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3-(2,4-Difluorophenyl)-1-(pyridin-4-yl)benzo[4,5]-imidazo[1,2-*d*][1,2,4]triazin-4(3*H*)-one

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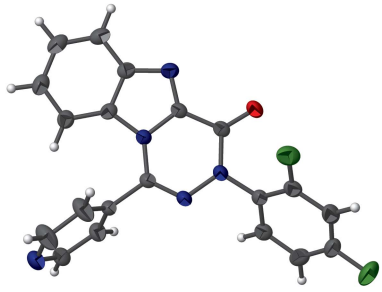
Keywords: crystal structure; difluorophenyl; pyridine; benzoimidazole; 1,2,4-triazinone.

CCDC reference: 1501316

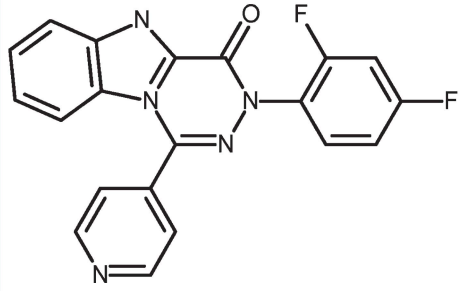
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{20}H_{11}F_2N_5O$, the central 13-membered ring system (r.m.s. deviation = 0.028 Å) makes a dihedral angle of 53.13 (7)° with the difluorophenyl ring and 79.98 (7)° with the pyridine ring. The crystal packing features aromatic π–π interactions between the 13-membered rings [shortest distance between ring centroids = 3.5682 (8) Å].

3D view



Chemical scheme



Structure description

Several benzimidazole-based compounds show anti-cancer activity (Thomas *et al.*, 2007) and some of them exhibit cytotoxic effects against a panel of human cancer cell lines (Refaat, 2010). For example, benzimidazole-4,7-diones exhibit cytotoxicity against colon, breast and lung cell lines (Gellis *et al.*, 2008). The good efficacy of imidazole-based compounds as anti-cancer agents prompted this study and the synthesis of a masked benzimidazole in a triazine ring as the new scaffold of a potential anti-cancer candidate. The tile compound is shown in Fig. 1. The central 13-membered ring is almost planar (r.m.s. deviation = 0.028 Å) and makes a dihedral angle of 53.13 (7)° with the difluorophenyl ring and 79.98 (7)° to the pyridine ring. In the crystal, two molecules related by a centre of inversion show a π–π interaction: the shortest distance between the ring centroids is 3.5682 (8)°.

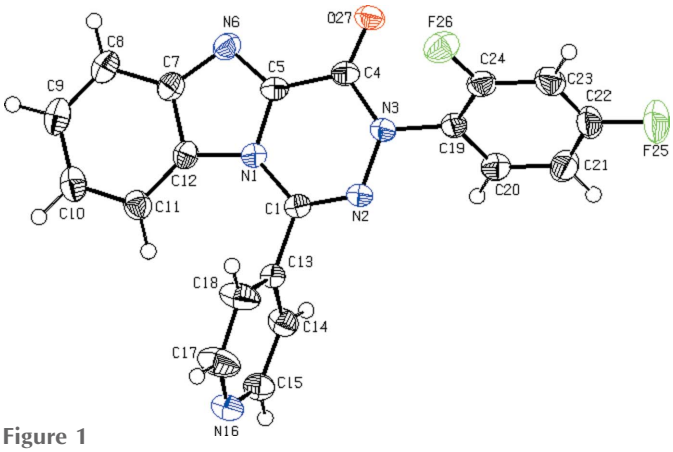


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

2.1 mmol of NaH was added slowly to a solution of 2.1 mmol of ethyl-2- benzimidazolcarboxylate in 20 ml dry THF and continued stirring at RT for about 20 minutes. To this flask, 2.0 mmol of *N*-(2,4-diflurophenyl)-4-pyridinecarbohydrazonoyl chloride was added slowly portionwise and in parallel 0.5 ml of Et_3N was added dropwise. The reaction was left stirring overnight, and it was monitored by TLC until it had finished. The solution was filtered and concentrated under vacuum. The solid residue was purified by column chromatography (hexane:ethyl acetate; 2:1 then 1:1). Yield: 30%. Colourless plates were obtained from the slow evaporation of a hexane/ ethyl acetate solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

BAT thanks the Palestinian research council (Ramallah) for funding.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{11}F_2N_5O$
M_r	375.34
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	193
a, *b*, *c* (Å)	27.1174 (13), 10.1914 (3), 11.9292 (6)
β (°)	94.075 (4)
V (Å^3)	3288.5 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.25 × 0.20 × 0.06
Data collection	
Diffractometer	Stoe IPDS 2T
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8967, 4043, 2985
R_{int}	0.023
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.666
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.098, 1.06
No. of reflections	4043
No. of parameters	253
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.22

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2006), *SIR2004* (Altomare *et al.*, 1995) and *SHELXL2014* (Sheldrick, 2015).

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full crystallographic data

IUCrData (2016). **1**, x161380 [doi:10.1107/S2414314616013808]

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Crystal data

$C_{20}H_{11}F_2N_5O$
M_r = 375.34
Monoclinic, $C2/c$
a = 27.1174 (13) Å
b = 10.1914 (3) Å
c = 11.9292 (6) Å
β = 94.075 (4)°
V = 3288.5 (2) Å^3
Z = 8
$F(000)$ = 1536
D_x = 1.516 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8710 reflections
θ = 2.1–28.3°
μ = 0.11 mm^{-1}
T = 193 K
Plate, colourless
0.25 × 0.20 × 0.06 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
8967 measured reflections
4043 independent reflections
2985 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 28.3°, θ_{min} = 2.7°
h = −36→36
k = −13→12
l = −13→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.098
S = 1.06
4043 reflections
253 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 1.3818P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.66970 (4)	0.29698 (11)	0.47943 (9)	0.0261 (2)
C1	0.63343 (4)	0.25857 (13)	0.39840 (11)	0.0264 (3)
N2	0.60540 (4)	0.15937 (11)	0.41154 (9)	0.0285 (2)
N3	0.61157 (4)	0.08983 (11)	0.51134 (9)	0.0284 (2)
C4	0.64512 (5)	0.11782 (13)	0.60141 (11)	0.0284 (3)
C5	0.67531 (4)	0.23382 (13)	0.58282 (11)	0.0277 (3)
N6	0.70939 (4)	0.28491 (12)	0.65223 (10)	0.0321 (3)
C7	0.72797 (5)	0.38863 (14)	0.59241 (12)	0.0307 (3)
C8	0.76485 (5)	0.47860 (15)	0.62792 (13)	0.0372 (3)
H8	0.7813	0.4733	0.7008	0.045*
C9	0.77631 (5)	0.57509 (15)	0.55318 (15)	0.0410 (4)
H9	0.8010	0.6377	0.5754	0.049*
C10	0.75252 (5)	0.58334 (15)	0.44541 (14)	0.0391 (3)
H10	0.7616	0.6514	0.3965	0.047*
C11	0.71615 (5)	0.49545 (14)	0.40798 (12)	0.0336 (3)
H11	0.7002	0.5006	0.3345	0.040*
C12	0.70418 (5)	0.39889 (13)	0.48411 (11)	0.0282 (3)
C13	0.62513 (5)	0.33508 (13)	0.29263 (11)	0.0273 (3)
C14	0.64386 (5)	0.29223 (15)	0.19487 (12)	0.0350 (3)
H14	0.6623	0.2131	0.1934	0.042*
C15	0.63516 (6)	0.36720 (16)	0.09878 (12)	0.0383 (3)
H15	0.6489	0.3379	0.0321	0.046*
N16	0.60912 (5)	0.47670 (14)	0.09364 (11)	0.0403 (3)
C17	0.59074 (7)	0.51542 (19)	0.18872 (15)	0.0525 (4)
H17	0.5715	0.5934	0.1869	0.063*
C18	0.59783 (7)	0.44954 (17)	0.28996 (13)	0.0474 (4)
H18	0.5843	0.4821	0.3558	0.057*
C19	0.58143 (5)	−0.02544 (13)	0.51111 (11)	0.0291 (3)
C20	0.58144 (5)	−0.11194 (15)	0.42175 (12)	0.0354 (3)
H20	0.6029	−0.0973	0.3634	0.042*
C21	0.55029 (6)	−0.21993 (16)	0.41718 (14)	0.0439 (4)
H21	0.5498	−0.2795	0.3558	0.053*
C22	0.52009 (6)	−0.23884 (16)	0.50342 (15)	0.0446 (4)
C23	0.51848 (5)	−0.15508 (17)	0.59252 (14)	0.0424 (4)
H23	0.4970	−0.1704	0.6507	0.051*
C24	0.54937 (5)	−0.04707 (15)	0.59455 (12)	0.0335 (3)
F25	0.48935 (4)	−0.34381 (11)	0.49899 (11)	0.0706 (4)
F26	0.54621 (3)	0.04138 (10)	0.67697 (7)	0.0446 (2)
O27	0.64870 (3)	0.05067 (10)	0.68590 (8)	0.0350 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0272 (5)	0.0265 (5)	0.0244 (5)	−0.0003 (4)	0.0002 (4)	0.0006 (4)
C1	0.0274 (6)	0.0285 (6)	0.0231 (6)	0.0014 (5)	0.0006 (4)	−0.0004 (5)

N2	0.0322 (5)	0.0295 (6)	0.0232 (5)	−0.0017 (4)	−0.0024 (4)	0.0043 (4)
N3	0.0305 (5)	0.0294 (6)	0.0246 (5)	−0.0027 (4)	−0.0020 (4)	0.0049 (4)
C4	0.0273 (6)	0.0316 (7)	0.0260 (6)	0.0030 (5)	−0.0002 (5)	0.0022 (5)
C5	0.0281 (6)	0.0309 (7)	0.0239 (6)	0.0023 (5)	−0.0007 (5)	0.0018 (5)
N6	0.0304 (5)	0.0342 (6)	0.0309 (6)	−0.0010 (5)	−0.0033 (4)	−0.0011 (5)
C7	0.0276 (6)	0.0312 (7)	0.0331 (7)	0.0012 (5)	0.0005 (5)	−0.0035 (6)
C8	0.0305 (7)	0.0382 (8)	0.0423 (8)	−0.0027 (6)	−0.0022 (6)	−0.0080 (6)
C9	0.0317 (7)	0.0364 (8)	0.0551 (10)	−0.0067 (6)	0.0042 (6)	−0.0104 (7)
C10	0.0384 (7)	0.0322 (7)	0.0479 (9)	−0.0058 (6)	0.0117 (6)	−0.0025 (6)
C11	0.0353 (7)	0.0318 (7)	0.0340 (7)	−0.0011 (5)	0.0064 (5)	−0.0005 (6)
C12	0.0262 (6)	0.0274 (6)	0.0312 (7)	−0.0002 (5)	0.0032 (5)	−0.0045 (5)
C13	0.0299 (6)	0.0276 (6)	0.0241 (6)	−0.0033 (5)	−0.0003 (5)	0.0018 (5)
C14	0.0399 (7)	0.0339 (7)	0.0319 (7)	0.0021 (6)	0.0064 (6)	0.0027 (6)
C15	0.0462 (8)	0.0424 (8)	0.0269 (7)	−0.0042 (6)	0.0073 (6)	0.0008 (6)
N16	0.0474 (7)	0.0429 (7)	0.0304 (6)	−0.0012 (6)	0.0008 (5)	0.0090 (6)
C17	0.0697 (11)	0.0496 (10)	0.0390 (9)	0.0248 (9)	0.0088 (8)	0.0120 (8)
C18	0.0680 (11)	0.0455 (9)	0.0298 (8)	0.0211 (8)	0.0111 (7)	0.0063 (7)
C19	0.0280 (6)	0.0296 (6)	0.0288 (7)	−0.0022 (5)	−0.0041 (5)	0.0046 (5)
C20	0.0370 (7)	0.0356 (7)	0.0329 (7)	−0.0001 (6)	−0.0020 (5)	0.0016 (6)
C21	0.0504 (9)	0.0349 (8)	0.0447 (9)	−0.0032 (7)	−0.0087 (7)	−0.0038 (7)
C22	0.0416 (8)	0.0368 (8)	0.0535 (10)	−0.0137 (6)	−0.0116 (7)	0.0114 (7)
C23	0.0339 (7)	0.0515 (9)	0.0410 (8)	−0.0109 (7)	−0.0033 (6)	0.0141 (7)
C24	0.0308 (6)	0.0395 (8)	0.0294 (7)	−0.0007 (6)	−0.0030 (5)	0.0034 (6)
F25	0.0691 (7)	0.0532 (7)	0.0871 (9)	−0.0348 (6)	−0.0104 (6)	0.0085 (6)
F26	0.0385 (4)	0.0609 (6)	0.0350 (5)	−0.0032 (4)	0.0062 (3)	−0.0062 (4)
O27	0.0350 (5)	0.0400 (6)	0.0292 (5)	−0.0021 (4)	−0.0036 (4)	0.0097 (4)

Geometric parameters (Å, º)

N1—C1	1.3857 (16)	C13—C14	1.3759 (19)
N1—C5	1.3900 (17)	C13—C18	1.381 (2)
N1—C12	1.3958 (16)	C14—C15	1.384 (2)
C1—N2	1.2810 (17)	C14—H14	0.9500
C1—C13	1.4867 (18)	C15—N16	1.320 (2)
N2—N3	1.3854 (15)	C15—H15	0.9500
N3—C4	1.3874 (16)	N16—C17	1.331 (2)
N3—C19	1.4310 (17)	C17—C18	1.383 (2)
C4—O27	1.2165 (16)	C17—H17	0.9500
C4—C5	1.4637 (19)	C18—H18	0.9500
C5—N6	1.3047 (16)	C19—C20	1.383 (2)
N6—C7	1.3898 (19)	C19—C24	1.385 (2)
C7—C8	1.4000 (19)	C20—C21	1.386 (2)
C7—C12	1.4064 (19)	C20—H20	0.9500
C8—C9	1.378 (2)	C21—C22	1.373 (3)
C8—H8	0.9500	C21—H21	0.9500
C9—C10	1.399 (2)	C22—F25	1.3550 (17)
C9—H9	0.9500	C22—C23	1.366 (3)
C10—C11	1.383 (2)	C23—C24	1.383 (2)

C10—H10	0.9500	C23—H23	0.9500
C11—C12	1.393 (2)	C24—F26	1.3412 (17)
C11—H11	0.9500		

C1—N1—C5	121.03 (11)	C14—C13—C18	118.57 (13)
C1—N1—C12	132.71 (11)	C14—C13—C1	120.68 (12)
C5—N1—C12	106.17 (10)	C18—C13—C1	120.74 (12)
N2—C1—N1	122.38 (12)	C13—C14—C15	118.39 (14)
N2—C1—C13	117.54 (11)	C13—C14—H14	120.8
N1—C1—C13	120.06 (11)	C15—C14—H14	120.8
C1—N2—N3	118.19 (10)	N16—C15—C14	124.37 (14)
N2—N3—C4	126.36 (11)	N16—C15—H15	117.8
N2—N3—C19	112.60 (10)	C14—C15—H15	117.8
C4—N3—C19	120.89 (11)	C15—N16—C17	116.28 (13)
O27—C4—N3	122.36 (12)	N16—C17—C18	124.27 (16)
O27—C4—C5	124.57 (12)	N16—C17—H17	117.9
N3—C4—C5	113.06 (11)	C18—C17—H17	117.9
N6—C5—N1	113.96 (12)	C13—C18—C17	118.11 (15)
N6—C5—C4	127.21 (12)	C13—C18—H18	120.9
N1—C5—C4	118.78 (11)	C17—C18—H18	120.9
C5—N6—C7	104.12 (11)	C20—C19—C24	119.14 (13)
N6—C7—C8	128.32 (13)	C20—C19—N3	119.56 (12)
N6—C7—C12	111.64 (11)	C24—C19—N3	121.12 (13)
C8—C7—C12	120.03 (13)	C19—C20—C21	120.19 (14)
C9—C8—C7	117.44 (14)	C19—C20—H20	119.9
C9—C8—H8	121.3	C21—C20—H20	119.9
C7—C8—H8	121.3	C22—C21—C20	118.43 (15)
C8—C9—C10	121.79 (14)	C22—C21—H21	120.8
C8—C9—H9	119.1	C20—C21—H21	120.8
C10—C9—H9	119.1	F25—C22—C23	118.03 (16)
C11—C10—C9	121.98 (14)	F25—C22—C21	118.65 (16)
C11—C10—H10	119.0	C23—C22—C21	123.29 (14)
C9—C10—H10	119.0	C22—C23—C24	117.26 (15)
C10—C11—C12	116.17 (14)	C22—C23—H23	121.4
C10—C11—H11	121.9	C24—C23—H23	121.4
C12—C11—H11	121.9	F26—C24—C23	118.53 (13)
C11—C12—N1	133.32 (12)	F26—C24—C19	119.74 (13)
C11—C12—C7	122.58 (12)	C23—C24—C19	121.65 (14)
N1—C12—C7	104.10 (11)		

C5—N1—C1—N2	−4.15 (19)	C5—N1—C12—C7	−0.28 (13)
C12—N1—C1—N2	179.67 (13)	N6—C7—C12—C11	179.80 (12)
C5—N1—C1—C13	174.45 (12)	C8—C7—C12—C11	0.8 (2)
C12—N1—C1—C13	−1.7 (2)	N6—C7—C12—N1	0.27 (15)
N1—C1—N2—N3	0.81 (19)	C8—C7—C12—N1	−178.71 (12)
C13—C1—N2—N3	−177.82 (11)	N2—C1—C13—C14	−80.58 (16)
C1—N2—N3—C4	1.05 (19)	N1—C1—C13—C14	100.75 (15)
C1—N2—N3—C19	−174.38 (12)	N2—C1—C13—C18	98.39 (17)

N2—N3—C4—O27	−179.03 (13)	N1—C1—C13—C18	−80.27 (17)
C19—N3—C4—O27	−3.9 (2)	C18—C13—C14—C15	1.1 (2)
N2—N3—C4—C5	0.36 (18)	C1—C13—C14—C15	−179.89 (13)
C19—N3—C4—C5	175.44 (12)	C13—C14—C15—N16	−1.4 (2)
C1—N1—C5—N6	−176.87 (12)	C14—C15—N16—C17	0.4 (2)
C12—N1—C5—N6	0.21 (15)	C15—N16—C17—C18	0.8 (3)
C1—N1—C5—C4	5.47 (18)	C14—C13—C18—C17	0.0 (2)
C12—N1—C5—C4	−177.45 (11)	C1—C13—C18—C17	−179.04 (16)
O27—C4—C5—N6	−1.5 (2)	N16—C17—C18—C13	−1.0 (3)
N3—C4—C5—N6	179.17 (13)	N2—N3—C19—C20	49.73 (16)
O27—C4—C5—N1	175.86 (13)	C4—N3—C19—C20	−125.98 (14)
N3—C4—C5—N1	−3.51 (17)	N2—N3—C19—C24	−125.31 (13)
N1—C5—N6—C7	−0.04 (15)	C4—N3—C19—C24	58.98 (17)
C4—C5—N6—C7	177.38 (13)	C24—C19—C20—C21	−1.1 (2)
C5—N6—C7—C8	178.73 (14)	N3—C19—C20—C21	−176.22 (13)
C5—N6—C7—C12	−0.15 (15)	C19—C20—C21—C22	−0.5 (2)
N6—C7—C8—C9	−178.81 (14)	C20—C21—C22—F25	179.60 (14)
C12—C7—C8—C9	0.0 (2)	C20—C21—C22—C23	1.4 (2)
C7—C8—C9—C10	−0.4 (2)	F25—C22—C23—C24	−178.73 (13)
C8—C9—C10—C11	0.1 (2)	C21—C22—C23—C24	−0.5 (2)
C9—C10—C11—C12	0.6 (2)	C22—C23—C24—F26	175.62 (13)
C10—C11—C12—N1	178.28 (14)	C22—C23—C24—C19	−1.2 (2)
C10—C11—C12—C7	−1.1 (2)	C20—C19—C24—F26	−174.80 (12)
C1—N1—C12—C11	−3.1 (2)	N3—C19—C24—F26	0.26 (19)
C5—N1—C12—C11	−179.73 (14)	C20—C19—C24—C23	2.0 (2)
C1—N1—C12—C7	176.32 (13)	N3—C19—C24—C23	177.06 (13)