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5-Chloro-6-(2-fluorobenzoyl)-1,3-benzoxazol-2(3*H*)-one

Abdullah Aydın,[a] Tijen Önkol,[b] Mehmet Akkurt,[c]* Orhan Büyükgüngör[d] and Serdar Ünlü[b]

[a]Department of Physics Education, Kastamonu Education Faculty, Gazi University, 37200 Kastamonu, Turkey, [b]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Gazi University, Etiler, 06330 Ankara, Turkey, [c]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, and [d]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit–Samsun, Turkey

Correspondence e-mail: aaydin@gazi.edu.tr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.073
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.

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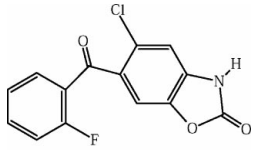
The title compound, $C_{17}H_{11}ClFNO_5$, has a non-planar configuration. The crystal structure is stabilized by the formation of bifurcated N—H···O/O′ hydrogen bonds involving the oxazole N atom and the two carbonyl O atoms of symmetry-related molecules.

Comment

Benzothiazolinone/2-benzoxazolinone derivatives exhibit a variety of pharmacological effects, including analgesic and anti-inflammatory activity (Fereira *et al.*, 1995; Ünlü *et al.*, 2003).



(I)

The molecular structure of the title compound, (I), a new benzoxazolinone derivative, is shown in Fig. 1, and selected geometric parameters are presented in Table 1. The hydrogen-bond contacts are shown in Fig. 2 and details are given in Table 2. The double-bond length for C1═O2 is 1.204 (2) Å, and the C4—Cl1 and C14—F1 bond lengths are 1.7410 (16) and 1.356 (2) Å, respectively. The bond lengths observed in (I) have normal values, and both the bond lengths and angles are comparable to those observed in related structures (Allen *et al.*, 1987; Aydın *et al.*, 2002).

The bicylic benzoxazole system of (I) is planar to within 0.015 Å, with the maximum deviations from the mean plane through the benzoxazole (O1/C1/N1/C2–C7) being −0.010 (1), 0.015 (1) and −0.012 (1) Å for atoms N1, C3 and C5, respectively. The dihedral angle between the benzoxazole and fluorophenyl ring systems is 74.7 (1)°, showing some deviation from planarity, as observed for similar compounds in the literature (Guilardi *et al.*, 2002; Chinnakali *et al.*, 1990).

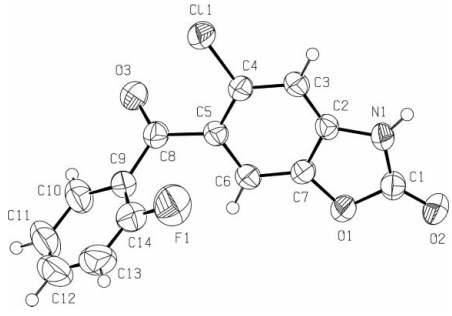


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The crystal structure of (I) is stabilized by N—H···O-type hydrogen-bond contacts, as shown in Fig. 2 (Table 2).

Experimental

A mixture of 200 g polyphosphoric acid (PPA), 0.1 mol of 5-chloro-2-oxo-3*H*-benzoxazole and 0.12 mol of 2-fluorobenzoic acid was heated at 413 K with stirring for 7 h. The mixture was then poured into a 800 ml of ice water and stirred for 8 h. The precipitate was washed with water to neutral pH, dried and crystallized from toluene (Pilli *et al.*, 1993).

Crystal data

$C_{14}H_7ClFNO_3$
M_r = 291.66
Orthorhombic, *Pbca*
a = 7.723 (7) Å
b = 14.255 (1) Å
c = 22.530 (2) Å
V = 2480.4 (4) Å^3
Z = 8
D_x = 1.562 Mg m^{-3}
Mo Kα radiation
Cell parameters from 5958 reflections
θ = 3.0–56.4°
μ = 0.33 mm^{-1}
T = 293 K
Plate, colorless
0.62 × 0.33 × 0.19 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (Stoe & Cie, 2002)
T_{min} = 0.823, T_{max} = 0.941
10 796 measured reflections
3080 independent reflections
1705 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 28.3°
$h = -8 \rightarrow 10$
$k = -18 \rightarrow 18$
$l = -26 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.073
S = 0.86
3080 reflections
189 parameters
H-atom parameters constraned
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0012 (2)

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.7410 (16)	O2—C1	1.204 (2)
F1—C14	1.356 (2)	O3—C8	1.2099 (19)
O1—C1	1.3876 (19)	N1—C1	1.343 (2)
O1—C7	1.3837 (18)	N1—C2	1.3820 (18)
C1—O1—C7	107.10 (11)	Cl1—C4—C5	120.41 (11)
C1—N1—C2	110.35 (13)	O1—C7—C2	108.97 (12)
O2—C1—N1	130.40 (15)	O1—C7—C6	128.15 (14)
O1—C1—O2	122.10 (14)	O3—C8—C9	119.61 (14)
O1—C1—N1	107.51 (13)	O3—C8—C5	119.98 (14)
N1—C2—C7	106.07 (13)	F1—C14—C9	118.98 (16)
N1—C2—C3	132.54 (13)	F1—C14—C13	117.61 (18)
Cl1—C4—C3	116.58 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.17	2.9243 (17)	147
N1—H1···O3^{ii}	0.86	2.60	2.9448 (17)	105

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

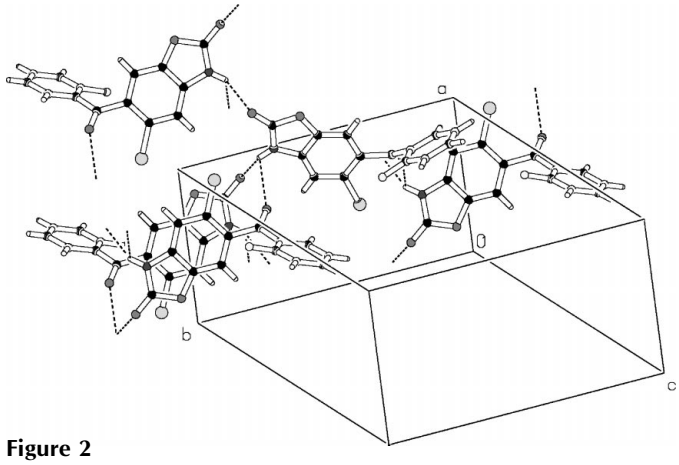


Figure 2
A view of the crystal packing in (I), showing the intermolecular hydrogen-bond contacts (dashed lines) between neighbouring molecules.

All the H atoms were placed in geometrically idealized positions, with C—H = 0.93 Å and N—H = 0.86 Å. The U_{iso}(H) values were constrained to be 1.2 times U_{eq} of the carrier atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995) and *WinGX* publication routines (Farrugia, 1999).

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