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4-(But-3-ynyloxy)-6-(4-iodo-1*H*-pyrazol-1-yl)pyrimidine

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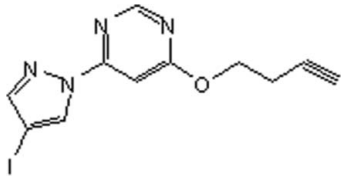
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; R factor = 0.028; wR factor = 0.069; data-to-parameter ratio = 17.6.

In the title compound, $C_{11}H_9IN_4O$, the dihedral angle between the pyrazole and pyrimidine rings is 6.30 (16)°. In the crystal, weak C—H···O interactions link the molecules.

Related literature

For pharmacological background, see: Ma *et al.* (2009); Shiga *et al.* (2003).



Experimental

Crystal data

$C_{11}H_9IN_4O$
M_r = 340.12
Monoclinic, $P2_1/c$
a = 19.511 (4) Å
b = 4.2670 (9) Å
c = 15.129 (3) Å
β = 109.18 (3)°
V = 1189.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.68 mm^{-1}
T = 173 K
0.16 × 0.15 × 0.14 mm

Data collection

Rigaku MM007HF + CCD (Saturn724+) diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
T_{min} = 0.674, T_{max} = 0.705
8082 measured reflections
2713 independent reflections
2577 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.069
S = 1.11
2713 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.95	2.40	3.249 (4)	148
C11—H11···N4^{ii}	0.95	2.52	3.392 (4)	153

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

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5171).

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supporting information

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4-(But-3-ynyloxy)-6-(4-iodo-1*H*-pyrazol-1-yl)pyrimidine

Yong-Hong Li, Ru-Liang Xie, Tao Zhang, Xiang-Dong Mei and Jun Ning

S1. Comment

Heterocyclic niteo acids and their derivatives are important starting materials in chemical synthesis. They are utilized as precursors to obtain various biologically active compounds (e.g. Ma *et al.*, 2009). Pyrazoles are an important class of compounds, which possess widespread pharmacological properties in agrochemicals (e.g. Shiga *et al.*, 2003). Pyrazolo-pyrimidine and related fused heterocycles are of interest as potential bioactive molecules. Recently, we have prepared the title compound (I), which has potential herbicidal activity. The crystal structure of the title compound is shown in Fig.1. The bond lengths and angles show no unusual features.

S2. Experimental

The title compound (0.1 g) was dissolved in a mixed solvent of ethanol and acetone (20 ml) at room temperature: colourless blocks of (I) were obtained through slow evaporation after two weeks.

S3. Refinement

All the hydrogen atoms were placed at their geometrical postion with C—H = 0.93–0.98Å and U_{iso}(H) = 1.2–1.5U_{ep}(C).

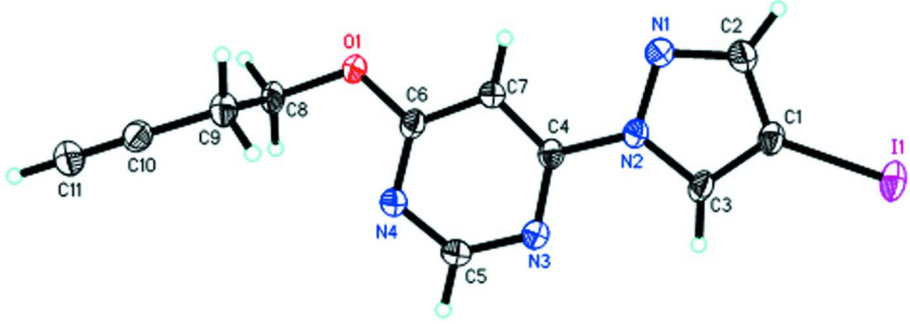

Figure 1

The molecular structure of (I) showing 50% displacement ellipsoids.

4-(But-3-ynyloxy)-6-(4-iodo-1*H*-pyrazol-1-yl)pyrimidine

Crystal data

$C_{11}H_9IN_4O$
M_r = 340.12

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 19.511 (4) Å
b = 4.2670 (9) Å
c = 15.129 (3) Å
β = 109.18 (3)°
V = 1189.6 (4) Å^3
Z = 4
F(000) = 656
D_x = 1.899 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3485 reflections
θ = 2.2–27.5°
μ = 2.68 mm^{-1}
T = 173 K
Block, colourless
0.16 × 0.15 × 0.14 mm

Data collection

Rigaku MM007HF + CCD (Saturn724+) diffractometer
Radiation source: Rotating Anode
Confocal monochromator
Detector resolution: 28.5714 pixels mm^{-1}
ω scans at fixed χ = 45°
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
T_{min} = 0.674, T_{max} = 0.705

8082 measured reflections
2713 independent reflections
2577 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −17→25
k = −3→5
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.069
S = 1.11
2713 reflections
154 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 1.6155P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
I1	0.073003 (10)	0.85153 (4)	0.354299 (12)	0.02946 (8)
O1	0.28021 (11)	−0.1265 (5)	0.89982 (14)	0.0257 (4)
N1	0.12131 (13)	0.5308 (7)	0.63643 (17)	0.0292 (5)
N2	0.17939 (12)	0.4249 (6)	0.61341 (15)	0.0217 (5)
N3	0.29051 (13)	0.1759 (6)	0.65001 (17)	0.0281 (5)
N4	0.34178 (13)	−0.1049 (6)	0.79327 (17)	0.0258 (5)
C1	0.11287 (15)	0.6716 (6)	0.48870 (19)	0.0236 (6)
C2	0.08146 (17)	0.6802 (7)	0.5602 (2)	0.0304 (7)

H2	0.0368	0.7814	0.5544	0.037*
C3	0.17555 (14)	0.5053 (7)	0.52485 (18)	0.0232 (5)
H3	0.2097	0.4556	0.4944	0.028*
C4	0.23353 (14)	0.2441 (7)	0.67752 (18)	0.0202 (5)
C5	0.34119 (16)	0.0049 (8)	0.7106 (2)	0.0323 (7)
H5	0.3823	−0.0458	0.6929	0.039*
C6	0.28450 (14)	−0.0285 (7)	0.81731 (18)	0.0218 (5)
C7	0.22707 (15)	0.1515 (6)	0.7616 (2)	0.0213 (5)
H7	0.1866	0.2062	0.7801	0.026*
C8	0.33899 (15)	−0.3164 (7)	0.9591 (2)	0.0254 (6)
H8A	0.3574	−0.4565	0.9198	0.030*
H8B	0.3209	−0.4486	1.0004	0.030*
C9	0.40039 (15)	−0.1086 (7)	1.0183 (2)	0.0266 (6)
H9A	0.4196	0.0174	0.9768	0.032*
H9B	0.3813	0.0378	1.0554	0.032*
C10	0.45916 (15)	−0.2943 (7)	1.0816 (2)	0.0255 (6)
C11	0.50610 (16)	−0.4493 (8)	1.1313 (2)	0.0322 (6)
H11	0.5439	−0.5741	1.1714	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03776 (13)	0.02646 (12)	0.01837 (11)	0.00202 (7)	0.00135 (8)	0.00445 (7)
O1	0.0241 (10)	0.0348 (11)	0.0192 (9)	0.0074 (8)	0.0083 (8)	0.0097 (8)
N1	0.0288 (12)	0.0389 (14)	0.0208 (12)	0.0103 (11)	0.0094 (9)	0.0032 (11)
N2	0.0217 (11)	0.0264 (11)	0.0155 (10)	0.0027 (9)	0.0039 (8)	0.0010 (9)
N3	0.0245 (12)	0.0402 (15)	0.0200 (12)	0.0071 (10)	0.0079 (10)	0.0063 (10)
N4	0.0227 (11)	0.0348 (13)	0.0193 (11)	0.0067 (9)	0.0061 (9)	0.0024 (10)
C1	0.0281 (14)	0.0230 (14)	0.0167 (13)	0.0016 (10)	0.0035 (11)	0.0009 (10)
C2	0.0287 (15)	0.0376 (17)	0.0235 (15)	0.0106 (12)	0.0063 (12)	0.0030 (12)
C3	0.0251 (13)	0.0276 (14)	0.0149 (12)	0.0001 (11)	0.0039 (10)	0.0035 (10)
C4	0.0204 (12)	0.0207 (12)	0.0167 (12)	−0.0006 (10)	0.0025 (9)	−0.0013 (10)
C5	0.0265 (14)	0.048 (2)	0.0252 (14)	0.0129 (13)	0.0124 (11)	0.0054 (13)
C6	0.0225 (12)	0.0248 (13)	0.0160 (12)	−0.0009 (10)	0.0037 (10)	0.0009 (10)
C7	0.0199 (12)	0.0253 (14)	0.0186 (13)	0.0031 (9)	0.0064 (10)	0.0015 (10)
C8	0.0262 (14)	0.0263 (14)	0.0208 (13)	0.0046 (11)	0.0041 (11)	0.0091 (11)
C9	0.0270 (14)	0.0268 (14)	0.0232 (14)	0.0032 (11)	0.0042 (11)	0.0041 (11)
C10	0.0251 (14)	0.0294 (14)	0.0221 (13)	−0.0019 (11)	0.0076 (11)	0.0010 (11)
C11	0.0270 (15)	0.0389 (17)	0.0289 (15)	0.0044 (13)	0.0066 (12)	0.0056 (14)

Geometric parameters (Å, º)

I1—C1	2.071 (3)	C3—H3	0.9500
O1—C6	1.345 (3)	C4—C7	1.376 (4)
O1—C8	1.449 (3)	C5—H5	0.9500
N1—C2	1.324 (4)	C6—C7	1.391 (4)
N1—N2	1.367 (3)	C7—H7	0.9500
N2—C3	1.361 (3)	C8—C9	1.523 (4)

N2—C4	1.406 (3)	C8—H8A	0.9900
N3—C5	1.325 (4)	C8—H8B	0.9900
N3—C4	1.341 (3)	C9—C10	1.462 (4)
N4—C6	1.325 (3)	C9—H9A	0.9900
N4—C5	1.332 (4)	C9—H9B	0.9900
C1—C3	1.363 (4)	C10—C11	1.178 (4)
C1—C2	1.408 (4)	C11—H11	0.9500
C2—H2	0.9500		

C6—O1—C8	118.1 (2)	N4—C5—H5	115.9
C2—N1—N2	103.7 (2)	N4—C6—O1	119.6 (2)
C3—N2—N1	112.7 (2)	N4—C6—C7	123.6 (2)
C3—N2—C4	127.1 (2)	O1—C6—C7	116.8 (2)
N1—N2—C4	120.2 (2)	C4—C7—C6	114.7 (2)
C5—N3—C4	114.3 (2)	C4—C7—H7	122.6
C6—N4—C5	115.0 (2)	C6—C7—H7	122.6
C3—C1—C2	105.4 (2)	O1—C8—C9	110.4 (2)
C3—C1—I1	125.8 (2)	O1—C8—H8A	109.6
C2—C1—I1	128.8 (2)	C9—C8—H8A	109.6
N1—C2—C1	112.2 (3)	O1—C8—H8B	109.6
N1—C2—H2	123.9	C9—C8—H8B	109.6
C1—C2—H2	123.9	H8A—C8—H8B	108.1
N2—C3—C1	106.2 (2)	C10—C9—C8	111.5 (2)
N2—C3—H3	126.9	C10—C9—H9A	109.3
C1—C3—H3	126.9	C8—C9—H9A	109.3
N3—C4—C7	124.2 (2)	C10—C9—H9B	109.3
N3—C4—N2	114.6 (2)	C8—C9—H9B	109.3
C7—C4—N2	121.2 (2)	H9A—C9—H9B	108.0
N3—C5—N4	128.2 (3)	C11—C10—C9	178.6 (4)
N3—C5—H5	115.9	C10—C11—H11	180.0

C2—N1—N2—C3	−0.3 (3)	N1—N2—C4—C7	4.6 (4)
C2—N1—N2—C4	−178.2 (3)	C4—N3—C5—N4	0.3 (5)
N2—N1—C2—C1	0.1 (4)	C6—N4—C5—N3	−0.8 (5)
C3—C1—C2—N1	0.1 (4)	C5—N4—C6—O1	−179.6 (3)
I1—C1—C2—N1	177.7 (2)	C5—N4—C6—C7	0.3 (4)
N1—N2—C3—C1	0.4 (3)	C8—O1—C6—N4	−0.6 (4)
C4—N2—C3—C1	178.1 (3)	C8—O1—C6—C7	179.6 (2)
C2—C1—C3—N2	−0.3 (3)	N3—C4—C7—C6	−1.3 (4)
I1—C1—C3—N2	−177.96 (19)	N2—C4—C7—C6	179.4 (2)
C5—N3—C4—C7	0.8 (4)	N4—C6—C7—C4	0.7 (4)
C5—N3—C4—N2	−179.8 (3)	O1—C6—C7—C4	−179.5 (2)
C3—N2—C4—N3	7.6 (4)	C6—O1—C8—C9	84.8 (3)
N1—N2—C4—N3	−174.8 (3)	O1—C8—C9—C10	177.7 (2)
C3—N2—C4—C7	−173.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.95	2.40	3.249 (4)	148
C11—H11···N4^{ii}	0.95	2.52	3.392 (4)	153

Symmetry codes: (i) x, $-y+1/2$, $z-1/2$; (ii) $-x+1$, $-y-1$, $-z+2$.