IUCrData

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3-Difluoromethyl-5-[4-(trifluoromethyl)phenyl]-isoxazole

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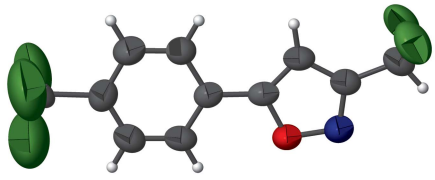
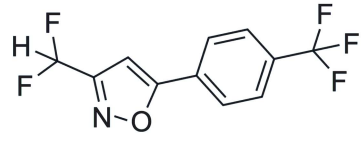
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $C_{11}H_6F_5NO$, lies on a mirror plane in the orthorhombic unit cell, with only two F atoms each of the difluoro and trifluoromethyl substituents lying out of the plane. In the crystal, molecules are linked by C—H···O, C—H···N and C—H···F hydrogen bonds, forming a layer structure parallel to the (001) plane. Weak π–π interactions promote the formation of a three-dimensional network.

3D view



Chemical scheme



Structure description

Nitrogen heterocycles are important scaffolds in bioactive natural and synthesized compounds (Fried *et al.*, 2001; Baraldi *et al.*, 2008; Vitaku *et al.*, 2014). Moreover, organic molecules with fluoroalkyl substituents often impart desirable physical and biological properties (Wang *et al.*, 2014).

The title compound crystallizes in the orthorhombic space group *Pbcm* with most atoms of the molecule in the mirror plane (Fig. 1). Only the F1 and F3 atoms of the difluoro and trifluoromethyl substituents lie out of this plane. In the crystal, molecules are linked by a combination of C—H···O, C—H···N and C—H···F hydrogen bonds (Table 1) together with a weak π–π stacking interaction between C5–C10 rings [centroid–centroid separation = 3.904 (2) Å] (Fig. 2). In more detail, a chain running along the [010] direction forms through C3—H3···O1 and C3—H3···N1 hydrogen bonds, Table 1. These [010] chains are joined by the C1—H1···F2 hydrogen bonds, forming a layer structure parallel to the (001) plane. Finally, molecules in these (001) layers are linked by the weak π–π stacking interactions to generate a three-dimensional network.

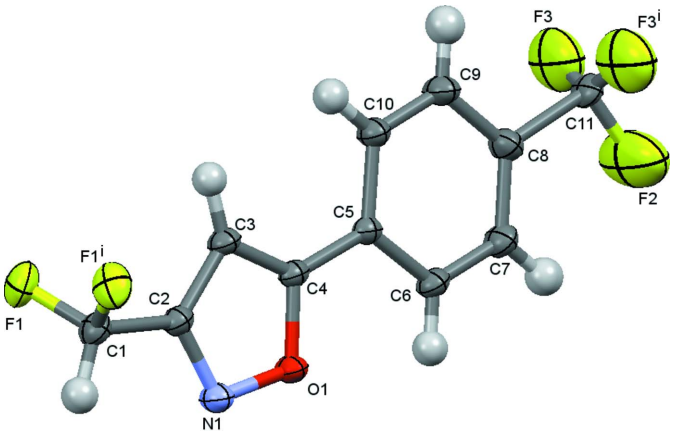


Figure 1
The structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $x, y, -z+\frac{1}{2}$.]

Synthesis and crystallization

The title compound was synthesized by a one-pot 'click synthesis' of fluoroalkylated isoxazoles from commercially avalilable 1-ethynyl-4-(trifluoromethyl)benzene and 2,2-difluoroethanamine, and the isolation and characterization have been reported previously (Zhang *et al.*, 2018). Crystals for X-ray data collection were obtained by slow evaporation of a petroleum ether/ethylacetate (*v*:*v* = 100:1) solution.

Refinement

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

Funding information

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.69	3.581 (7)	160
C3—H3···N1^{i}	0.93	2.62	3.534 (6)	170
C1—H1···F2^{ii}	0.98	2.65	3.258 (6)	121

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_6F_5NO$
M_r	263.17
Crystal system, space group	Orthorhombic, *Pbcm*
Temperature (K)	293
a, *b*, *c* (Å)	13.1081 (13), 11.1491 (13), 7.1798 (11)
V (Å^3)	1049.3 (2)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.17
Crystal size (mm)	0.24 × 0.22 × 0.20
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.961, 0.967
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5659, 1066, 707
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.605
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.079, 0.292, 1.07
No. of reflections	1066
No. of parameters	103
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.49

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.* 2009).

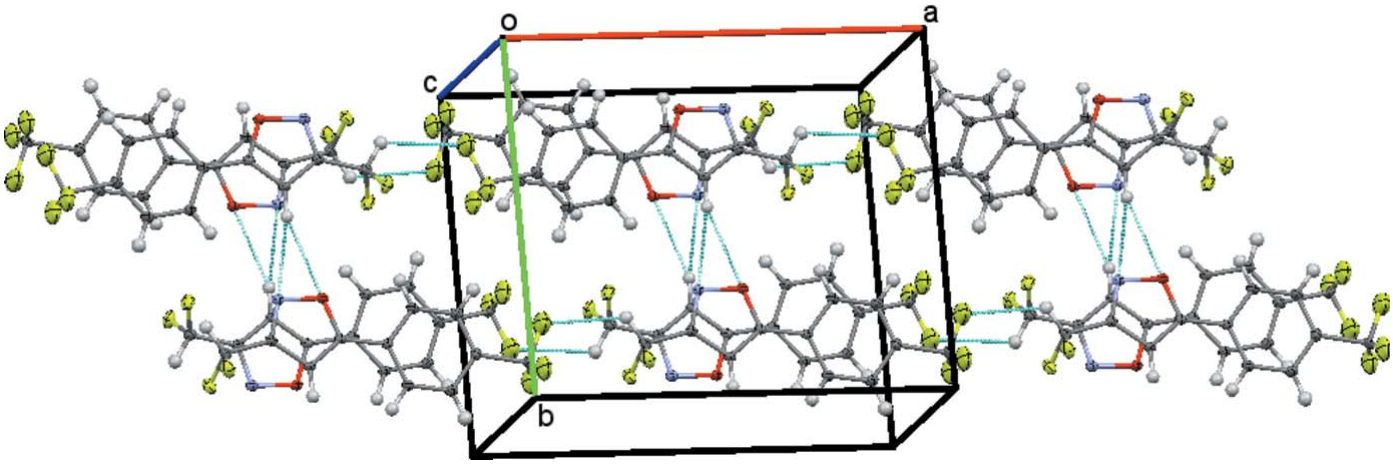

Figure 2
Overall packing of the title compound viewed along the *c*-axis direction. Hydrogen bonds are drawn as dashed lines.

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References

Baraldi, P. G., Tabrizi, M. A., Gessi, S. & Borea, P. A. (2008). *Chem. Rev.* **108**, 238–263.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Fried, L. E., Manaa, M. R., Pagoria, P. F. & Simpson, R. L. (2001). *Annu. Rev. Mater. Res.* **31**, 291–321.
Rigaku OD (2015). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Vitaku, E., Smith, D. T. & Njardarson, J. T. (2014). *J. Med. Chem.* **57**, 10257–10274.
Wang, J., Sánchez-Roselló, M., Aceña, J. L., del Pozo, C., Sorochinsky, A. E., Fustero, S., Soloshonok, V. A. & Liu, H. (2014). *Chem. Rev.* **114**, 2432–2506.
Zhang, X.-W., Hu, W.-L., Chen, S. & Hu, X.-G. (2018). *Org. Lett.* **20**, 860–863.

full crystallographic data

IUCrData (2018). **3**, x180445 [https://doi.org/10.1107/S2414314618004455]

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Crystal data

$C_{11}H_6F_5NO$
$M_r = 263.17$
Orthorhombic, *Pbcm*
$a = 13.1081$ (13) Å
$b = 11.1491$ (13) Å
$c = 7.1798$ (11) Å
$V = 1049.3$ (2) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.666$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1887 reflections
$\theta = 3.7$–$26.4°$
$\mu = 0.17$ mm^{-1}
$T = 293$ K
Block, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.0353 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
$T_{min} = 0.961$, $T_{max} = 0.967$
5659 measured reflections
1066 independent reflections
707 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.5°$, $\theta_{min} = 3.6°$
$h = -15\rightarrow15$
$k = -13\rightarrow12$
$l = -7\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.292$
$S = 1.07$
1066 reflections
103 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1997P)^2 + 0.0686P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ 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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.5503 (3)	0.1715 (3)	0.2500	0.0724 (13)
O1	0.4442 (3)	0.1696 (2)	0.2500	0.0689 (11)
F1	0.7082 (2)	0.3834 (2)	0.3982 (4)	0.1078 (12)
C4	0.4094 (4)	0.2842 (3)	0.2500	0.0544 (12)
C5	0.2998 (4)	0.3006 (4)	0.2500	0.0542 (12)
C3	0.4895 (4)	0.3580 (4)	0.2500	0.0650 (14)
H3	0.4882	0.4414	0.2500	0.078*
C9	0.1574 (4)	0.4343 (5)	0.2500	0.0844 (17)
H9	0.1312	0.5118	0.2500	0.101*
C8	0.0914 (4)	0.3356 (4)	0.2500	0.0794 (16)
C6	0.2315 (4)	0.2038 (4)	0.2500	0.0683 (14)
H6	0.2568	0.1259	0.2500	0.082*
C10	0.2593 (4)	0.4164 (4)	0.2500	0.0704 (15)
H10	0.3031	0.4821	0.2500	0.084*
C11	−0.0320 (6)	0.3614 (6)	0.2500	0.0780 (19)
C7	0.1293 (5)	0.2218 (4)	0.2500	0.0797 (17)
H7	0.0851	0.1565	0.2500	0.096*
C1	0.6860 (4)	0.3158 (5)	0.2500	0.0734 (15)
H1	0.7276	0.2428	0.2500	0.088*
C2	0.5753 (4)	0.2845 (4)	0.2500	0.0607 (13)
F2	−0.0709 (6)	0.2549 (10)	0.2500	0.327 (7)
F3	−0.0478 (5)	0.3962 (6)	0.3638 (11)	0.260 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.066 (3)	0.053 (3)	0.098 (3)	0.0016 (18)	0.000	0.000
O1	0.064 (2)	0.0454 (19)	0.097 (3)	−0.0009 (14)	0.000	0.000
F1	0.0887 (19)	0.124 (2)	0.111 (2)	−0.0307 (14)	−0.0063 (14)	−0.0265 (15)
C4	0.073 (3)	0.043 (2)	0.047 (2)	−0.0011 (19)	0.000	0.000
C5	0.064 (3)	0.048 (2)	0.050 (2)	0.003 (2)	0.000	0.000
C3	0.074 (3)	0.041 (2)	0.080 (3)	−0.004 (2)	0.000	0.000
C9	0.079 (4)	0.055 (3)	0.118 (5)	0.005 (2)	0.000	0.000
C8	0.066 (3)	0.072 (4)	0.100 (4)	0.009 (3)	0.000	0.000
C6	0.078 (4)	0.047 (3)	0.080 (3)	−0.009 (2)	0.000	0.000
C10	0.066 (3)	0.053 (3)	0.092 (4)	−0.001 (2)	0.000	0.000
C11	0.058 (4)	0.060 (3)	0.115 (6)	−0.021 (3)	0.000	0.000
C7	0.074 (4)	0.062 (3)	0.103 (4)	−0.014 (3)	0.000	0.000
C1	0.070 (3)	0.063 (3)	0.088 (4)	−0.015 (3)	0.000	0.000
C2	0.069 (3)	0.051 (3)	0.062 (3)	−0.005 (2)	0.000	0.000

F2	0.127 (6)	0.223 (8)	0.63 (2)	0.010 (7)	0.000	0.000
F3	0.101 (3)	0.264 (6)	0.414 (15)	−0.013 (4)	−0.016 (5)	−0.005 (5)

Geometric parameters (Å, º)

N1—C2	1.302 (5)	C8—C7	1.363 (7)
N1—O1	1.392 (6)	C8—C11	1.643 (10)
O1—C4	1.356 (5)	C6—C7	1.354 (8)
F1—C1	1.336 (3)	C6—H6	0.9300
C4—C3	1.334 (7)	C10—H10	0.9300
C4—C5	1.449 (7)	C11—F3^{i}	0.928 (8)
C5—C10	1.395 (6)	C11—F3	0.928 (8)
C5—C6	1.402 (7)	C11—F2	1.293 (13)
C3—C2	1.393 (7)	C7—H7	0.9300
C3—H3	0.9300	C1—F1^{i}	1.336 (3)
C9—C10	1.351 (7)	C1—C2	1.492 (7)
C9—C8	1.400 (7)	C1—H1	0.9800
C9—H9	0.9300	F3—F3^{i}	1.634 (16)
C2—N1—O1	105.4 (3)	C9—C10—H10	119.6
C4—O1—N1	108.8 (3)	C5—C10—H10	119.6
C3—C4—O1	108.5 (5)	F3^{i}—C11—F3	123.3 (15)
C3—C4—C5	134.6 (4)	F3^{i}—C11—F2	107.2 (7)
O1—C4—C5	116.9 (4)	F3—C11—F2	107.2 (7)
C10—C5—C6	118.0 (5)	F3^{i}—C11—C8	107.1 (8)
C10—C5—C4	119.6 (4)	F3—C11—C8	107.1 (8)
C6—C5—C4	122.4 (4)	F2—C11—C8	103.1 (6)
C4—C3—C2	105.8 (4)	C6—C7—C8	119.9 (5)
C4—C3—H3	127.1	C6—C7—H7	120.1
C2—C3—H3	127.1	C8—C7—H7	120.1
C10—C9—C8	119.7 (5)	F1—C1—F1^{i}	105.6 (4)
C10—C9—H9	120.2	F1—C1—C2	110.1 (3)
C8—C9—H9	120.2	F1^{i}—C1—C2	110.1 (3)
C7—C8—C9	120.5 (5)	F1—C1—H1	110.3
C7—C8—C11	121.5 (5)	F1^{i}—C1—H1	110.3
C9—C8—C11	118.1 (4)	C2—C1—H1	110.3
C7—C6—C5	121.2 (5)	N1—C2—C3	111.5 (5)
C7—C6—H6	119.4	N1—C2—C1	118.1 (4)
C5—C6—H6	119.4	C3—C2—C1	130.4 (4)
C9—C10—C5	120.8 (5)	C11—F3—F3^{i}	28.3 (7)
C2—N1—O1—C4	0.0	C7—C8—C11—F3	112.9 (8)
N1—O1—C4—C3	0.0	C9—C8—C11—F3	−67.1 (8)
N1—O1—C4—C5	180.0	C7—C8—C11—F2	0.0
C3—C4—C5—C10	0.0	C9—C8—C11—F2	180.0
O1—C4—C5—C10	180.0	C5—C6—C7—C8	0.0
C3—C4—C5—C6	180.0	C9—C8—C7—C6	0.0
O1—C4—C5—C6	0.0	C11—C8—C7—C6	180.0

O1—C4—C3—C2	0.0	O1—N1—C2—C3	0.0
C5—C4—C3—C2	180.0	O1—N1—C2—C1	180.0
C10—C9—C8—C7	0.0	C4—C3—C2—N1	0.0
C10—C9—C8—C11	180.0	C4—C3—C2—C1	180.0
C10—C5—C6—C7	0.0	F1—C1—C2—N1	−122.0 (3)
C4—C5—C6—C7	180.0	F1^{i}—C1—C2—N1	122.0 (3)
C8—C9—C10—C5	0.0	F1—C1—C2—C3	58.0 (3)
C6—C5—C10—C9	0.0	F1^{i}—C1—C2—C3	−58.0 (3)
C4—C5—C10—C9	180.0	F2—C11—F3—F3^{i}	−125.1 (18)
C7—C8—C11—F3^{i}	−112.9 (8)	C8—C11—F3—F3^{i}	124.8 (15)
C9—C8—C11—F3^{i}	67.1 (8)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{ii}	0.93	2.69	3.581 (7)	160
C3—H3···N1^{ii}	0.93	2.62	3.534 (6)	170
C1—H1···F2^{iii}	0.98	2.65	3.258 (6)	121

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