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N-(2,3,4-Trifluorophenyl)pyrrolidine-1-carboxamide

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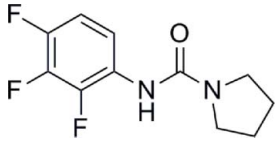
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.151; data-to-parameter ratio = 13.9.

In the title compound, $C_{11}H_{11}F_3N_2O$, a urea derivative, the best plane through the pyrrole ring makes a dihedral angle of 9.69 (13)° with the benzene ring. The amino H atom is shielded, so that it is not involved in any hydrogen-bonding interactions.

Related literature

For background to this class of compounds, see Zheng *et al.* (2010).



Experimental

Crystal data

$C_{11}H_{11}F_3N_2O$
M_r = 244.22
Monoclinic, $P2_1/n$
a = 6.0708 (4) Å
b = 24.2124 (15) Å
c = 7.4232 (6) Å
β = 100.508 (7)°
V = 1072.83 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.35 × 0.35 × 0.25 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)
T_{min} = 0.964, T_{max} = 1.000
4465 measured reflections
2190 independent reflections
1374 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.151
S = 1.04
2190 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5716).

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supporting information

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N-(2,3,4-Trifluorophenyl)pyrrolidine-1-carboxamide

Shuchen Pei, Jie Li, Boyi Qu, Li Hai and Yong Wu

S1. Comment

The compound *N*-(2,3,4-trifluorophenyl)pyrrolidine-1-carboxamide is one of urea derivatives. It has been established that urea derivatives have got a significant placein modern medicinal chemistry. Urea derivatives have been reported in the literature as anticancer agent, anticonvulsant, CXCR3 antagonist,antibacterial and so on. Our interests in synthesizing urea derivatives prompted us to develop an efficient methodology for synthesizing *N*-(2,3,4-trifluorophenyl)-pyrrolidine-1-carboxamide. In our synthetic work, we obtained the title compound, and its crystal structure is reported here. The three fluorine atoms of the attached benzene ring are close to being coplanar with the ring, whereas the pyrrole ring is not coplanar with the benzene ring.

S2. Experimental

The title compound was obtained as a derivative of urea. To a solution of triphosgene (350 mg, 1.19 mmol) and triethyl-amine (680 mg, 6.80 mmol) in anhydrous acetonitrile (5 ml) at ice bath, the solution of 2,3,4-trifluoroaniline (500 mg, 3.40 mmol) and triethylamine (680 mg, 6.80 mmol) in anhydrous acetonitrile (5 ml) were added dropwise.The mixture was stirred for 1 h. And then the solition of tetrahydropyrrole (240 mg,3.40 mmol) and triethylamine (680 mg, 6.80 mmol) in anhydrous acetonitrile (5 ml) were added dropwise. The reaction mixture was then removed from the cooling bath and stirred at room temperature overnight. On completion of the reaction, the mixture was poured into water. The aqueous layer was extracted with ethyl acetate and the organic layer was separated. The organic layers were washed with brine and dried over sodium sulfate, filtered, and concentrated *in vacuo*. The purification of the residue by silica gel column chromatography eluting with EtOAc-petroleum ether (1:10) yielded the white solid 660 mg (yield 86.7%) of *N*-(2,3,4-trifluorophenyl)pyrrolidine-1-carboxamide. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation in ethyl acetate at room temperature.

S3. Refinement

H atoms bonded to C were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The amino H atom was freely refined.

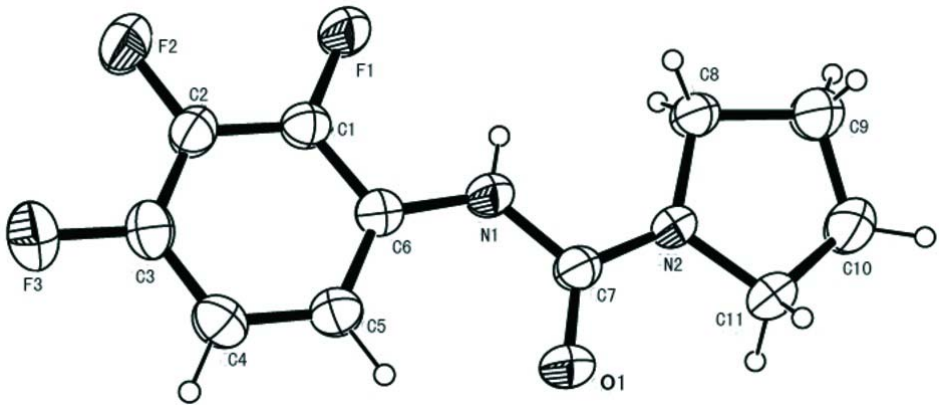

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Figure 2

A packing diagram for the title compound.

N-(2,3,4-Trifluorophenyl)pyrrolidine-1-carboxamide

Crystal data

$C_{11}H_{11}F_3N_2O$
$M_r = 244.22$
Monoclinic, $P2_1/n$
$a = 6.0708$ (4) Å
$b = 24.2124$ (15) Å
$c = 7.4232$ (6) Å
$\beta = 100.508$ (7)°
$V = 1072.83$ (13) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.512$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 1445 reflections
$\theta = 2.9$–$28.9°$
$\mu = 0.13$ mm^{-1}
$T = 293$ K
Block, colorless
0.35 × 0.35 × 0.25 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0874 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2007)
T_{min} = 0.964, T_{max} = 1.000
4465 measured reflections
2190 independent reflections
1374 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 26.4°, θ_{min} = 2.9°
h = −7→6
k = −30→27
l = −8→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.151
S = 1.04
2190 reflections
158 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
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.3084P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
F1	0.1373 (2)	−0.04074 (6)	0.1339 (2)	0.0855 (6)
F2	0.2120 (3)	−0.14684 (7)	0.2355 (3)	0.0987 (7)
F3	0.6185 (3)	−0.17950 (6)	0.4120 (2)	0.0908 (6)
O1	0.7849 (3)	0.08144 (8)	0.2954 (3)	0.0827 (6)
N1	0.4504 (4)	0.03783 (9)	0.2033 (3)	0.0586 (6)
H1	0.325 (4)	0.0407 (10)	0.159 (4)	0.061 (9)*
N2	0.4818 (3)	0.13161 (8)	0.1745 (3)	0.0577 (6)
C1	0.3428 (4)	−0.05587 (11)	0.2237 (3)	0.0577 (6)
C2	0.3786 (5)	−0.10989 (11)	0.2741 (4)	0.0626 (7)
C3	0.5855 (5)	−0.12546 (11)	0.3645 (4)	0.0649 (7)
C4	0.7542 (5)	−0.08815 (11)	0.4034 (4)	0.0676 (7)
H4	0.8950	−0.0992	0.4640	0.081*
C5	0.7159 (4)	−0.03335 (11)	0.3522 (4)	0.0645 (7)
H5	0.8315	−0.0078	0.3799	0.077*
C6	0.5075 (4)	−0.01615 (10)	0.2603 (3)	0.0521 (6)

C7	0.5845 (4)	0.08395 (10)	0.2289 (3)	0.0558 (6)
C8	0.2441 (4)	0.13970 (10)	0.1006 (4)	0.0637 (7)
H8A	0.1513	0.1238	0.1807	0.076*
H8B	0.2032	0.1234	−0.0203	0.076*
C9	0.2219 (5)	0.20173 (12)	0.0924 (5)	0.0955 (11)
H9A	0.1181	0.2128	−0.0170	0.115*
H9B	0.1664	0.2153	0.1987	0.115*
C10	0.4431 (5)	0.22379 (13)	0.0893 (5)	0.0987 (11)
H10A	0.4614	0.2594	0.1501	0.118*
H10B	0.4652	0.2284	−0.0359	0.118*
C11	0.6072 (5)	0.18321 (11)	0.1877 (4)	0.0744 (8)
H11A	0.7371	0.1800	0.1293	0.089*
H11B	0.6562	0.1939	0.3146	0.089*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0581 (10)	0.0683 (10)	0.1208 (14)	−0.0094 (8)	−0.0083 (9)	0.0058 (9)
F2	0.0865 (13)	0.0638 (10)	0.1378 (17)	−0.0188 (9)	−0.0004 (11)	0.0062 (10)
F3	0.1087 (15)	0.0639 (11)	0.0979 (14)	0.0123 (9)	0.0135 (10)	0.0105 (9)
O1	0.0462 (11)	0.0746 (13)	0.1207 (18)	−0.0060 (9)	−0.0020 (10)	0.0012 (11)
N1	0.0477 (13)	0.0583 (14)	0.0662 (15)	−0.0060 (11)	0.0010 (11)	−0.0014 (10)
N2	0.0468 (12)	0.0521 (12)	0.0723 (14)	−0.0087 (10)	0.0060 (10)	−0.0041 (10)
C1	0.0524 (15)	0.0623 (16)	0.0570 (15)	−0.0028 (13)	0.0064 (11)	−0.0034 (12)
C2	0.0659 (17)	0.0541 (16)	0.0686 (17)	−0.0082 (14)	0.0147 (13)	−0.0042 (13)
C3	0.082 (2)	0.0562 (16)	0.0581 (16)	0.0061 (15)	0.0158 (14)	−0.0003 (12)
C4	0.0633 (17)	0.0739 (18)	0.0628 (17)	0.0100 (15)	0.0039 (13)	0.0012 (14)
C5	0.0563 (16)	0.0664 (17)	0.0675 (17)	−0.0033 (14)	0.0029 (12)	−0.0041 (13)
C6	0.0538 (15)	0.0576 (15)	0.0451 (14)	−0.0014 (12)	0.0097 (11)	−0.0049 (11)
C7	0.0506 (15)	0.0584 (15)	0.0588 (15)	−0.0065 (13)	0.0112 (11)	−0.0061 (12)
C8	0.0516 (15)	0.0632 (16)	0.0736 (18)	−0.0067 (13)	0.0045 (12)	0.0055 (13)
C9	0.069 (2)	0.069 (2)	0.144 (3)	−0.0005 (17)	0.007 (2)	0.0207 (19)
C10	0.082 (2)	0.0619 (19)	0.146 (3)	−0.0105 (18)	0.003 (2)	0.0102 (19)
C11	0.0585 (17)	0.0621 (17)	0.101 (2)	−0.0144 (14)	0.0086 (15)	−0.0058 (15)

Geometric parameters (Å, º)

F1—C1	1.353 (3)	C4—C5	1.388 (4)
F2—C2	1.341 (3)	C5—H5	0.9300
F3—C3	1.360 (3)	C5—C6	1.387 (3)
O1—C7	1.228 (3)	C8—H8A	0.9700
N1—H1	0.77 (3)	C8—H8B	0.9700
N1—C6	1.398 (3)	C8—C9	1.508 (4)
N1—C7	1.375 (3)	C9—H9A	0.9700
N2—C7	1.338 (3)	C9—H9B	0.9700
N2—C8	1.461 (3)	C9—C10	1.449 (4)
N2—C11	1.457 (3)	C10—H10A	0.9700
C1—C2	1.367 (4)	C10—H10B	0.9700

C1—C6	1.378 (3)	C10—C11	1.492 (4)
C2—C3	1.365 (4)	C11—H11A	0.9700
C3—C4	1.357 (4)	C11—H11B	0.9700
C4—H4	0.9300		

C6—N1—H1	112.6 (19)	N2—C7—N1	115.3 (2)
C7—N1—H1	120 (2)	N2—C8—H8A	111.2
C7—N1—C6	127.6 (2)	N2—C8—H8B	111.2
C7—N2—C8	127.1 (2)	N2—C8—C9	102.9 (2)
C7—N2—C11	120.7 (2)	H8A—C8—H8B	109.1
C11—N2—C8	112.3 (2)	C9—C8—H8A	111.2
F1—C1—C2	118.7 (2)	C9—C8—H8B	111.2
F1—C1—C6	118.6 (2)	C8—C9—H9A	110.3
C2—C1—C6	122.7 (2)	C8—C9—H9B	110.3
F2—C2—C1	120.2 (2)	H9A—C9—H9B	108.6
F2—C2—C3	120.7 (2)	C10—C9—C8	106.9 (2)
C3—C2—C1	119.0 (2)	C10—C9—H9A	110.3
F3—C3—C2	118.2 (3)	C10—C9—H9B	110.3
C4—C3—F3	121.0 (3)	C9—C10—H10A	110.4
C4—C3—C2	120.8 (3)	C9—C10—H10B	110.4
C3—C4—H4	120.1	C9—C10—C11	106.7 (3)
C3—C4—C5	119.7 (3)	H10A—C10—H10B	108.6
C5—C4—H4	120.1	C11—C10—H10A	110.4
C4—C5—H5	119.5	C11—C10—H10B	110.4
C6—C5—C4	120.9 (3)	N2—C11—C10	103.7 (2)
C6—C5—H5	119.5	N2—C11—H11A	111.0
C1—C6—N1	117.5 (2)	N2—C11—H11B	111.0
C1—C6—C5	116.8 (2)	C10—C11—H11A	111.0
C5—C6—N1	125.7 (2)	C10—C11—H11B	111.0
O1—C7—N1	122.3 (2)	H11A—C11—H11B	109.0
O1—C7—N2	122.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···F1	0.77 (3)	2.27 (3)	2.672 (3)	113 (2)