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Ethyl 1-(2,6-difluorobenzyl)-1*H*-1,2,3-triazole-4-carboxylate

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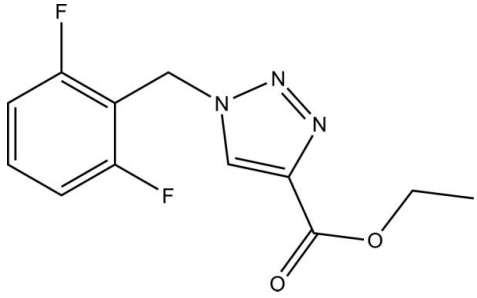
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.055; wR factor = 0.163; data-to-parameter ratio = 13.3.

In the title compound, $C_{12}H_{11}F_2N_3O_2$, the dihedral angle between the triazole and phenyl rings is 73.74 (9)°. In the crystal, molecules are linked into chains along [010] *via* weak C—H···O and C—H···N hydrogen bonds.

Related literature

The title compound is an intermediate in the synthesis of rufinamide, a new anti-epilepsy drug (Herranz, 2008). For synthetic procedures, see: Abu-Orabi *et al.* (1989); Wang & Xie (2004). For a related structure, see: Xiao *et al.* (2008).



Experimental

Crystal data

$C_{12}H_{11}F_2N_3O_2$
M_r = 267.24
Monoclinic, $P2_1/c$
a = 9.4540 (19) Å
b = 10.963 (2) Å
c = 12.167 (2) Å
β = 93.21 (3)°
V = 1259.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.965, T_{max} = 0.977
3270 measured reflections
2316 independent reflections
1629 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.163
S = 1.03
2316 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 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
C7—H7B···O1^{i}	0.97	2.47	3.415 (3)	166
C8—H8···N3^{i}	0.93	2.61	3.536 (3)	172

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

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2364).

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supporting information

Acta Cryst. (2011). E**67**, o127 [https://doi.org/10.1107/S1600536810050014]

Ethyl 1-(2,6-difluorobenzyl)-1*H*-1,2,3-triazole-4-carboxylate

Jing Jia and Dingqiang Lu

S1. Comment

Epilepsia has been a common disease for a long time and has been on an increase year after year. Rufinamide is a new drug to cure epilepsia; it is a triazole derivative (Herranz *et al.*, 2008). We report herein the crystal structure of the title compound which is a key intermediate in the synthesis of rufinamide. In the title compound (Fig. 1), the planes of the triazole and phenyl rings are not coplanar [dihedral angle 73.74 (9)°]. The discrete molecules are linked through weak C—H···O and C—H···N hydrogen bonds, forming one-dimensional chains along [010] direction (Fig. 2 and Tab. 1). In the structure of the title compound, the bond lengths and angles agree with the corresponding values reported for a related compound (Xiao *et al.*, 2008).

S2. Experimental

The title compound, was prepared by following procedures reported earlier (Wang *et al.*, 2004; Abu-Orabi *et al.*, 1989). To a solution of 2-(azidomethyl)-1,3-difluorobenzene (1.69 g, 10 mmol) in etanol (50 mL), ethyl propiolate (0.98 g, 10 mmol) was added and the mixture was heated under reflux for 10 h. After removing the solvent under reduced pressure the residue was dissolved and the title compound recrystallized from petroleum ether-methanol mixture (15:2), to provide crystals suitable for X-ray diffraction (yield 2.31 g, 86.3%).

S3. Refinement

H atoms were palced in geometrically calculated position and were refined using a riding model, with C—H = 0.93, 0.96 and 0.97 Å, for aryl, methyl and methylene type H-atoms, respectively, and U_{iso}(H) = 1.5 and 1.2 U_{eq}(C) for methyl and nonmethyl H-atoms, respectively.

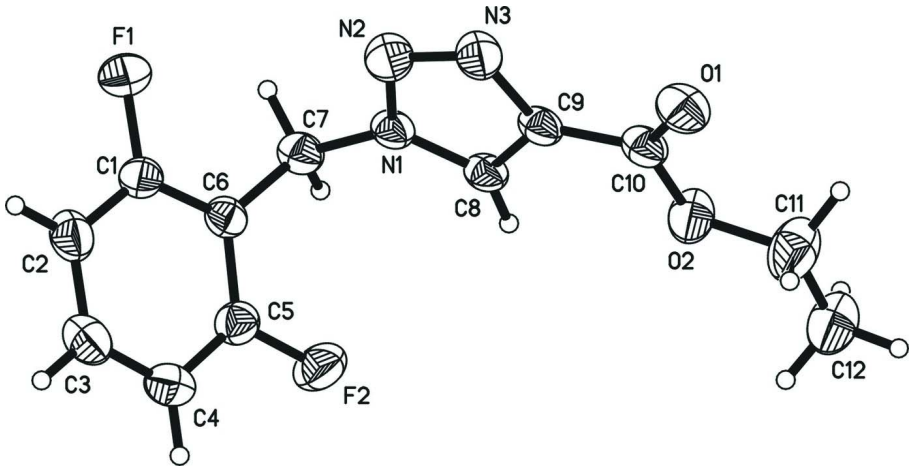

Figure 1

ORTEP view of the title compound. The dispalcement ellipsoids are drawn at 30% probability level.

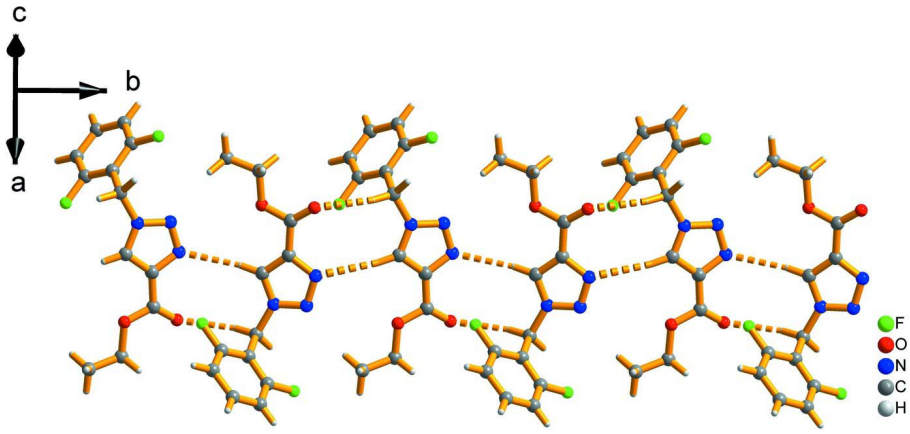

Figure 2

A one-dimensional chain of the title compound.

Ethyl 1-(2,6-difluorobenzyl)-1*H*-1,2,3-triazole-4-carboxylate

Crystal data

$C_{12}H_{11}F_2N_3O_2$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.4540\ (19)$ Å
$b = 10.963\ (2)$ Å
$c = 12.167\ (2)$ Å
$\beta = 93.21\ (3)°$
$V = 1259.1\ (4)$ Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.410$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–13°$
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans

Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.965, T_{max} = 0.977
3270 measured reflections
2316 independent reflections
1629 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

θ_{max} = 25.4°, θ_{min} = 2.2°
h = 0→11
k = −3→13
l = −14→14
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.163
S = 1.03
2316 reflections
174 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
$w = 1/[\sigma^2(F_o^2) + (0.0873P)^2 + 0.337P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.029 (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}
F1	0.94152 (18)	0.27880 (14)	0.02828 (16)	0.0890 (6)
F2	0.78127 (17)	0.61613 (15)	0.22000 (14)	0.0829 (6)
O1	0.4165 (2)	0.20776 (17)	0.46337 (16)	0.0744 (6)
O2	0.3594 (2)	0.39959 (17)	0.41799 (17)	0.0776 (6)
N1	0.62925 (19)	0.35347 (16)	0.16945 (17)	0.0537 (5)
N2	0.6558 (3)	0.2343 (2)	0.1898 (2)	0.0751 (7)
N3	0.5829 (2)	0.20178 (19)	0.2728 (2)	0.0709 (7)
C1	0.9678 (3)	0.3771 (2)	0.0933 (2)	0.0618 (7)
C2	1.1060 (3)	0.4031 (3)	0.1262 (3)	0.0733 (8)
H2	1.1796	0.3538	0.1049	0.088*
C3	1.1325 (3)	0.5034 (3)	0.1910 (3)	0.0751 (8)
H3	1.2252	0.5220	0.2146	0.090*
C4	1.0243 (3)	0.5767 (3)	0.2217 (2)	0.0687 (7)
H4	1.0426	0.6459	0.2644	0.082*
C5	0.8887 (3)	0.5456 (2)	0.1878 (2)	0.0583 (6)
C6	0.8534 (2)	0.4459 (2)	0.12140 (19)	0.0522 (6)
C7	0.7032 (3)	0.4176 (2)	0.0843 (2)	0.0593 (7)

H7B	0.6538	0.4930	0.0658	0.071*
H7A	0.7023	0.3676	0.0185	0.071*
C8	0.5379 (2)	0.3966 (2)	0.2397 (2)	0.0524 (6)
H8	0.5015	0.4753	0.2428	0.063*
C9	0.5093 (2)	0.3001 (2)	0.3059 (2)	0.0538 (6)
C10	0.4248 (3)	0.2941 (2)	0.4032 (2)	0.0577 (6)
C11	0.2781 (4)	0.4100 (3)	0.5169 (3)	0.1053 (13)
H11A	0.2056	0.3473	0.5162	0.126*
H11B	0.3404	0.3988	0.5822	0.126*
C12	0.2137 (4)	0.5286 (3)	0.5186 (3)	0.0955 (11)
H12A	0.1482	0.5373	0.4559	0.143*
H12C	0.2857	0.5901	0.5163	0.143*
H12B	0.1641	0.5376	0.5849	0.143*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0929 (12)	0.0560 (10)	0.1202 (14)	−0.0053 (8)	0.0250 (10)	−0.0258 (9)
F2	0.0797 (11)	0.0754 (11)	0.0936 (12)	0.0159 (9)	0.0056 (9)	−0.0255 (9)
O1	0.0857 (13)	0.0520 (11)	0.0853 (13)	−0.0042 (9)	0.0038 (10)	0.0175 (10)
O2	0.0897 (14)	0.0537 (11)	0.0929 (14)	0.0125 (10)	0.0351 (11)	0.0150 (10)
N1	0.0500 (11)	0.0360 (10)	0.0751 (13)	−0.0006 (8)	0.0039 (9)	−0.0005 (9)
N2	0.0744 (15)	0.0421 (12)	0.1107 (18)	0.0101 (11)	0.0221 (14)	0.0037 (12)
N3	0.0678 (14)	0.0404 (12)	0.1064 (18)	0.0058 (10)	0.0220 (13)	0.0086 (12)
C1	0.0709 (17)	0.0402 (12)	0.0759 (16)	−0.0045 (12)	0.0198 (13)	−0.0027 (11)
C2	0.0581 (16)	0.0583 (17)	0.105 (2)	0.0027 (13)	0.0198 (15)	0.0076 (15)
C3	0.0602 (16)	0.0719 (19)	0.093 (2)	−0.0104 (15)	0.0015 (15)	0.0048 (16)
C4	0.0718 (17)	0.0629 (16)	0.0706 (17)	−0.0111 (14)	−0.0030 (13)	−0.0067 (13)
C5	0.0621 (15)	0.0524 (14)	0.0607 (14)	0.0023 (12)	0.0061 (12)	−0.0008 (11)
C6	0.0562 (13)	0.0444 (12)	0.0563 (13)	−0.0040 (11)	0.0066 (10)	0.0055 (10)
C7	0.0614 (15)	0.0506 (14)	0.0658 (15)	−0.0042 (11)	0.0018 (12)	−0.0001 (12)
C8	0.0439 (12)	0.0359 (12)	0.0769 (16)	0.0018 (9)	−0.0008 (11)	−0.0022 (11)
C9	0.0460 (12)	0.0375 (12)	0.0775 (16)	−0.0028 (10)	−0.0004 (11)	0.0011 (11)
C10	0.0534 (13)	0.0434 (13)	0.0759 (16)	−0.0046 (11)	−0.0003 (12)	0.0039 (12)
C11	0.141 (3)	0.077 (2)	0.103 (3)	0.020 (2)	0.058 (2)	0.0216 (19)
C12	0.105 (2)	0.095 (3)	0.088 (2)	0.018 (2)	0.0283 (18)	0.0080 (19)

Geometric parameters (Å, º)

F1—C1	1.352 (3)	C4—C5	1.368 (4)
F2—C5	1.352 (3)	C4—H4	0.9300
O1—C10	1.202 (3)	C5—C6	1.390 (3)
O2—C10	1.329 (3)	C6—C7	1.498 (3)
O2—C11	1.468 (4)	C7—H7B	0.9700
N1—C8	1.335 (3)	C7—H7A	0.9700
N1—N2	1.351 (3)	C8—C9	1.366 (3)
N1—C7	1.462 (3)	C8—H8	0.9300
N2—N3	1.304 (3)	C9—C10	1.466 (4)

N3—C9	1.356 (3)	C11—C12	1.437 (4)
C1—C2	1.374 (4)	C11—H11A	0.9700
C1—C6	1.377 (3)	C11—H11B	0.9700
C2—C3	1.368 (4)	C12—H12A	0.9600
C2—H2	0.9300	C12—H12C	0.9600
C3—C4	1.369 (4)	C12—H12B	0.9600
C3—H3	0.9300		

C10—O2—C11	116.6 (2)	C6—C7—H7B	109.3
C8—N1—N2	110.2 (2)	N1—C7—H7A	109.3
C8—N1—C7	129.4 (2)	C6—C7—H7A	109.3
N2—N1—C7	120.2 (2)	H7B—C7—H7A	108.0
N3—N2—N1	107.8 (2)	N1—C8—C9	105.03 (19)
N2—N3—C9	108.3 (2)	N1—C8—H8	127.5
F1—C1—C2	118.4 (2)	C9—C8—H8	127.5
F1—C1—C6	117.4 (2)	N3—C9—C8	108.6 (2)
C2—C1—C6	124.2 (2)	N3—C9—C10	121.0 (2)
C3—C2—C1	118.3 (3)	C8—C9—C10	130.2 (2)
C3—C2—H2	120.8	O1—C10—O2	123.7 (2)
C1—C2—H2	120.8	O1—C10—C9	125.8 (2)
C2—C3—C4	120.9 (3)	O2—C10—C9	110.4 (2)
C2—C3—H3	119.5	C12—C11—O2	108.9 (3)
C4—C3—H3	119.5	C12—C11—H11A	109.9
C5—C4—C3	118.2 (3)	O2—C11—H11A	109.9
C5—C4—H4	120.9	C12—C11—H11B	109.9
C3—C4—H4	120.9	O2—C11—H11B	109.9
F2—C5—C4	118.5 (2)	H11A—C11—H11B	108.3
F2—C5—C6	117.3 (2)	C11—C12—H12A	109.5
C4—C5—C6	124.2 (2)	C11—C12—H12C	109.5
C1—C6—C5	114.1 (2)	H12A—C12—H12C	109.5
C1—C6—C7	123.8 (2)	C11—C12—H12B	109.5
C5—C6—C7	122.0 (2)	H12A—C12—H12B	109.5
N1—C7—C6	111.7 (2)	H12C—C12—H12B	109.5
N1—C7—H7B	109.3		

C8—N1—N2—N3	−0.5 (3)	C8—N1—C7—C6	100.9 (3)
C7—N1—N2—N3	176.4 (2)	N2—N1—C7—C6	−75.2 (3)
N1—N2—N3—C9	0.0 (3)	C1—C6—C7—N1	99.0 (3)
F1—C1—C2—C3	179.3 (3)	C5—C6—C7—N1	−81.2 (3)
C6—C1—C2—C3	0.1 (4)	N2—N1—C8—C9	0.7 (3)
C1—C2—C3—C4	−0.6 (4)	C7—N1—C8—C9	−175.8 (2)
C2—C3—C4—C5	1.4 (4)	N2—N3—C9—C8	0.4 (3)
C3—C4—C5—F2	178.5 (3)	N2—N3—C9—C10	−175.6 (2)
C3—C4—C5—C6	−1.9 (4)	N1—C8—C9—N3	−0.7 (3)
F1—C1—C6—C5	−179.7 (2)	N1—C8—C9—C10	174.9 (2)
C2—C1—C6—C5	−0.5 (4)	C11—O2—C10—O1	2.7 (4)
F1—C1—C6—C7	0.1 (4)	C11—O2—C10—C9	−176.0 (3)
C2—C1—C6—C7	179.4 (2)	N3—C9—C10—O1	3.0 (4)

F2—C5—C6—C1	−179.0 (2)	C8—C9—C10—O1	−172.1 (3)
C4—C5—C6—C1	1.4 (4)	N3—C9—C10—O2	−178.3 (2)
F2—C5—C6—C7	1.2 (3)	C8—C9—C10—O2	6.6 (4)
C4—C5—C6—C7	−178.4 (2)	C10—O2—C11—C12	−179.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···F1	0.97	2.46	2.833 (3)	103
C7—H7*B*···O1^{i}	0.97	2.47	3.415 (3)	166
C8—H8···N3^{i}	0.93	2.61	3.536 (3)	172

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