Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Lili Cui* and Xingquan He

Department of Chemistry and Chemical Engineering, Changchun University of Science and Technology, Changchun 130022, People's Republic of China
Correspondence e-mail: cuilili1127@gmail.com

Received 11 November 2013; accepted 19 December 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.140; data-to-parameter ratio = 14.4.

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-phenoxypyridine, 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
β = 101.07 (3)°
V = 1154.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.41 × 0.36 × 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*.

The project was sponsored by the Natural Science Foundation of China (No. 21273024) and the Natural Science Foundation of Jilin Province, China (No. 201215135).

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.

supporting information

Acta Cryst. (2014). E**70**, o100 [https://doi.org/10.1107/S1600536813034181]

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

Lili Cui and Xingquan He

S1. 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.

S2. 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 °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.

S3. 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).

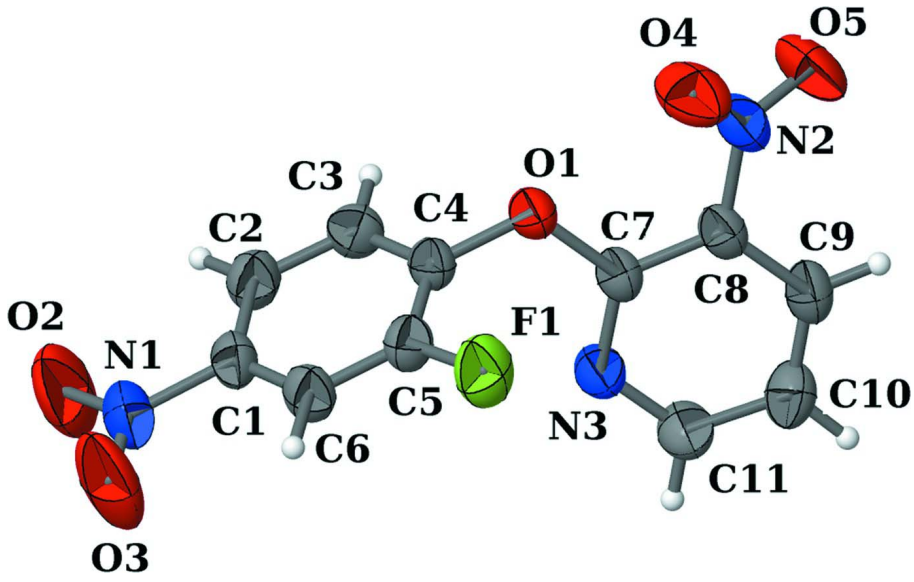

Figure 1

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

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

Data collection

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

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.140$
$S = 0.94$
2611 reflections
181 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
$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2]$
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)		