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4-(3-Fluoro-4-nitrophenyl)morpholin-3-one

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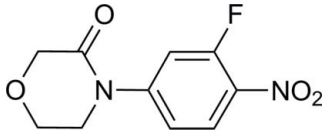
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.104; data-to-parameter ratio = 16.7.

In the title compound, $C_{10}H_9FN_2O_4$, the dihedral angle between the benzene ring and the nitro group plane is 11.29 (3)°. The morpholinone ring adopts a twist-chair conformation. In the crystal, molecules are linked by intermolecular C—H···O hydrogen bonds into a chain along the a-axis direction.

Related literature

The title compound is an intermediate in the preparation of derivatives of the factor Xa inhibitor rivaroxaban (systematic name (*S*)-5-chloro-*N*-{[2-oxo-3-[4-(3-oxomorpholin-4-yl)-phenyl]oxazolidin-5-yl]methyl}thiophene-2-carboxamide). For the bioactivity and applications of rivaroxaban, see: Pinto *et al.* (2010); Haas (2008); Squizzato *et al.* (2009); Samama & Gerotziafas (2010); Van Huis *et al.* (2009). For the synthesis of other derivatives with morpholone, see: Van Huis *et al.* (2009); Zbinden *et al.* (2009).



Experimental

Crystal data

$C_{10}H_9FN_2O_4$
M_r = 240.19
Triclinic, $P\overline{1}$
a = 6.6408 (7) Å
b = 7.3788 (10) Å
c = 10.8546 (14) Å
α = 73.30 (3)°
β = 75.39 (3)°
γ = 74.30 (3)°
V = 481.60 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 113 K
0.22 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: ψ scan (*CrystalClear*; Rigaku/MSC, 2009)
T_{min} = 0.970, T_{max} = 0.986
6470 measured reflections
2569 independent reflections
1734 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.104
S = 0.97
2569 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2^{i}	0.95	2.39	3.2635 (16)	153
C2—H2B···O3^{ii}	0.99	2.50	3.3244 (19)	140
C1—H1B···O4^{iii}	0.99	2.57	3.515 (2)	161

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: *CrystalStructure* (Rigaku/MSC, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2328).

References

Haas, S. (2008). *J. Thromb. Thrombolysis*, **25**, 52–60.
Pinto, D. J. P., Smallheer, J. M., Cheney, D. L., Knabb, R. M. & Wexler, R. R. (2010). *J. Med. Chem.* **53**, 6243–6274.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2009). *CrystalClear* and *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Samama, M. M. & Gerotziafas, G. T. (2010). *J. Thromb. Thrombolysis*, **29**, 92–104.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Squizzato, A., Dentali, F., Steidl, L. & Ageno, W. (2009). *Int. Emerg. Med.* **4**, 479–484.
Van Huis, C. A., *et al.* (2009). *Bioorg. Med. Chem.* **17**, 2501–2511.
Zbinden, K. G., *et al.* (2009). *Eur. J. Med. Chem.* **44**, 2787–2795.

supplementary materials

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4-(3-Fluoro-4-nitrophenyl)morpholin-3-one

C.-J. Huang, J. Wu, Z.-Q. Cai and J. Yuan

Comment

Rivaroxaban is an oral, direct factor Xa inhibitor for the prevention and treatment of arterial and venous thrombosis (Pinto *et al.*, 2010; Haas, 2008; Squizzato *et al.*, 2009).

The title compound (Fig. 1) is important intermediate in the preparation of derivatives of Rivaroxaban. Some derivatives of Rivaroxaban have been reported for having high affinity for human FXa (Squizzato *et al.*, 2009; Samama *et al.*, 2010; Van Huis *et al.*, 2009). Herein, the synthesis and the crystal structure of the title compound are reported.

In the title compound, $C_{10}H_9F_1N_2O_4$, the dihedral angle between benzene ring and the plane of nitro group is 11.29 (3)°. The morpholone ring adopts a twist-chair conformation. In the crystal packing molecules are linked by intermolecular C—H···O hydrogen bonds into a chain (Table 1).

Experimental

Potassium carbonate (6.73 g, 0.0488 mol) was added to a suspension of 2-(2-chloroethoxy)-*N*-(3-fluoro-4-nitrophenyl)acetamide (9.00 g, 0.0325 mol) in acetonitrile (200 mL).The reaction mixture was stirred at 385 K for 5 h. The mixture was evaporated in vacuo. Water was added.The reaction mixture was filtered, washed with water, and dried to obtain yellow solid (7.19 g).Colourless single crystals suitable for X-ray diffraction were obtained by recrystallisation from ethanol and ethyl acetate.

Refinement

All H atoms were geometrically positioned (C—H 0.95–0.99 Å) and treated as riding, with U_{iso}(H) = 1.2Ueq(C).

Figures

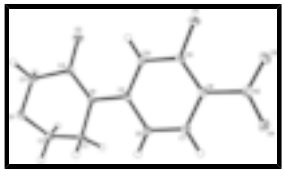

Fig. 1. The structure of $C_{10}H_9F_1N_2O_4$ with all non-H atom-labelling scheme and displacement ellipsoids drawn at the 50% probability level.

4-(3-Fluoro-4-nitrophenyl)morpholin-3-one

Crystal data

$C_{10}H_9FN_2O_4$	$Z = 2$
$M_r = 240.19$	$F(000) = 248$

Triclinic, $P\overline{1}$	D_x = 1.656 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 6.6408 (7) Å	Cell parameters from 1909 reflections
b = 7.3788 (10) Å	θ = 2.0–31.1°
c = 10.8546 (14) Å	μ = 0.14 mm^{-1}
α = 73.30 (3)°	T = 113 K
β = 75.39 (3)°	Prism, colourless
γ = 74.30 (3)°	0.22 × 0.20 × 0.10 mm
V = 481.60 (14) Å^3	

Data collection

Rigaku Saturn CCD area-detector diffractometer	2569 independent reflections
Radiation source: rotating anode	1734 reflections with $I > 2\sigma(I)$
multilayer	R_{int} = 0.041
Detector resolution: 14.63 pixels mm^{-1}	θ_{max} = 29.1°, θ_{min} = 2.0°
ω and φ scans	h = −9→9
Absorption correction: ψ scan (*CrystalClear*; Rigaku/MSC, 2009)	k = −10→9
T_{min} = 0.970, T_{max} = 0.986	l = −14→14
6470 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.039	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.104	H-atom parameters constrained
S = 0.97	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
2569 reflections	$(\Delta/\sigma)_{max} < 0.001$
154 parameters	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.25 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.38071 (13)	0.73231 (13)	−0.24062 (8)	0.0292 (2)
O1	0.23003 (15)	0.94560 (12)	0.40672 (8)	0.0192 (2)
O2	0.04787 (15)	0.82852 (15)	0.16307 (9)	0.0262 (2)
O3	0.74637 (16)	0.59970 (13)	−0.37865 (9)	0.0241 (2)
O4	1.03428 (15)	0.62270 (13)	−0.32888 (9)	0.0234 (2)
N1	0.39251 (16)	0.79057 (14)	0.18692 (10)	0.0139 (2)
N2	0.84043 (18)	0.63168 (15)	−0.30564 (10)	0.0165 (2)
C1	0.5325 (2)	0.77071 (18)	0.27977 (12)	0.0161 (3)
H1A	0.6152	0.8739	0.2456	0.019*
H1B	0.6347	0.6443	0.2855	0.019*
C2	0.4068 (2)	0.78419 (19)	0.41571 (12)	0.0201 (3)
H2A	0.3550	0.6634	0.4599	0.024*
H2B	0.5001	0.7996	0.4685	0.024*
C3	0.0821 (2)	0.90024 (19)	0.35316 (12)	0.0183 (3)
H3A	−0.0349	1.0155	0.3382	0.022*
H3B	0.0196	0.7956	0.4183	0.022*
C4	0.1736 (2)	0.83638 (18)	0.22515 (12)	0.0164 (3)
C5	0.4968 (2)	0.75696 (16)	0.06139 (12)	0.0132 (3)
C6	0.7205 (2)	0.71941 (17)	0.03016 (12)	0.0160 (3)
H6	0.7988	0.7192	0.0924	0.019*
C7	0.8283 (2)	0.68282 (17)	−0.09000 (12)	0.0161 (3)
H7	0.9795	0.6591	−0.1095	0.019*
C8	0.7182 (2)	0.68025 (17)	−0.18264 (12)	0.0143 (3)
C9	0.4967 (2)	0.72366 (18)	−0.15317 (12)	0.0158 (3)
C10	0.3858 (2)	0.76283 (17)	−0.03458 (12)	0.0160 (3)
H10	0.2345	0.7938	−0.0178	0.019*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0201 (5)	0.0495 (5)	0.0226 (4)	−0.0038 (4)	−0.0079 (4)	−0.0158 (4)
O1	0.0176 (5)	0.0226 (5)	0.0198 (5)	−0.0014 (4)	−0.0046 (4)	−0.0102 (4)
O2	0.0134 (5)	0.0476 (6)	0.0235 (5)	−0.0093 (5)	−0.0009 (4)	−0.0173 (5)
O3	0.0289 (6)	0.0285 (5)	0.0192 (5)	−0.0068 (4)	−0.0067 (4)	−0.0099 (4)
O4	0.0162 (5)	0.0297 (5)	0.0214 (5)	−0.0027 (4)	0.0012 (4)	−0.0078 (4)
N1	0.0125 (5)	0.0173 (5)	0.0125 (5)	−0.0031 (4)	−0.0020 (4)	−0.0049 (4)
N2	0.0192 (6)	0.0143 (5)	0.0148 (5)	−0.0029 (5)	−0.0020 (5)	−0.0028 (4)
C1	0.0135 (6)	0.0200 (7)	0.0159 (6)	−0.0013 (5)	−0.0055 (5)	−0.0056 (5)
C2	0.0196 (7)	0.0234 (7)	0.0158 (6)	0.0000 (6)	−0.0051 (5)	−0.0053 (5)
C3	0.0162 (7)	0.0218 (7)	0.0173 (6)	−0.0031 (5)	−0.0026 (5)	−0.0063 (5)
C4	0.0147 (7)	0.0177 (7)	0.0163 (6)	−0.0045 (5)	−0.0007 (5)	−0.0044 (5)
C5	0.0153 (6)	0.0117 (6)	0.0129 (6)	−0.0041 (5)	−0.0021 (5)	−0.0029 (5)
C6	0.0154 (7)	0.0182 (7)	0.0162 (6)	−0.0045 (5)	−0.0048 (5)	−0.0043 (5)
C7	0.0133 (6)	0.0181 (7)	0.0175 (6)	−0.0041 (5)	−0.0027 (5)	−0.0047 (5)

C8	0.0161 (7)	0.0134 (6)	0.0132 (6)	−0.0033 (5)	−0.0014 (5)	−0.0036 (5)
C9	0.0166 (7)	0.0180 (7)	0.0153 (6)	−0.0042 (5)	−0.0071 (5)	−0.0036 (5)
C10	0.0111 (6)	0.0188 (7)	0.0183 (6)	−0.0018 (5)	−0.0035 (5)	−0.0051 (5)

Geometric parameters (Å, °)

F1—C9	1.3438 (13)	C2—H2B	0.9900
O1—C3	1.4118 (14)	C3—C4	1.5223 (18)
O1—C2	1.4286 (16)	C3—H3A	0.9900
O2—C4	1.2193 (14)	C3—H3B	0.9900
O3—N2	1.2292 (13)	C5—C10	1.4050 (16)
O4—N2	1.2349 (13)	C5—C6	1.4061 (18)
N1—C4	1.3823 (16)	C6—C7	1.3831 (17)
N1—C5	1.4223 (16)	C6—H6	0.9500
N1—C1	1.4885 (15)	C7—C8	1.3910 (16)
N2—C8	1.4630 (16)	C7—H7	0.9500
C1—C2	1.5171 (18)	C8—C9	1.3912 (18)
C1—H1A	0.9900	C9—C10	1.3795 (18)
C1—H1B	0.9900	C10—H10	0.9500
C2—H2A	0.9900		
C3—O1—C2	107.55 (9)	C4—C3—H3B	108.5
C4—N1—C5	123.48 (11)	H3A—C3—H3B	107.5
C4—N1—C1	120.11 (10)	O2—C4—N1	124.24 (12)
C5—N1—C1	116.39 (10)	O2—C4—C3	117.50 (12)
O3—N2—O4	123.82 (11)	N1—C4—C3	118.25 (11)
O3—N2—C8	118.74 (11)	C10—C5—C6	118.08 (11)
O4—N2—C8	117.43 (10)	C10—C5—N1	122.88 (11)
N1—C1—C2	112.26 (10)	C6—C5—N1	119.03 (11)
N1—C1—H1A	109.2	C7—C6—C5	120.88 (12)
C2—C1—H1A	109.2	C7—C6—H6	119.6
N1—C1—H1B	109.2	C5—C6—H6	119.6
C2—C1—H1B	109.2	C6—C7—C8	120.88 (12)
H1A—C1—H1B	107.9	C6—C7—H7	119.6
O1—C2—C1	109.95 (10)	C8—C7—H7	119.6
O1—C2—H2A	109.7	C7—C8—C9	118.10 (12)
C1—C2—H2A	109.7	C7—C8—N2	118.60 (11)
O1—C2—H2B	109.7	C9—C8—N2	123.31 (11)
C1—C2—H2B	109.7	F1—C9—C10	116.88 (12)
H2A—C2—H2B	108.2	F1—C9—C8	121.12 (12)
O1—C3—C4	114.95 (11)	C10—C9—C8	121.99 (12)
O1—C3—H3A	108.5	C9—C10—C5	119.97 (12)
C4—C3—H3A	108.5	C9—C10—H10	120.0
O1—C3—H3B	108.5	C5—C10—H10	120.0
C4—N1—C1—C2	6.24 (15)	N1—C5—C6—C7	178.93 (10)
C5—N1—C1—C2	−172.77 (10)	C5—C6—C7—C8	−0.66 (19)
C3—O1—C2—C1	70.39 (13)	C6—C7—C8—C9	2.70 (19)
N1—C1—C2—O1	−46.84 (14)	C6—C7—C8—N2	−177.12 (11)
C2—O1—C3—C4	−52.83 (13)	O3—N2—C8—C7	168.82 (11)
C5—N1—C4—O2	9.8 (2)	O4—N2—C8—C7	−10.19 (16)

C1—N1—C4—O2	−169.11 (11)	O3—N2—C8—C9	−10.99 (18)
C5—N1—C4—C3	−170.40 (11)	O4—N2—C8—C9	170.00 (11)
C1—N1—C4—C3	10.66 (17)	C7—C8—C9—F1	176.97 (10)
O1—C3—C4—O2	−167.37 (11)	N2—C8—C9—F1	−3.22 (19)
O1—C3—C4—N1	12.84 (16)	C7—C8—C9—C10	−1.95 (19)
C4—N1—C5—C10	−1.57 (18)	N2—C8—C9—C10	177.87 (11)
C1—N1—C5—C10	177.41 (11)	F1—C9—C10—C5	−179.83 (10)
C4—N1—C5—C6	177.30 (11)	C8—C9—C10—C5	−0.87 (19)
C1—N1—C5—C6	−3.73 (16)	C6—C5—C10—C9	2.90 (18)
C10—C5—C6—C7	−2.16 (18)	N1—C5—C10—C9	−178.23 (11)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O2^{i}	0.95	2.39	3.2635 (16)	153
C2—H2B···O3^{ii}	0.99	2.50	3.3244 (19)	140
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Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*, *y*, *z*+1; (iii) −*x*+2, −*y*+1, −*z*.

Fig. 1