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## Structure Reports

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**N-(2,3,4-Trifluorophenyl)morpholine-4-carboxamide**

Li Jie, Pei Shuchen, Hai Li and Wu Yong\*

Key Laboratory of Drug Targeting of the Education Ministry, West China School of Pharmacy, Sichuan University, Chengdu 610041, People's Republic of China  
Correspondence e-mail: wyong@scu.edu.cn

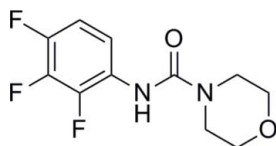
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.103; data-to-parameter ratio = 12.0.

In title molecule,  $\text{C}_{11}\text{H}_{11}\text{F}_3\text{N}_2\text{O}_2$ , the central  $-\text{N}-\text{C}(=\text{O})-\text{N}-$  unit is essentially planar [maximum deviation =  $0.013$  (2) Å] and forms a dihedral angle of  $57.33$  (9)° with the benzene ring. The morpholine ring is in a chair conformation. In the crystal, molecules are linked into chains along [001] by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds.

## Related literature

For background to urea derivatives as antibacterial and anti-fungal agents, see: Zheng *et al.* (2010).



## Experimental

## Crystal data

$\text{C}_{11}\text{H}_{11}\text{F}_3\text{N}_2\text{O}_2$   
 $M_r = 260.22$   
Monoclinic,  $P2_1/c$   
 $a = 7.8515$  (4) Å

$b = 17.8264$  (6) Å  
 $c = 8.6872$  (4) Å  
 $\beta = 109.790$  (5)°  
 $V = 1144.09$  (8) Å<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.14$  mm<sup>-1</sup>

$T = 293$  K  
 $0.40 \times 0.35 \times 0.30$  mm

## Data collection

Agilent Xcalibur Eos diffractometer  
Absorption correction: multi-scan  
(*CrysAlis PRO*; Agilent, 2011)  
 $T_{\min} = 0.994$ ,  $T_{\max} = 1.000$

4250 measured reflections  
2009 independent reflections  
1587 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.103$   
 $S = 1.07$   
2009 reflections  
167 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.15$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.23$  e Å<sup>-3</sup>

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O2}^i$	0.843 (18)	2.120 (19)	2.9306 (19)	161.2 (17)

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

Data collection: *CrysAlis PRO* (Agilent, 2011); 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) and *PLATON* (Spek, 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: LH5385).

## References

- Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.  
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.  
Zheng, Q. Z., Cheng, H. K., Zhang, X. M., Liu, K., Jiao, Q. C. & Zhu, H. L. (2010). *Eur. J. Med. Chem.* **45**, 3207–3212.

**supplementary materials**

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## *N*-(2,3,4-Trifluorophenyl)morpholine-4-carboxamide

L. Jie, P. Shuchen, H. Li and W. Yong

### Comment

The title compound (I) was made as part of our work on urea derivatives. Urea derivatives have been reported in the literature as antibacterial and antifungal agents (Zheng *et al.*, 2010). The crystal structure of (I) is reported herein here.

The molecular structure of the title compound is shown in Fig. 1. The central -N—C(=O)—N- unit is essentially planar (the maximum deviation from atoms N2/C7/O2/O1 is 0.013 Å for C7) and forms a dihedral angle of 57.33 (9)° with the benzene ring. The morpholine ring is in a chair conformation. In the crystal, molecules are linked into chains along [001] by N—H···O hydrogen bonds (Fig. 2).

### Experimental

To a solution of triphosgene (350 mg, 1.19 mmol) and triethylamine (680 mg, 6.80 mmol) in anhydrous acetonitrile (5 ml) at ice bath, a solution of 2,3,4-trifluoroaniline (500 mg, 3.40 mmol) and triethylamine (680 mg, 6.80 mmol) in anhydrous acetonitrile (5 ml) was added dropwise. The mixture was stirred for 1 h. Then, a solution of morpholine (300 mg, 3.40 mmol) and triethylamine (680 mg, 6.80 mmol) in anhydrous acetonitrile (5 ml) was 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 (1:8) yielded the white solid 670 mg (yield 76.2%) of *N*-(2,3,4-trifluorophenyl)morpholine-4-carboxamide. Colorless crystals suitable for X-ray analysis were obtained by slow evaporation in ethyl acetate at room temperature.

### Refinement

H atoms bonded to C atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The H atom bonded to N was refined independently with an isotropic displacement parameter.

### Figures

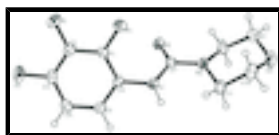


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

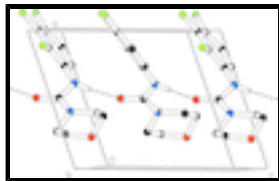


Fig. 2. Part of the crystal structure with hydrogen bonds shown as dashed lines.

## *N*-(2,3,4-Trifluorophenyl)morpholine-4-carboxamide

### Crystal data

$C_{11}H_{11}F_3N_2O_2$	$F(000) = 536$
$M_r = 260.22$	$D_x = 1.511 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.7107 \text{ \AA}$
$a = 7.8515 (4) \text{ \AA}$	Cell parameters from 1772 reflections
$b = 17.8264 (6) \text{ \AA}$	$\theta = 3.0\text{--}29.1^\circ$
$c = 8.6872 (4) \text{ \AA}$	$\mu = 0.14 \text{ mm}^{-1}$
$\beta = 109.790 (5)^\circ$	$T = 293 \text{ K}$
$V = 1144.09 (8) \text{ \AA}^3$	Block, colorless
$Z = 4$	$0.40 \times 0.35 \times 0.30 \text{ mm}$

### Data collection

Agilent Xcalibur Eos diffractometer	2009 independent reflections
Radiation source: Enhance (Mo) X-ray Source graphite	1587 reflections with $I > 2\sigma(I)$
Detector resolution: $16.0874 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.016$
$\omega$ scans	$\theta_{\text{max}} = 25.0^\circ$ , $\theta_{\text{min}} = 3.0^\circ$
Absorption correction: multi-scan ( <i>Crys.Alis PRO</i> ; Agilent, 2011)	$h = -7 \rightarrow 9$
$T_{\text{min}} = 0.994$ , $T_{\text{max}} = 1.000$	$k = -13 \rightarrow 21$
4250 measured reflections	$l = -10 \rightarrow 7$

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.103$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 0.1807P]$
2009 reflections	where $P = (F_o^2 + 2F_c^2)/3$
167 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.88764 (17)	0.67437 (6)	0.52547 (15)	0.0574 (4)
F2	1.12312 (17)	0.76456 (7)	0.45200 (16)	0.0681 (4)
F3	1.11974 (18)	0.91342 (7)	0.50200 (18)	0.0764 (4)
O1	0.2304 (2)	0.51651 (8)	0.66738 (18)	0.0617 (4)
O2	0.50780 (18)	0.68346 (7)	0.37155 (14)	0.0427 (4)
N1	0.6148 (2)	0.73140 (8)	0.63043 (18)	0.0384 (4)
N2	0.4106 (2)	0.63276 (8)	0.56564 (16)	0.0370 (4)
C1	0.8741 (3)	0.74877 (10)	0.5421 (2)	0.0380 (4)
C2	0.9976 (3)	0.79446 (11)	0.5078 (2)	0.0434 (5)
C3	0.9947 (3)	0.87006 (11)	0.5337 (2)	0.0469 (5)
C4	0.8722 (3)	0.90070 (11)	0.5949 (2)	0.0471 (5)
H4	0.8739	0.9519	0.6159	0.057*
C5	0.7451 (3)	0.85485 (10)	0.6255 (2)	0.0397 (5)
H5	0.6604	0.8757	0.6662	0.048*
C6	0.7420 (2)	0.77841 (9)	0.59651 (19)	0.0331 (4)
C7	0.5081 (2)	0.68272 (9)	0.5135 (2)	0.0328 (4)
C8	0.2658 (3)	0.59059 (10)	0.4469 (2)	0.0422 (5)
H8A	0.2909	0.5865	0.3452	0.051*
H8B	0.1523	0.6173	0.4244	0.051*
C9	0.2489 (3)	0.51341 (11)	0.5100 (3)	0.0546 (6)
H9A	0.1442	0.4885	0.4342	0.065*
H9B	0.3553	0.4842	0.5165	0.065*
C10	0.3837 (3)	0.55182 (12)	0.7792 (2)	0.0587 (6)
H10A	0.4916	0.5240	0.7841	0.070*
H10B	0.3735	0.5512	0.8874	0.070*
C11	0.4023 (3)	0.63175 (11)	0.7304 (2)	0.0484 (5)
H11A	0.2996	0.6610	0.7340	0.058*
H11B	0.5115	0.6538	0.8063	0.058*
H1	0.576 (3)	0.7459 (10)	0.705 (2)	0.039 (5)*

## supplementary materials

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### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
F1	0.0633 (8)	0.0337 (6)	0.0872 (9)	0.0021 (5)	0.0412 (7)	-0.0068 (6)
F2	0.0613 (8)	0.0643 (8)	0.1001 (10)	0.0016 (6)	0.0552 (8)	-0.0071 (7)
F3	0.0789 (10)	0.0583 (8)	0.1138 (11)	-0.0191 (7)	0.0613 (9)	0.0059 (7)
O1	0.0655 (10)	0.0587 (10)	0.0722 (10)	-0.0197 (8)	0.0380 (9)	0.0057 (8)
O2	0.0571 (9)	0.0439 (8)	0.0345 (7)	-0.0081 (6)	0.0253 (6)	-0.0018 (5)
N1	0.0493 (10)	0.0379 (9)	0.0361 (8)	-0.0088 (7)	0.0252 (8)	-0.0071 (7)
N2	0.0437 (9)	0.0384 (8)	0.0333 (8)	-0.0090 (7)	0.0188 (7)	0.0005 (6)
C1	0.0448 (11)	0.0291 (9)	0.0438 (10)	0.0012 (8)	0.0199 (9)	-0.0017 (8)
C2	0.0428 (11)	0.0460 (12)	0.0497 (11)	0.0019 (9)	0.0266 (9)	-0.0013 (9)
C3	0.0494 (12)	0.0427 (11)	0.0549 (12)	-0.0112 (10)	0.0259 (10)	0.0050 (9)
C4	0.0572 (13)	0.0308 (10)	0.0576 (12)	-0.0045 (9)	0.0252 (11)	-0.0012 (9)
C5	0.0449 (11)	0.0349 (10)	0.0435 (10)	0.0010 (9)	0.0206 (9)	-0.0045 (8)
C6	0.0398 (10)	0.0326 (9)	0.0294 (8)	-0.0031 (8)	0.0150 (8)	-0.0008 (7)
C7	0.0379 (10)	0.0300 (9)	0.0338 (9)	0.0033 (8)	0.0165 (8)	0.0014 (7)
C8	0.0408 (11)	0.0433 (11)	0.0449 (11)	-0.0065 (9)	0.0177 (9)	-0.0050 (9)
C9	0.0563 (14)	0.0431 (12)	0.0725 (14)	-0.0106 (10)	0.0326 (12)	-0.0082 (10)
C10	0.0637 (15)	0.0638 (14)	0.0549 (13)	-0.0065 (12)	0.0283 (12)	0.0179 (11)
C11	0.0588 (13)	0.0536 (12)	0.0389 (10)	-0.0124 (10)	0.0247 (9)	0.0023 (9)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

F1—C1	1.342 (2)	C3—C4	1.361 (3)
F2—C2	1.347 (2)	C4—H4	0.9300
F3—C3	1.349 (2)	C4—C5	1.383 (3)
O1—C9	1.424 (2)	C5—H5	0.9300
O1—C10	1.413 (3)	C5—C6	1.384 (2)
O2—C7	1.232 (2)	C8—H8A	0.9700
N1—C6	1.409 (2)	C8—H8B	0.9700
N1—C7	1.383 (2)	C8—C9	1.504 (3)
N1—H1	0.843 (18)	C9—H9A	0.9700
N2—C7	1.349 (2)	C9—H9B	0.9700
N2—C8	1.459 (2)	C10—H10A	0.9700
N2—C11	1.455 (2)	C10—H10B	0.9700
C1—C2	1.374 (3)	C10—C11	1.508 (3)
C1—C6	1.382 (2)	C11—H11A	0.9700
C2—C3	1.368 (3)	C11—H11B	0.9700
C10—O1—C9	109.68 (15)	O2—C7—N2	122.31 (16)
C6—N1—H1	116.2 (13)	N2—C7—N1	116.05 (15)
C7—N1—C6	121.05 (14)	N2—C8—H8A	109.5
C7—N1—H1	118.2 (13)	N2—C8—H8B	109.5
C7—N2—C8	119.83 (14)	N2—C8—C9	110.94 (15)
C7—N2—C11	123.95 (15)	H8A—C8—H8B	108.0
C11—N2—C8	113.93 (15)	C9—C8—H8A	109.5
F1—C1—C2	118.37 (17)	C9—C8—H8B	109.5

F1—C1—C6	120.70 (16)	O1—C9—C8	111.42 (17)
C2—C1—C6	120.91 (17)	O1—C9—H9A	109.3
F2—C2—C1	119.96 (17)	O1—C9—H9B	109.3
F2—C2—C3	120.24 (18)	C8—C9—H9A	109.3
C3—C2—C1	119.78 (18)	C8—C9—H9B	109.3
F3—C3—C2	118.52 (18)	H9A—C9—H9B	108.0
F3—C3—C4	120.61 (18)	O1—C10—H10A	109.3
C4—C3—C2	120.83 (18)	O1—C10—H10B	109.3
C3—C4—H4	120.4	O1—C10—C11	111.65 (17)
C3—C4—C5	119.28 (18)	H10A—C10—H10B	108.0
C5—C4—H4	120.4	C11—C10—H10A	109.3
C4—C5—H5	119.5	C11—C10—H10B	109.3
C4—C5—C6	121.08 (18)	N2—C11—C10	109.20 (16)
C6—C5—H5	119.5	N2—C11—H11A	109.8
C1—C6—N1	120.74 (15)	N2—C11—H11B	109.8
C1—C6—C5	117.99 (17)	C10—C11—H11A	109.8
C5—C6—N1	121.17 (16)	C10—C11—H11B	109.8
O2—C7—N1	121.59 (16)	H11A—C11—H11B	108.3

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ O2 <sup>i</sup>	0.843 (18)	2.120 (19)	2.9306 (19)	161.2 (17)

Symmetry codes: (i) *x*,  $-y+3/2$ , *z*+1/2.

Fig. 1

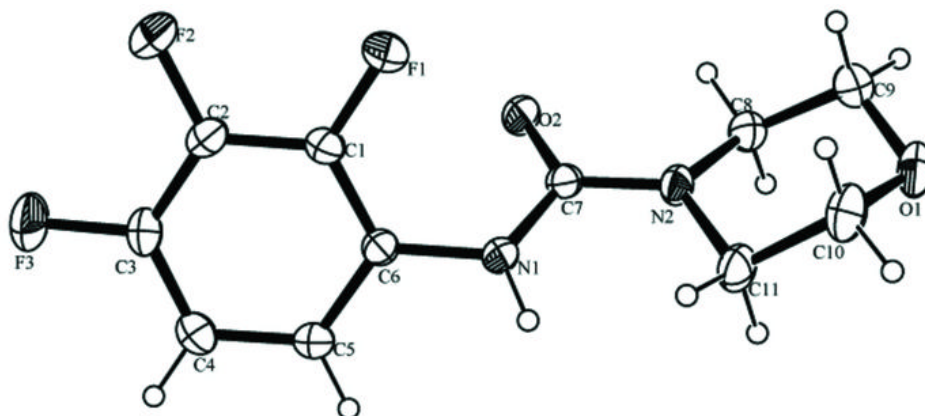




Fig. 2

