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(*Z*)-1-(2,4-Difluorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone oxime

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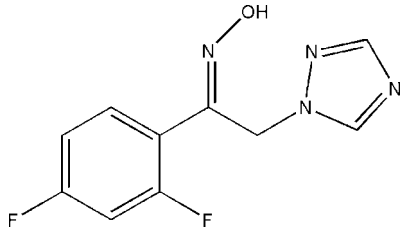
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.171; data-to-parameter ratio = 12.9.

In the title compound, $C_{10}H_8F_2N_4O$, the dihedral angle between the rings is 65.4 (1)°. In the crystal, intermolecular O—H···N and C—H···F hydrogen bonds link the molecules in a stacked arrangement along the *a* and *c* axes, respectively.

Related literature

For applications of related compounds, see: Foroumadi *et al.* (2003); Mixich & Thiele (1979); Wolfgang *et al.* (1981). For a related structure, see: Tao *et al.* (2007). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_8F_2N_4O$
M_r = 238.20
Monoclinic, $P2_1/n$
a = 8.6320 (17) Å
b = 12.433 (3) Å
c = 10.417 (2) Å
β = 104.85 (3)°
V = 1080.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.964, T_{max} = 0.988
3127 measured reflections
1987 independent reflections
1217 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.171
S = 1.01
1987 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N4^{i}	0.82	1.94	2.764 (3)	176
C10—H10···F1^{ii}	0.93	2.47	3.289 (4)	148

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2136).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Foroumadi, A., Soltani, F. & Asadipour, A. (2003). *Boll. Chim. Farm.* **142**, 130–134.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Mixich, G. & Thiele, K. (1979). *Arzneim. Forsch.* **29**, 1510–1513.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D**65, 148-155.
Tao, X., Yuan, L., Zhang, X.-Q., Jing, C. & Wang, J.-T. (2007). *Acta Cryst.* E**63**, o1330–o1331.
Wolfgang, K., Karl, H. B., Helmut, T., Wilhelm, B. & Paul-Ernst, F. (1981). US Patent 4 264 772.

supplementary materials

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(*Z*)-1-(2,4-Difluorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone oxime

G. Yu, C. Li, T. Xiao, S. Li and X. Tian

Comment

The title compound, $C_{10}H_8O_1N_4F_2$, is the key intermediate in the synthesis of a new kind of antifungal drug (Tao *et al.*, 2007; Foroumadi *et al.*, 2003; Wolfgang *et al.*, 1981) and exhibits a chemical structure similar to oxiconazole (Mixich & Thiele, 1979). We designed and synthesized the title compound, and we herein report its crystal structure (Fig. 1).

The bond lengths are within normal ranges (Allen *et al.*, 1987). The dihedral angle between rings A (N2-N4/C9/C10) and B (C1-C6) is 65.4 (1) °. In the crystal structure, intermolecular intermolecular O–H···N and C-H···F hydrogen bonds (Table 2) link the molecules in a stacked arrangement along along the *a* and *c* axes, respectively (Fig. 2).

Experimental

Hydroxylammonium chloride (3 g, 43.2 mmol) dissolved in ethanol (20 ml) was dropwise added to a solution of 1-(2,4-difluorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone (5 g,22.4 mmol) in ethanol (50 ml) which contained CH_3COONa (4 g, 48.8 mmol) under reflux conditions for 4 h. The mixture was placed in ice-water (100 ml), and the crystalline product was isolated by filtration, washed with water (100 ml). The crystals were obtained by dissolving the product in ethanol (20 ml) and evaporating acetone slowly at room temperature for about 7 d.

Refinement

The H atom of the hydroxy group was located in a Fourier difference map but was constrained to ride on the parent atom with O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O). The other H atoms were positioned geometrically with C—H = 0.93 Å (aromatic) and 0.97 Å (methylene) and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

Figures

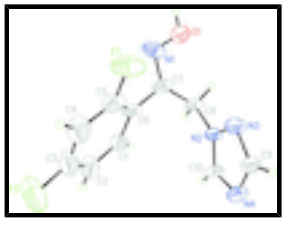

Fig. 1. The molecular structure of the title compound showing displacement ellipsoids drawn at the 50% probability level.

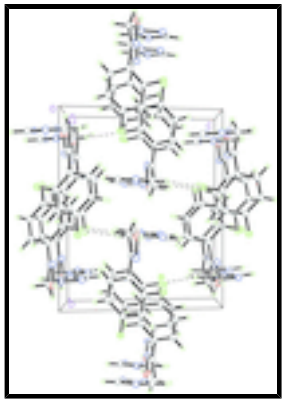

Fig. 2. A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

(*Z*)-1-(2,4-Difluorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone oxime

Crystal data

$C_{10}H_8F_2N_4O$	$F(000) = 488$
$M_r = 238.20$	$D_x = 1.464$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 400 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.6320$ (17) Å	Cell parameters from 25 reflections
$b = 12.433$ (3) Å	$\theta = 9$–$13°$
$c = 10.417$ (2) Å	$\mu = 0.12$ mm^{-1}
$\beta = 104.85$ (3)°	$T = 293$ K
$V = 1080.6$ (4) Å^3	Black, white
$Z = 4$	0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1217 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.042$
graphite	$\theta_{max} = 25.4°$, $\theta_{min} = 2.6°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 10$
Absorption correction: ψ scan (North *et al.*, 1968)	$k = -5 \rightarrow 14$
$T_{min} = 0.964$, $T_{max} = 0.988$	$l = -12 \rightarrow 12$
3127 measured reflections	3 standard reflections every 200 reflections
1987 independent reflections	intensity decay: 1%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H-atom parameters constrained
$wR(F^2) = 0.171$	$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
1987 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.034 (5)

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}
O1	−0.0689 (3)	0.1304 (2)	0.0266 (2)	0.0885 (7)
H1A	−0.1670	0.1325	0.0102	0.133*
F1	0.0628 (2)	0.39466 (19)	−0.12253 (18)	0.1082 (8)
N1	−0.0109 (3)	0.2303 (3)	0.0363 (2)	0.0787 (8)
C1	0.3526 (3)	0.3487 (3)	0.1896 (3)	0.0689 (8)
H1	0.3862	0.2944	0.2520	0.083*
N2	0.3725 (2)	0.13025 (17)	0.0097 (2)	0.0535 (6)
C2	0.4296 (4)	0.4452 (3)	0.2083 (3)	0.0880 (11)
H2	0.5142	0.4572	0.2826	0.106*
F2	0.4618 (4)	0.61943 (18)	0.1316 (3)	0.1508 (12)
N3	0.3361 (2)	0.1384 (2)	−0.1226 (2)	0.0654 (7)
C3	0.3801 (5)	0.5240 (3)	0.1156 (4)	0.0917 (10)
N4	0.6005 (2)	0.1436 (2)	−0.0387 (2)	0.0679 (7)
C4	0.2564 (4)	0.5106 (3)	0.0065 (3)	0.0944 (11)
H4	0.2229	0.5659	−0.0545	0.113*
C5	0.1822 (3)	0.4114 (3)	−0.0101 (3)	0.0707 (9)
C6	0.2250 (3)	0.3288 (2)	0.0798 (2)	0.0538 (7)
C7	0.1461 (3)	0.2225 (3)	0.0625 (2)	0.0619 (8)
C8	0.2467 (3)	0.1224 (2)	0.0803 (3)	0.0605 (8)
H8A	0.2951	0.1110	0.1741	0.073*
H8B	0.1789	0.0609	0.0471	0.073*
C9	0.4781 (3)	0.1454 (2)	−0.1470 (3)	0.0663 (8)
H9	0.4918	0.1511	−0.2324	0.080*
C10	0.5286 (3)	0.1362 (2)	0.0578 (3)	0.0597 (7)
H10	0.5810	0.1353	0.1477	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0712 (14)	0.0982 (19)	0.0951 (17)	0.0097 (14)	0.0198 (12)	−0.0037 (14)
F1	0.0744 (12)	0.160 (2)	0.0717 (12)	−0.0057 (12)	−0.0146 (10)	0.0297 (11)
N1	0.0610 (14)	0.115 (3)	0.0608 (15)	−0.0278 (15)	0.0177 (11)	−0.0027 (15)
C1	0.0595 (16)	0.078 (2)	0.0599 (16)	0.0107 (16)	−0.0024 (13)	−0.0075 (15)
N2	0.0337 (10)	0.0662 (15)	0.0611 (13)	−0.0045 (10)	0.0133 (9)	0.0006 (11)
C2	0.095 (2)	0.087 (3)	0.0639 (19)	0.012 (2)	−0.0120 (18)	−0.0177 (19)
F2	0.183 (3)	0.0744 (16)	0.161 (2)	−0.0211 (16)	−0.017 (2)	−0.0079 (15)
N3	0.0441 (11)	0.093 (2)	0.0577 (13)	−0.0116 (12)	0.0107 (10)	−0.0005 (12)
C3	0.106 (3)	0.067 (2)	0.091 (2)	0.002 (2)	0.004 (2)	−0.014 (2)
N4	0.0449 (12)	0.0795 (18)	0.0822 (16)	−0.0011 (11)	0.0216 (12)	−0.0008 (13)
C4	0.108 (3)	0.078 (3)	0.085 (2)	0.016 (2)	0.002 (2)	0.014 (2)
C5	0.0594 (16)	0.100 (3)	0.0456 (15)	0.0192 (18)	0.0007 (13)	0.0074 (16)
C6	0.0283 (11)	0.084 (2)	0.0503 (14)	0.0060 (12)	0.0120 (10)	−0.0023 (14)
C7	0.0463 (13)	0.096 (2)	0.0464 (14)	0.0039 (15)	0.0175 (11)	0.0027 (14)
C8	0.0363 (12)	0.080 (2)	0.0674 (17)	−0.0076 (13)	0.0175 (12)	0.0078 (15)
C9	0.0470 (14)	0.089 (2)	0.0659 (17)	−0.0109 (15)	0.0205 (13)	−0.0046 (16)
C10	0.0297 (11)	0.075 (2)	0.0724 (17)	0.0046 (12)	0.0091 (11)	0.0088 (14)

Geometric parameters (Å, °)

O1—N1	1.334 (3)	N3—C9	1.317 (3)
O1—H1A	0.8200	C3—C4	1.356 (5)
F1—C5	1.364 (3)	N4—C10	1.313 (3)
N1—C7	1.315 (3)	N4—C9	1.333 (3)
C1—C2	1.362 (4)	C4—C5	1.380 (5)
C1—C6	1.392 (3)	C4—H4	0.9300
C1—H1	0.9300	C5—C6	1.375 (4)
N2—C10	1.314 (3)	C6—C7	1.477 (4)
N2—N3	1.337 (3)	C7—C8	1.502 (4)
N2—C8	1.463 (3)	C8—H8A	0.9700
C2—C3	1.365 (5)	C8—H8B	0.9700
C2—H2	0.9300	C9—H9	0.9300
F2—C3	1.368 (4)	C10—H10	0.9300
N1—O1—H1A	109.5	F1—C5—C4	117.9 (3)
C7—N1—O1	107.1 (3)	C6—C5—C4	123.2 (3)
C2—C1—C6	121.9 (3)	C5—C6—C1	116.4 (3)
C2—C1—H1	119.0	C5—C6—C7	123.4 (2)
C6—C1—H1	119.0	C1—C6—C7	120.1 (3)
C10—N2—N3	109.6 (2)	N1—C7—C6	112.2 (3)
C10—N2—C8	129.3 (2)	N1—C7—C8	128.2 (3)
N3—N2—C8	120.97 (18)	C6—C7—C8	119.5 (2)
C1—C2—C3	118.6 (3)	N2—C8—C7	111.3 (2)
C1—C2—H2	120.7	N2—C8—H8A	109.4
C3—C2—H2	120.7	C7—C8—H8A	109.4

C9—N3—N2	102.7 (2)	N2—C8—H8B	109.4
C4—C3—C2	122.8 (4)	C7—C8—H8B	109.4
C4—C3—F2	118.6 (4)	H8A—C8—H8B	108.0
C2—C3—F2	118.6 (3)	N3—C9—N4	114.2 (2)
C10—N4—C9	102.8 (2)	N3—C9—H9	122.9
C3—C4—C5	117.1 (3)	N4—C9—H9	122.9
C3—C4—H4	121.5	N4—C10—N2	110.6 (2)
C5—C4—H4	121.5	N4—C10—H10	124.7
F1—C5—C6	118.8 (3)	N2—C10—H10	124.7
C6—C1—C2—C3	−0.5 (5)	O1—N1—C7—C6	−178.2 (2)
C10—N2—N3—C9	2.1 (3)	O1—N1—C7—C8	−0.5 (4)
C8—N2—N3—C9	179.0 (2)	C5—C6—C7—N1	−50.9 (3)
C1—C2—C3—C4	0.7 (6)	C1—C6—C7—N1	130.8 (3)
C1—C2—C3—F2	−177.3 (3)	C5—C6—C7—C8	131.1 (3)
C2—C3—C4—C5	−1.5 (6)	C1—C6—C7—C8	−47.1 (3)
F2—C3—C4—C5	176.6 (3)	C10—N2—C8—C7	112.4 (3)
C3—C4—C5—F1	−176.8 (3)	N3—N2—C8—C7	−63.8 (3)
C3—C4—C5—C6	2.0 (5)	N1—C7—C8—N2	135.8 (3)
F1—C5—C6—C1	177.1 (2)	C6—C7—C8—N2	−46.7 (3)
C4—C5—C6—C1	−1.7 (4)	N2—N3—C9—N4	−0.9 (3)
F1—C5—C6—C7	−1.2 (4)	C10—N4—C9—N3	−0.7 (4)
C4—C5—C6—C7	180.0 (3)	C9—N4—C10—N2	2.0 (3)
C2—C1—C6—C5	0.9 (4)	N3—N2—C10—N4	−2.7 (3)
C2—C1—C6—C7	179.3 (3)	C8—N2—C10—N4	−179.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···N4^{i}	0.82	1.94	2.764 (3)	176
C10—H10···F1^{ii}	0.93	2.47	3.289 (4)	148

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

Fig. 1

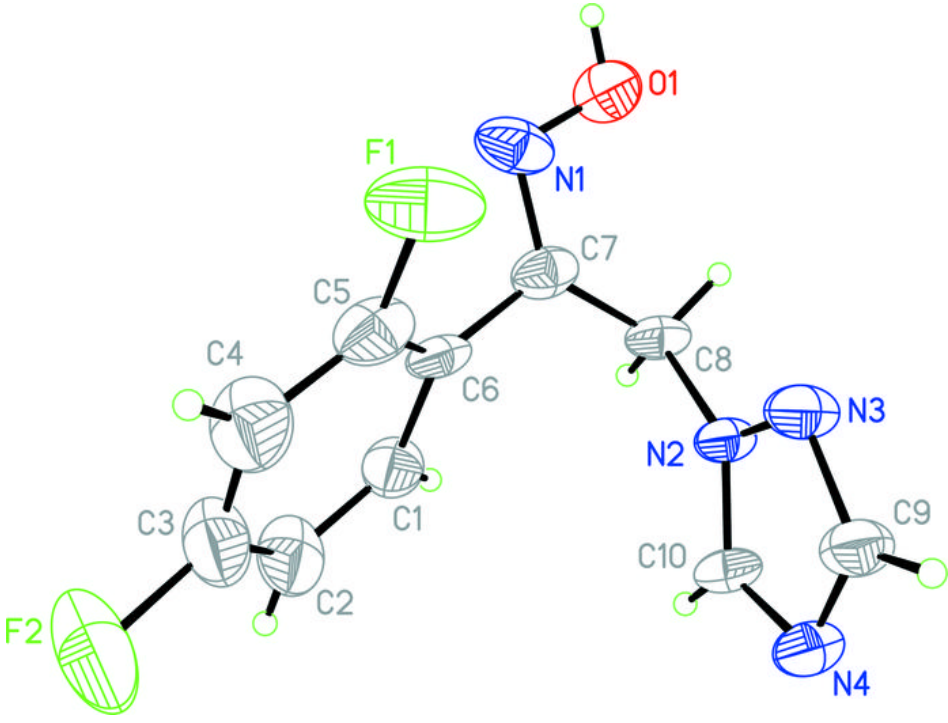

Fig. 2