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5-(2-Ethoxy-4-fluorophenyl)-1,2,4-triazolo[1,5-*a*]-pyrimidine

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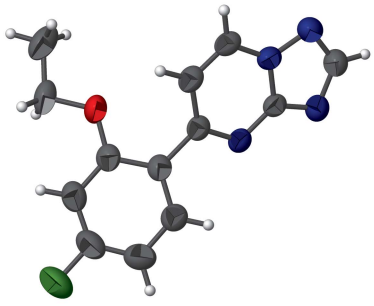
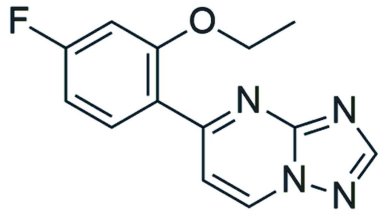
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{11}FN_4O$, the dihedral angle between the triazolopyrimidine ring system and fluorophenyl ring is 39.16 (12)°. In the crystal, C—H···N hydrogen bonds link the molecules resulting in $R_2^2(8)$ ring motifs and $C(8)$ chain motifs.

3D view



Chemical scheme



Structure description

1,2,4-Triazolo[1,5-*a*]pyrimidine derivatives are used in the field of pharmaceutics, agriculture and other areas (Mopper & Zhou, 1990). As part of our work on the synthesis and crystal structure determination of 1,2,4-triazolo[1,5-*a*]pyrimidine derivatives (Gilandoust *et al.*, 2016), the title compound is reported here.

Fig. 1 represents the *ORTEP* drawing of the title compound of which the geometric parameters (bond lengths and bond angles) are in the normal range. The dihedral angle between the triazolopyrimidine and fluorophenyl rings is 39.16 (12)°. An intramolecular C17—H17···O1 contact (Table 1) is observed.

The crystal structure features C—H···N hydrogen bonds (Fig. 2, Table 1). The C5—H5···N4 and C16—H16···N2 hydrogen bonds lead to the formation of infinite chains along the *b* axis [$C(8)$ chain motifs] and inversion dimers [$R_2^2(8)$ ring motifs], respectively.

Synthesis and crystallization

5-Bromo-[1,2,4]-triazolo[1,5-*a*]pyrimidines (1 mmol), 2-ethoxy-4-fluorobenzenboronic acid (1.2 mmol) and K_2CO_3 (3 mmol) were added to a mixture of ethanol, water and 1,4-dioxan in the ratio (1:1:5). The reaction mixture was stirred in a sealed tube for 15 min in

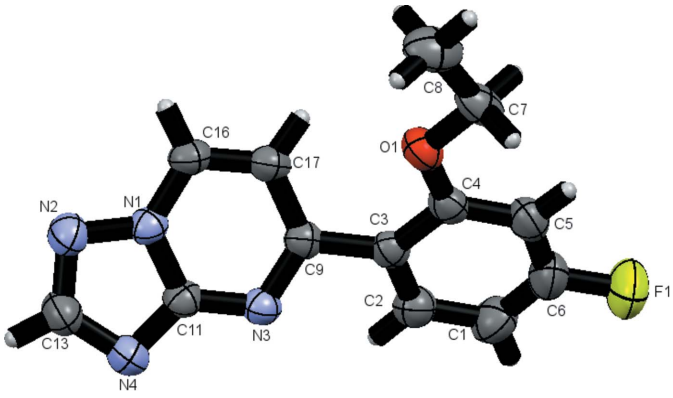


Figure 1
A view of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level and the intramolecular hydrogen bond is shown as a dashed line.

the presence of nitrogen gas to create an inert atmosphere after which the catalyst $[PdCl_2(PPh_3)_2]$ was added (0.1 mmol). The reaction mass was heated to 120–130°C for 35 min in a sealed tube and the progress of the reaction was monitored by TLC. The resultant mixture was filtered through a Celite bed and the filtrate was concentrated under reduced pressure to remove the ethanol by using a rotary evaporator. The reaction mass was extracted with ethyl acetate followed by a brine wash and dried over anhydrous sodium sulfate. The organic layer was evaporated under reduced pressure to get the crude product, which was purified by column chromatography using 60:120 mesh silica gel and EtOAc:hexane as eluent (40:60 ml) to get the desired triazolopyrimidine as a white solid. Good quality single crystals suitable for X-ray diffraction studies were obtained by the slow evaporation method using ethanol as a solvent.

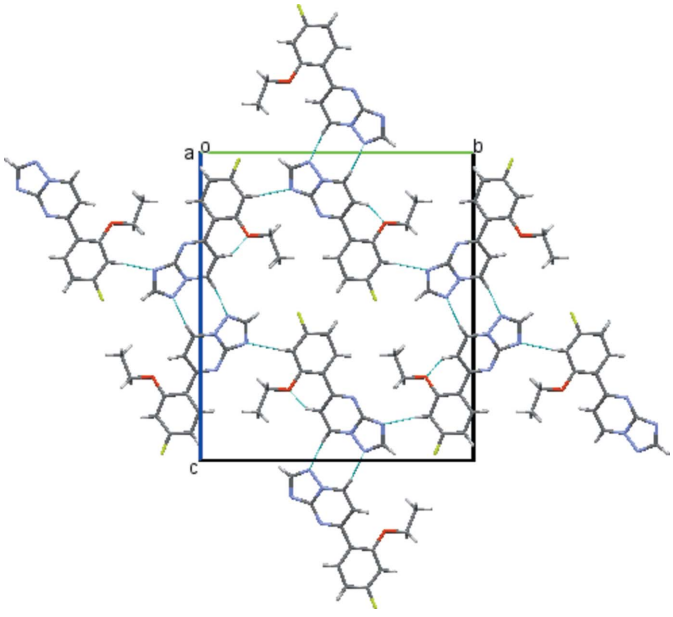


Figure 2
A view along the *a* axis of the crystal packing of the title compound. Hydrogen bonds are drawn as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···N4^{i}	0.93	2.60	3.492 (4)	162
C16—H16···N2^{ii}	0.93	2.49	3.407 (4)	168
C17—H17···O1	0.93	2.39	2.811 (3)	107

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{11}FN_4O$
M_r	258.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	3.9166 (3), 16.6608 (11), 18.8096 (14)
β (°)	93.232 (7)
V (Å^3)	1225.44 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.32 × 0.23 × 0.21
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.966, 0.979
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10165, 2171, 1458
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.061, 0.182, 1.08
No. of reflections	2171
No. of parameters	174
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.26

Computer programs: *CrystalClear SM Expert* (Rigaku, 2011), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161770 [https://doi.org/10.1107/S2414314616017703]

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Crystal data

$C_{13}H_{11}FN_4O$
$M_r = 258.26$
Monoclinic, $P2_1/c$
$a = 3.9166$ (3) Å
$b = 16.6608$ (11) Å
$c = 18.8096$ (14) Å
$\beta = 93.232$ (7)°
$V = 1225.44$ (16) Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.400$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2171 reflections
$\theta = 2.2$–$25.0°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, yellow
0.32 × 0.23 × 0.21 mm

Data collection

Rigaku Saturn724+ diffractometer
profile data from ω–scans
Absorption correction: multi-scan (NUMABS; Rigaku, 1999)
$T_{min} = 0.966$, $T_{max} = 0.979$
10165 measured reflections
2171 independent reflections
1458 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -4 \rightarrow 3$
$k = -19 \rightarrow 19$
$l = -22 \rightarrow 22$
2171 standard reflections

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.182$
$S = 1.08$
2171 reflections
174 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3483P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.023 (4)

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}
F1	0.5117 (7)	0.35926 (13)	0.52220 (10)	0.1067 (9)
O1	0.7212 (5)	0.32471 (10)	0.77097 (10)	0.0567 (6)
N1	1.1380 (5)	0.55271 (12)	0.91136 (11)	0.0459 (6)
N2	1.1990 (6)	0.60040 (14)	0.97014 (12)	0.0574 (7)
N3	0.8590 (5)	0.56069 (12)	0.79604 (11)	0.0464 (6)
N4	0.8923 (6)	0.66793 (13)	0.88210 (12)	0.0575 (7)
C1	0.7414 (9)	0.4662 (2)	0.59031 (16)	0.0701 (9)
H1	0.7509	0.4977	0.5497	0.084*
C2	0.8464 (7)	0.49545 (17)	0.65655 (15)	0.0573 (8)
H2	0.9233	0.5482	0.6605	0.069*
C3	0.8405 (7)	0.44865 (15)	0.71738 (14)	0.0468 (7)
C4	0.7224 (7)	0.36906 (16)	0.71033 (14)	0.0503 (7)
C5	0.6084 (8)	0.33987 (18)	0.64441 (16)	0.0607 (8)
H5	0.5237	0.2879	0.6395	0.073*
C6	0.6231 (9)	0.3892 (2)	0.58674 (17)	0.0707 (9)
C7	0.5874 (8)	0.24438 (16)	0.76772 (18)	0.0627 (9)
H7A	0.7172	0.2116	0.7363	0.075*
H7B	0.3500	0.2448	0.7500	0.075*
C8	0.6178 (9)	0.2119 (2)	0.8419 (2)	0.0806 (11)
H8A	0.4932	0.2458	0.8727	0.121*
H8B	0.8542	0.2106	0.8584	0.121*
H8C	0.5253	0.1586	0.8424	0.121*
C9	0.9482 (6)	0.48491 (14)	0.78707 (13)	0.0436 (6)
C11	0.9540 (6)	0.59429 (15)	0.85917 (13)	0.0447 (7)
C13	1.0413 (8)	0.66697 (18)	0.94821 (16)	0.0619 (8)
H13	1.0338	0.7117	0.9777	0.074*
C16	1.2387 (7)	0.47580 (16)	0.90249 (15)	0.0499 (7)
H16	1.3677	0.4485	0.9378	0.060*
C17	1.1421 (7)	0.44085 (16)	0.83985 (14)	0.0494 (7)
H17	1.2026	0.3879	0.8312	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.152 (2)	0.0993 (17)	0.0656 (13)	0.0004 (14)	−0.0248 (13)	−0.0214 (11)
O1	0.0669 (13)	0.0402 (10)	0.0630 (13)	−0.0063 (9)	0.0050 (10)	−0.0024 (9)
N1	0.0472 (13)	0.0443 (13)	0.0459 (13)	−0.0022 (10)	−0.0002 (10)	0.0059 (10)
N2	0.0691 (17)	0.0534 (15)	0.0491 (14)	−0.0032 (12)	−0.0021 (12)	0.0003 (11)
N3	0.0482 (13)	0.0396 (12)	0.0511 (13)	0.0002 (9)	−0.0006 (11)	0.0040 (9)
N4	0.0680 (17)	0.0449 (14)	0.0591 (15)	0.0030 (11)	−0.0016 (13)	−0.0019 (11)
C1	0.078 (2)	0.078 (2)	0.0537 (19)	0.0043 (18)	−0.0027 (17)	0.0062 (16)
C2	0.0580 (18)	0.0527 (17)	0.0609 (18)	0.0015 (13)	−0.0006 (14)	0.0026 (14)
C3	0.0402 (14)	0.0441 (15)	0.0557 (16)	0.0026 (11)	0.0000 (12)	−0.0014 (12)
C4	0.0502 (16)	0.0438 (15)	0.0572 (17)	0.0068 (12)	0.0053 (13)	−0.0050 (13)
C5	0.0619 (19)	0.0533 (18)	0.067 (2)	0.0035 (14)	0.0006 (16)	−0.0133 (15)

C6	0.079 (2)	0.075 (2)	0.0562 (19)	0.0072 (18)	−0.0095 (17)	−0.0152 (17)
C7	0.0519 (17)	0.0401 (15)	0.097 (2)	−0.0062 (13)	0.0091 (16)	−0.0051 (15)
C8	0.070 (2)	0.0553 (19)	0.116 (3)	−0.0083 (16)	0.007 (2)	0.0229 (19)
C9	0.0376 (14)	0.0410 (14)	0.0521 (15)	−0.0030 (11)	0.0025 (11)	0.0028 (11)
C11	0.0452 (15)	0.0416 (14)	0.0471 (15)	−0.0040 (11)	0.0015 (12)	0.0068 (12)
C13	0.078 (2)	0.0495 (18)	0.0576 (18)	−0.0016 (15)	0.0008 (16)	−0.0050 (14)
C16	0.0480 (16)	0.0453 (15)	0.0559 (17)	0.0032 (12)	−0.0011 (13)	0.0113 (13)
C17	0.0456 (15)	0.0415 (15)	0.0610 (18)	0.0033 (11)	0.0020 (13)	0.0055 (12)

Geometric parameters (Å, º)

F1—C6	1.361 (3)	C3—C4	1.408 (4)
O1—C4	1.359 (3)	C3—C9	1.483 (3)
O1—C7	1.437 (3)	C4—C5	1.382 (4)
N1—N2	1.371 (3)	C5—H5	0.9300
N1—C11	1.372 (3)	C5—C6	1.365 (4)
N1—C16	1.354 (3)	C7—H7A	0.9700
N2—C13	1.324 (4)	C7—H7B	0.9700
N3—C9	1.323 (3)	C7—C8	1.495 (4)
N3—C11	1.346 (3)	C8—H8A	0.9600
N4—C11	1.327 (3)	C8—H8B	0.9600
N4—C13	1.344 (3)	C8—H8C	0.9600
C1—H1	0.9300	C9—C17	1.420 (3)
C1—C2	1.379 (4)	C13—H13	0.9300
C1—C6	1.364 (5)	C16—H16	0.9300
C2—H2	0.9300	C16—C17	1.349 (4)
C2—C3	1.386 (4)	C17—H17	0.9300
C4—O1—C7	119.3 (2)	O1—C7—H7B	110.4
N2—N1—C11	110.2 (2)	O1—C7—C8	106.7 (2)
C16—N1—N2	127.5 (2)	H7A—C7—H7B	108.6
C16—N1—C11	122.3 (2)	C8—C7—H7A	110.4
C13—N2—N1	100.2 (2)	C8—C7—H7B	110.4
C9—N3—C11	116.6 (2)	C7—C8—H8A	109.5
C11—N4—C13	102.3 (2)	C7—C8—H8B	109.5
C2—C1—H1	121.3	C7—C8—H8C	109.5
C6—C1—H1	121.3	H8A—C8—H8B	109.5
C6—C1—C2	117.4 (3)	H8A—C8—H8C	109.5
C1—C2—H2	119.0	H8B—C8—H8C	109.5
C1—C2—C3	121.9 (3)	N3—C9—C3	115.9 (2)
C3—C2—H2	119.0	N3—C9—C17	122.6 (2)
C2—C3—C4	118.2 (2)	C17—C9—C3	121.4 (2)
C2—C3—C9	118.9 (2)	N3—C11—N1	121.9 (2)
C4—C3—C9	122.9 (2)	N4—C11—N1	109.4 (2)
O1—C4—C3	116.7 (2)	N4—C11—N3	128.7 (2)
O1—C4—C5	123.0 (3)	N2—C13—N4	118.0 (3)
C5—C4—C3	120.2 (3)	N2—C13—H13	121.0
C4—C5—H5	120.8	N4—C13—H13	121.0

C6—C5—C4	118.4 (3)	N1—C16—H16	121.7
C6—C5—H5	120.8	C17—C16—N1	116.5 (2)
F1—C6—C1	118.6 (3)	C17—C16—H16	121.7
F1—C6—C5	117.5 (3)	C9—C17—H17	120.0
C1—C6—C5	123.8 (3)	C16—C17—C9	120.0 (2)
O1—C7—H7A	110.4	C16—C17—H17	120.0
O1—C4—C5—C6	−179.5 (3)	C4—C5—C6—F1	179.6 (3)
N1—N2—C13—N4	−1.2 (4)	C4—C5—C6—C1	−0.6 (5)
N1—C16—C17—C9	0.7 (4)	C6—C1—C2—C3	1.4 (5)
N2—N1—C11—N3	−179.6 (2)	C7—O1—C4—C3	176.9 (2)
N2—N1—C11—N4	0.0 (3)	C7—O1—C4—C5	−1.8 (4)
N2—N1—C16—C17	179.0 (2)	C9—N3—C11—N1	0.9 (4)
N3—C9—C17—C16	0.9 (4)	C9—N3—C11—N4	−178.6 (3)
C1—C2—C3—C4	−0.1 (4)	C9—C3—C4—O1	−2.1 (4)
C1—C2—C3—C9	−178.3 (3)	C9—C3—C4—C5	176.6 (3)
C2—C1—C6—F1	178.7 (3)	C11—N1—N2—C13	0.7 (3)
C2—C1—C6—C5	−1.0 (5)	C11—N1—C16—C17	−1.5 (4)
C2—C3—C4—O1	179.8 (2)	C11—N3—C9—C3	179.7 (2)
C2—C3—C4—C5	−1.5 (4)	C11—N3—C9—C17	−1.7 (4)
C2—C3—C9—N3	37.3 (4)	C11—N4—C13—N2	1.2 (4)
C2—C3—C9—C17	−141.3 (3)	C13—N4—C11—N1	−0.6 (3)
C3—C4—C5—C6	1.9 (4)	C13—N4—C11—N3	179.0 (3)
C3—C9—C17—C16	179.4 (2)	C16—N1—N2—C13	−179.7 (3)
C4—O1—C7—C8	−179.5 (2)	C16—N1—C11—N3	0.7 (4)
C4—C3—C9—N3	−140.8 (3)	C16—N1—C11—N4	−179.7 (2)
C4—C3—C9—C17	40.6 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···N4^{i}	0.93	2.60	3.492 (4)	162
C16—H16···N2^{ii}	0.93	2.49	3.407 (4)	168
C17—H17···O1	0.93	2.39	2.811 (3)	107

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*+3, −*y*+1, −*z*+2.