen atmosphere for 4 h at 333 K. Water was added to the reaction medium and the solid product obtained was collected by filtration and washed with water. The residue was chromatographed [EtOAc: *n*-hexane (1:2)] and recrystallized from ethanol, affording 0.311 mg (85%) of (I); m.p. 485–487 K. The assigned structure was substantiated by IR, ¹H NMR and MS data.

**Figure 2**

The intermolecular hydrogen bonds in (I).

Crystal data

C₁₇H₁₄Cl₂FN₃O·H₂O
M_r = 384.23
 Orthorhombic, *Pbca*
a = 7.4455 (7) Å
b = 15.310 (3) Å
c = 29.487 (3) Å
V = 3361.2 (8) Å³
Z = 8
D_x = 1.519 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 25 reflections
 $\theta = 10.2$ – 18.0°
 $\mu = 0.41$ mm⁻¹
T = 295 (2) K
 Prism, light yellow
 0.48 × 0.36 × 0.24 mm

Data collection

Enraf–Nonius Turbo-CAD-4 diffractometer
 Non-profiled ω scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.826$, $T_{\max} = 0.907$
 3893 measured reflections
 3403 independent reflections
 2174 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.079$
 $\theta_{\text{max}} = 26.3^\circ$
 $h = 0 \rightarrow 9$
 $k = 0 \rightarrow 19$
 $l = 0 \rightarrow 36$
 3 standard reflections
 frequency: 120 min
 intensity decay: 4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.106$
 $S = 1.02$
 3403 reflections
 238 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 1.0249P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C2—N3	1.321 (3)	C6—N2	1.425 (3)
C2—N1	1.363 (3)	C15—Cl1	1.741 (3)
C2—C14	1.476 (3)	C17—Cl2	1.732 (2)
C5—F1	1.367 (3)		
N3—C2—N1	112.3 (2)	O1—C12—C13	112.6 (2)
N3—C2—C14	120.9 (2)	N2—C13—C12	109.7 (2)
N1—C2—C14	126.8 (2)	C19—C14—C2	116.4 (2)
C4—C5—F1	117.5 (2)	C14—C15—C11	121.95 (19)
C5—C6—N2	118.5 (2)	C16—C17—C12	119.4 (2)
N1—C8—C9	105.1 (2)	C2—N1—C8	107.31 (19)
N3—C9—C8	110.5 (2)	C13—N2—C10	108.3 (2)
N2—C10—C11	108.8 (2)	C2—N3—C9	104.77 (19)
O1—C11—C10	111.7 (2)	C11—O1—C12	109.3 (2)

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...C11	0.93 (2)	2.45 (3)	3.029 (2)	120.5 (19)
C19—H19...N3	0.93	2.41	2.776 (3)	104
C10—H10B...F1	0.97	2.34	2.944 (3)	119
N1—H1...O2 ⁱ	0.93 (2)	2.18 (2)	2.986 (3)	145 (2)
O2—H21...N3	0.93 (2)	2.07 (2)	3.002 (3)	172 (3)
O2—H22...N2 ⁱⁱ	1.00 (2)	2.07 (3)	3.057 (3)	168 (4)
C12—H12A...F1 ⁱ	0.97	2.38	3.257 (3)	150
C12—H12A...Cl2 ⁱⁱⁱ	0.97	2.78	3.408 (3)	123

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

The H atoms of N1 and the water molecule were located from a difference Fourier map and refined freely. The other H atoms were positioned geometrically, at distances 0.93 and 0.97 Å for methylene and aromatic H atoms, respectively, and refined riding on their parent atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *PLATON* (Spek, 2000).

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