organic compounds

2-(2-Fluoro-4-nitrophenoxy)-3-nitro-pyridine

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In the title compound, $C_{11}H_6FN_3O_5$, the dihedral angle between the aromatic rings is 72.4 (3)°. The NO_2 groups form dihedral angles of 40.8 (2) and 4.8 (2)°, respectively, with the attached pyridine and benzene rings. The crystal structure features π–π stacking between centrosymmetrically related pairs of pyridine rings [centroid–centroid separation = 3.800 (3) Å].

Related literature

For applications and the biological activity of 2-phenoxy-pyridine, see: Chao *et al.* (2013).
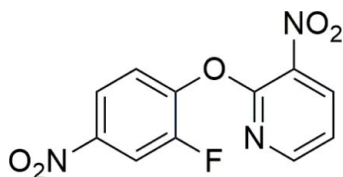
Experimental

Crystal data

$C_{11}H_6FN_3O_5$
$M_r = 279.19$

Monoclinic, $P2_1/c$
$a = 7.5275\ (15)$ Å
$b = 21.804\ (4)$ Å
$c = 7.1681\ (14)$ Å
$\beta = 101.07\ (3)°$
$V = 1154.6\ (4)$ Å^3

$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.14$ mm^{-1}
$T = 293$ K
$0.41 \times 0.36 \times 0.22$ mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.945$, $T_{max} = 0.970$

10606 measured reflections
2611 independent reflections
1351 reflections with $I > 2\sigma(I)$
$R_{int} = 0.083$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.140$
$S = 0.94$
2611 reflections

181 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2504).

References

Chao, H., Turdi, H., Herpin, T. F., Roberge, J. Y., Liu, Y., Schnur, D. M., Poss, M. A., Rehfuss, R., Hua, J., Wu, Q., Price, L. A., Abell, L. M., Schumacher, W. A., Bostwick, J. S., Steinbacher, T. E., Stewart, A. B., Ogletree, M. L., Huang, C. S., Chang, M., Cacace, A. M., Arcuri, M. J., Celani, D., Wexler, R. R. & Lawrence, R. M. (2013). *J. Med. Chem.* **56**, 1704–1714.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

2-(2-Fluoro-4-nitrophenoxy)-3-nitropyridine

Lili Cui and Xingquan He

1. Comment

2-Phenoxypyridines have been shown to be small molecule P2Y1 antagonists (Chao *et al.* 2013). Here, the crystal structure of 2-(2-fluoro-4-nitrophenoxy)- 3-nitropyridine is reported.

2. Experimental

To a solution of 2-chloro-3-nitropyridine (1.0 g, 6.3 mmol) and 2-fluoro-4-nitrophenol (1.48 g, 9.45 mmol) in toluene (35 ml) was added *N,N*-diisopropylethylamine (3.3 ml, 18.9 mmol). The reaction mixture was stirred for 48 h under reflux. The reaction mixture was concentrated *in vacuo*, diluted with water, and extracted with EtOAc. The organic phase was washed with brine, dried over anhydrous $MgSO_4$, and concentrated *in vacuo* to yield the crude product as a solid. Purification by recrystallization from methanol gave the desired product, 2-(2-fluoro-4-nitrophenoxy)-3- nitropyridine (yellow solid, 0.80 g, 46%, 97.1–98.4 ^{o}C). ^{1}H NMR (DMSO-*d6*, 300 Hz), 8.70 (dd, J = 8.1, 1.8 Hz, 1H), 8.47 (dd, J = 4.8, 1.8 Hz, 1H), 8.41 (dd, J = 10.2, 2.7 Hz, 1H), 8.25–8.20 (m, 1H), 7.80–7.74 (m 1H), 7.54–7.50 (m, 1H); ES–MS: 279.8 $[(M + H^+)]$. Crystals of the title compound for X-ray diffraction were obtained by slow evaporation of $MeOH/CH_2Cl_2$ solution.

3. Refinement

All hydrogen atoms were fixed at calculated positions and refined by using a riding model with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *RAPID-AUTO* (Rigaku, 1998); 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: *SHELXTL* (Sheldrick, 2008).
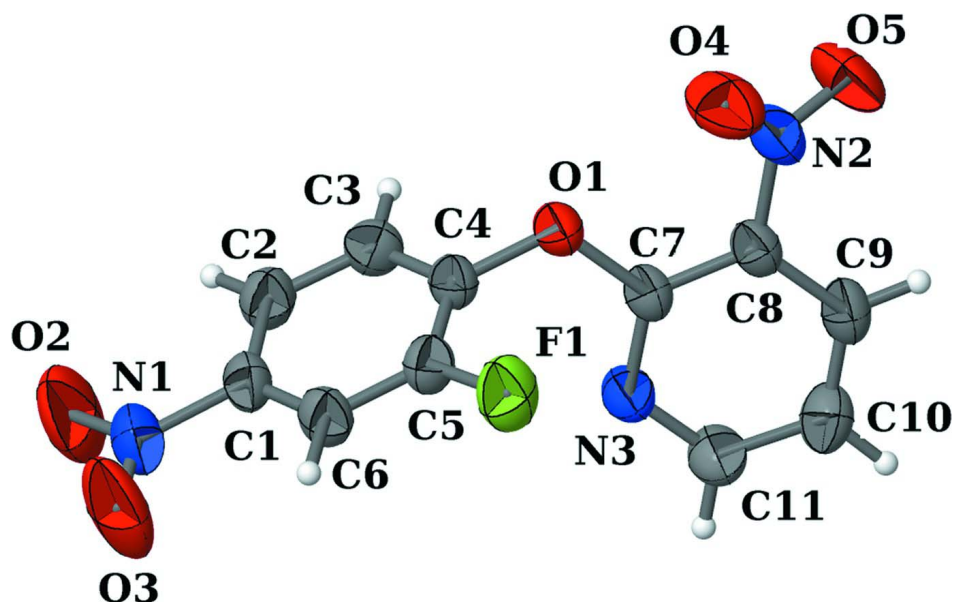
Figure 1

The molecular structure of the title compound. Displacement ellipsoids are shown at the 50% probability level.

2-(2-Fluoro-4-nitrophenoxy)-3-nitropyridine

Crystal data

$C_{11}H_6FN_3O_5$	$F(000) = 568$
$M_r = 279.19$	$D_x = 1.606$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point = 370.1–371.4 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.5275$ (15) Å	Cell parameters from 7455 reflections
$b = 21.804$ (4) Å	$\theta = 3.1$–27.7°
$c = 7.1681$ (14) Å	$\mu = 0.14$ mm^{-1}
$\beta = 101.07$ (3)°	$T = 293$ K
$V = 1154.6$ (4) Å^3	Block, colourless
$Z = 4$	$0.41 \times 0.36 \times 0.22$ mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer	10606 measured reflections
Radiation source: fine-focus sealed tube	2611 independent reflections
Graphite monochromator	1351 reflections with $I > 2\sigma(I)$
Detector resolution: 0.1 pixels mm^{-1}	$R_{int} = 0.083$
oscillation scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$h = -9 \rightarrow 9$
	$k = -28 \rightarrow 28$
$T_{min} = 0.945$, $T_{max} = 0.970$	$l = -9 \rightarrow 8$

Refinement

Refinement on F^2	181 parameters
Least-squares matrix: full	0 restraints
$R[F^2 > 2\sigma(F^2)] = 0.062$	Primary atom site location: structure-invariant direct methods
$wR(F^2) = 0.140$	
$S = 0.94$	Secondary atom site location: difference Fourier map
2611 reflections	

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ 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}
C5	0.7119 (3)	0.37835 (11)	0.5574 (3)	0.0470 (6)
C4	0.8649 (3)	0.34302 (12)	0.6136 (3)	0.0453 (6)
C1	0.5475 (3)	0.29000 (12)	0.4494 (3)	0.0492 (6)
C3	0.8592 (3)	0.28155 (12)	0.5825 (3)	0.0535 (7)
H3	0.9634	0.2580	0.6169	0.064*
C6	0.5501 (3)	0.35242 (12)	0.4752 (3)	0.0516 (7)
H6	0.4464	0.3761	0.4385	0.062*
C2	0.6968 (3)	0.25381 (13)	0.4988 (4)	0.0554 (7)
H2	0.6904	0.2117	0.4772	0.066*
C7	1.0406 (3)	0.39397 (11)	0.8728 (3)	0.0430 (6)
C8	1.1950 (3)	0.42593 (11)	0.9561 (3)	0.0446 (6)
C9	1.2093 (3)	0.44800 (12)	1.1372 (3)	0.0524 (6)
H9	1.3124	0.4691	1.1956	0.063*
C10	1.0688 (4)	0.43849 (13)	1.2313 (3)	0.0598 (7)
H10	1.0732	0.4535	1.3536	0.072*
C11	0.9221 (4)	0.40613 (13)	1.1387 (3)	0.0580 (7)
H11	0.8268	0.3995	1.2018	0.070*
F1	0.7237 (2)	0.43888 (7)	0.5831 (2)	0.0720 (5)
O1	1.0278 (2)	0.37145 (9)	0.6923 (2)	0.0550 (5)
O4	1.3069 (3)	0.45113 (12)	0.6873 (3)	0.0846 (7)
O5	1.4970 (2)	0.43310 (12)	0.9461 (4)	0.0937 (8)
O2	0.3673 (3)	0.20648 (13)	0.3537 (5)	0.1251 (11)
O3	0.2448 (3)	0.29424 (13)	0.3109 (4)	0.1255 (12)
N3	0.9075 (3)	0.38343 (10)	0.9628 (3)	0.0508 (5)
N2	1.3435 (3)	0.43708 (10)	0.8543 (4)	0.0593 (6)
N1	0.3735 (3)	0.26136 (14)	0.3662 (4)	0.0716 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C5	0.0507 (13)	0.0405 (14)	0.0488 (12)	−0.0032 (11)	0.0074 (11)	−0.0045 (10)
C4	0.0391 (12)	0.0565 (16)	0.0385 (11)	−0.0039 (11)	0.0029 (10)	−0.0049 (10)

C1	0.0444 (13)	0.0523 (16)	0.0473 (13)	−0.0054 (11)	−0.0005 (11)	−0.0089 (11)
C3	0.0452 (13)	0.0559 (17)	0.0568 (14)	0.0103 (11)	0.0034 (12)	−0.0036 (12)
C6	0.0428 (13)	0.0531 (17)	0.0541 (14)	0.0053 (11)	−0.0028 (11)	−0.0027 (11)
C2	0.0579 (15)	0.0461 (16)	0.0600 (15)	0.0013 (12)	0.0060 (13)	−0.0083 (11)
C7	0.0374 (12)	0.0461 (14)	0.0429 (11)	−0.0031 (10)	0.0009 (10)	−0.0003 (10)
C8	0.0356 (11)	0.0426 (14)	0.0526 (13)	−0.0010 (10)	0.0008 (11)	0.0020 (10)
C9	0.0477 (13)	0.0484 (15)	0.0546 (14)	−0.0047 (11)	−0.0060 (12)	−0.0031 (11)
C10	0.0678 (17)	0.0625 (18)	0.0448 (13)	−0.0060 (14)	0.0003 (13)	−0.0058 (12)
C11	0.0575 (15)	0.0730 (19)	0.0447 (13)	−0.0122 (14)	0.0126 (12)	−0.0008 (12)
F1	0.0736 (10)	0.0425 (9)	0.0955 (11)	−0.0038 (7)	0.0051 (9)	−0.0074 (8)
O1	0.0392 (9)	0.0785 (13)	0.0472 (9)	−0.0119 (8)	0.0080 (7)	−0.0158 (8)
O4	0.0682 (13)	0.116 (2)	0.0747 (13)	−0.0145 (13)	0.0261 (11)	0.0135 (13)
O5	0.0355 (10)	0.121 (2)	0.1207 (18)	−0.0093 (11)	0.0043 (11)	−0.0072 (15)
O2	0.0874 (18)	0.0756 (19)	0.202 (3)	−0.0328 (14)	0.0027 (18)	−0.0372 (19)
O3	0.0584 (14)	0.109 (2)	0.183 (3)	−0.0043 (14)	−0.0421 (17)	−0.0136 (19)
N3	0.0449 (11)	0.0617 (14)	0.0457 (10)	−0.0105 (10)	0.0086 (9)	−0.0026 (9)
N2	0.0392 (11)	0.0564 (15)	0.0805 (16)	−0.0078 (10)	0.0073 (11)	−0.0048 (12)
N1	0.0568 (15)	0.0735 (19)	0.0797 (16)	−0.0166 (13)	0.0011 (13)	−0.0196 (14)

Geometric parameters (Å, º)

C5—F1	1.333 (3)	C7—C8	1.388 (3)
C5—C6	1.369 (3)	C8—C9	1.369 (3)
C5—C4	1.380 (3)	C8—N2	1.468 (3)
C4—C3	1.358 (3)	C9—C10	1.375 (4)
C4—O1	1.393 (3)	C9—H9	0.9300
C1—C2	1.363 (3)	C10—C11	1.370 (3)
C1—C6	1.373 (3)	C10—H10	0.9300
C1—N1	1.470 (3)	C11—N3	1.339 (3)
C3—C2	1.392 (3)	C11—H11	0.9300
C3—H3	0.9300	O4—N2	1.215 (3)
C6—H6	0.9300	O5—N2	1.218 (2)
C2—H2	0.9300	O2—N1	1.200 (3)
C7—N3	1.312 (3)	O3—N1	1.209 (3)
C7—O1	1.370 (3)		
F1—C5—C6	119.9 (2)	C9—C8—C7	119.4 (2)
F1—C5—C4	118.9 (2)	C9—C8—N2	118.9 (2)
C6—C5—C4	121.3 (2)	C7—C8—N2	121.6 (2)
C3—C4—C5	120.3 (2)	C8—C9—C10	119.0 (2)
C3—C4—O1	120.3 (2)	C8—C9—H9	120.5
C5—C4—O1	119.3 (2)	C10—C9—H9	120.5
C2—C1—C6	123.3 (2)	C11—C10—C9	117.6 (2)
C2—C1—N1	119.0 (2)	C11—C10—H10	121.2
C6—C1—N1	117.7 (2)	C9—C10—H10	121.2
C4—C3—C2	119.8 (2)	N3—C11—C10	124.0 (3)
C4—C3—H3	120.1	N3—C11—H11	118.0
C2—C3—H3	120.1	C10—C11—H11	118.0
C5—C6—C1	117.1 (2)	C7—O1—C4	116.05 (18)
C5—C6—H6	121.4	C7—N3—C11	117.8 (2)

C1—C6—H6	121.4	O4—N2—O5	124.2 (2)
C1—C2—C3	118.2 (3)	O4—N2—C8	118.75 (19)
C1—C2—H2	120.9	O5—N2—C8	117.0 (2)
C3—C2—H2	120.9	O2—N1—O3	123.3 (3)
N3—C7—O1	118.66 (19)	O2—N1—C1	118.2 (3)
N3—C7—C8	122.1 (2)	O3—N1—C1	118.4 (3)
O1—C7—C8	119.2 (2)		