organic compounds

4-(2-Oxa-6-azaspiro[3.3]hept-6-yl)-benzonitrile

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In the title compound, $C_{12}H_{12}N_2O$, the azetidine ring (r.m.s. deviation = 0.021 Å) and the oxetane ring (r.m.s. deviation = 0.014 Å) are nearly perpendicular to each other [dihedral angle = 89.7 (1)°]. The azetidine ring is twisted out of the plane of the benzene ring by 18.3 (1)°. In the crystal structure, molecules are linked to form chains along the c axis by C—H···O hydrogen bonds.

Related literature

The title compound is a key intermediate to synthesize (pyrrolo[3,4-c]pyrazol-3-yl)benzamide derivatives. For the anti-tumor effect of these derivatives, see: Fancelli *et al.* (2005).
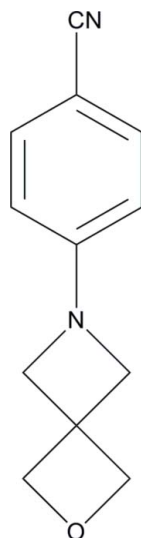
Experimental

Crystal data

$C_{12}H_{12}N_2O$	V = 1002.2 (7) Å^3
M_r = 200.24	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 9.484 (4) Å	μ = 0.09 mm^{-1}
b = 11.033 (4) Å	T = 113 K
c = 10.419 (4) Å	$0.60 \times 0.60 \times 0.27$ mm
β = 113.186 (5)°	

Data collection

Rigaku AFC10/Saturn 724-Plus diffractometer	2234 independent reflections
	1872 reflections with $I > 2\sigma(I)$
7662 measured reflections	R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041	136 parameters
$wR(F^2)$ = 0.099	H-atom parameters constrained
S = 1.00	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
2234 reflections	$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^i	0.95	2.47	3.371 (2)	159

Symmetry code: (i) $x, y, z + 1$.

References

Fancelli, D., Berta, D., Bindi, S., Cameron, A., Cappella, P., Carpinelli, P., Catana, C., Forte, B., Giordano, P., Giorgini, M. L., Mantegani, S., Marsiglio, A., Meroni, M., Moll, J., Pittal\'a, V., Roletto, F., Severino, D., Soncini, C., Storici, P., Tonani, R., Varasi, M., Vulpetti, A. & Vianello, P.*et al.* (2005). *J. Med. Chem.* **48**, 3080–3084.

Rigaku (2008). *CrystalClear*. Rigaku Corporation, The Woodlands, Texas, USA.

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supporting information

4-(2-Oxa-6-azaspiro[3.3]hept-6-yl)benzonitrile

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S1. Experimental

A DMSO solution of 2-oxa-6-azaspiro[3,3]heptane (0.99 g, 0.01 mol) with 4-fluorobenzonitrile (1.21 g, 0.01 mol) was heated to reflux for 5 h, then water (100 ml) was added into the solution. The mixture was extracted with CH_2Cl_2. Then the solvent was removed to give a red powder. Single crystals were obtained from a CH_2Cl_2 solution after 3 days.

S2. Refinement

H atoms were placed in the calculated positions (C—H = 0.95–0.99 Å) and refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$.
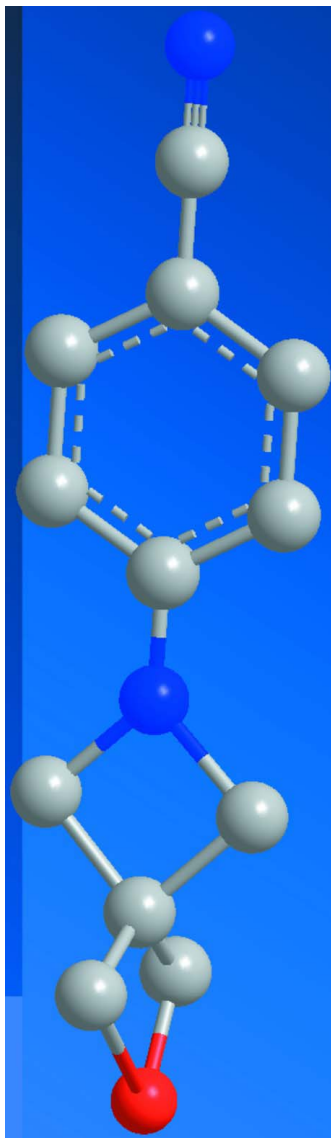
Figure 1

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4-(2-Oxa-6-azaspiro[3.3]hept-6-yl)benzonitrile

Crystal data

$C_{12}H_{12}N_2O$	$F(000) = 424$
$M_r = 200.24$	$D_x = 1.327$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.484$ (4) Å	Cell parameters from 2956 reflections
$b = 11.033$ (4) Å	$\theta = 3.1–27.5°$
$c = 10.419$ (4) Å	$\mu = 0.09$ mm^{-1}
$\beta = 113.186$ (5)°	$T = 113$ K
$V = 1002.2$ (7) Å^3	Prism, colourless
$Z = 4$	$0.60 \times 0.60 \times 0.27$ mm

Data collection

Rigaku AFC10/Saturn 724-Plus diffractometer	2234 independent reflections
Radiation source: rotating anode	1872 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.029$
Detector resolution: 28.5714 pixels mm⁻¹	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
φ and ω scans	$h = -12 \rightarrow 12$
7662 measured reflections	$k = -14 \rightarrow 11$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.099$	
$S = 1.00$	H-atom parameters constrained
2234 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 0.36P]$ where $P = (F_o^2 + 2F_c^2)/3$
136 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.30$ e Å⁻³
	$\Delta\rho_{min} = -0.16$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
O1	0.53098 (12)	0.21116 (9)	−0.00129 (10)	0.0272 (3)
N1	0.81838 (14)	0.46062 (12)	1.07793 (13)	0.0275 (3)
N2	0.58259 (13)	0.37004 (10)	0.38573 (11)	0.0186 (3)
C1	0.74599 (15)	0.47583 (12)	0.59326 (14)	0.0178 (3)
H1	0.7933	0.5192	0.5421	0.021*
C2	0.79050 (15)	0.49646 (12)	0.73352 (14)	0.0186 (3)
H2	0.8679	0.5547	0.7788	0.022*
C3	0.72270 (15)	0.43219 (12)	0.81086 (14)	0.0177 (3)
C4	0.60810 (15)	0.34638 (12)	0.74350 (14)	0.0183 (3)
H4	0.5624	0.3022	0.7954	0.022*
C5	0.56183 (15)	0.32601 (12)	0.60278 (14)	0.0178 (3)
H5	0.4835	0.2683	0.5577	0.021*
C6	0.62984 (14)	0.39026 (12)	0.52463 (13)	0.0165 (3)
C7	0.49702 (15)	0.26581 (12)	0.30534 (14)	0.0178 (3)
H7A	0.3842	0.2751	0.2702	0.021*
H7B	0.5303	0.1871	0.3534	0.021*
C8	0.56407 (14)	0.29329 (12)	0.19522 (14)	0.0167 (3)

C9	0.65782 (15)	0.39702 (12)	0.28976 (13)	0.0177 (3)
H9A	0.7699	0.3827	0.3302	0.021*
H9B	0.6331	0.4782	0.2461	0.021*
C10	0.45979 (16)	0.31151 (12)	0.04116 (14)	0.0204 (3)
H10A	0.3499	0.2979	0.0208	0.024*
H10B	0.4749	0.3907	0.0037	0.024*
C11	0.63751 (16)	0.19237 (13)	0.14221 (14)	0.0226 (3)
H11A	0.7455	0.2092	0.1575	0.027*
H11B	0.6281	0.1114	0.1790	0.027*
C12	0.77569 (15)	0.44870 (12)	0.95891 (15)	0.0202 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0353 (6)	0.0266 (6)	0.0202 (5)	0.0027 (5)	0.0113 (5)	−0.0041 (4)
N1	0.0257 (7)	0.0323 (7)	0.0231 (7)	−0.0015 (5)	0.0082 (5)	−0.0038 (6)
N2	0.0203 (6)	0.0187 (6)	0.0180 (6)	−0.0073 (4)	0.0086 (5)	−0.0027 (5)
C1	0.0179 (6)	0.0147 (6)	0.0217 (7)	−0.0007 (5)	0.0086 (5)	0.0028 (5)
C2	0.0156 (6)	0.0151 (6)	0.0223 (7)	0.0007 (5)	0.0043 (5)	−0.0017 (5)
C3	0.0165 (6)	0.0176 (6)	0.0177 (7)	0.0041 (5)	0.0054 (5)	−0.0004 (5)
C4	0.0177 (6)	0.0181 (6)	0.0212 (7)	0.0025 (5)	0.0098 (5)	0.0022 (5)
C5	0.0157 (6)	0.0163 (6)	0.0212 (7)	−0.0017 (5)	0.0069 (5)	−0.0012 (5)
C6	0.0151 (6)	0.0147 (6)	0.0188 (7)	0.0023 (5)	0.0058 (5)	0.0008 (5)
C7	0.0184 (6)	0.0157 (6)	0.0195 (7)	−0.0029 (5)	0.0076 (5)	−0.0014 (5)
C8	0.0161 (6)	0.0149 (6)	0.0193 (7)	0.0004 (5)	0.0071 (5)	0.0002 (5)
C9	0.0171 (6)	0.0186 (6)	0.0172 (6)	−0.0020 (5)	0.0065 (5)	0.0004 (5)
C10	0.0210 (7)	0.0184 (6)	0.0195 (7)	−0.0010 (5)	0.0057 (5)	−0.0002 (6)
C11	0.0257 (7)	0.0215 (7)	0.0227 (7)	0.0046 (6)	0.0117 (6)	0.0016 (6)
C12	0.0175 (6)	0.0195 (6)	0.0235 (8)	0.0024 (5)	0.0079 (5)	−0.0010 (6)

Geometric parameters (Å, º)

O1—C11	1.4525 (17)	C5—C6	1.4125 (19)
O1—C10	1.4534 (17)	C5—H5	0.95
N1—C12	1.1504 (18)	C7—C8	1.5451 (18)
N2—C6	1.3544 (17)	C7—H7A	0.99
N2—C7	1.4653 (17)	C7—H7B	0.99
N2—C9	1.4695 (17)	C8—C11	1.5274 (18)
C1—C2	1.3711 (19)	C8—C10	1.5314 (18)
C1—C6	1.4127 (18)	C8—C9	1.5431 (18)
C1—H1	0.95	C9—H9A	0.99
C2—C3	1.4049 (19)	C9—H9B	0.99
C2—H2	0.95	C10—H10A	0.99
C3—C4	1.4034 (19)	C10—H10B	0.99
C3—C12	1.4335 (19)	C11—H11A	0.99
C4—C5	1.3734 (19)	C11—H11B	0.99
C4—H4	0.95		

C11—O1—C10	90.79 (9)	H7A—C7—H7B	111.1
C6—N2—C7	128.02 (11)	C11—C8—C10	85.12 (10)
C6—N2—C9	130.44 (11)	C11—C8—C9	122.78 (11)
C7—N2—C9	94.47 (10)	C10—C8—C9	122.94 (11)
C2—C1—C6	120.13 (12)	C11—C8—C7	120.34 (11)
C2—C1—H1	119.9	C10—C8—C7	121.30 (11)
C6—C1—H1	119.9	C9—C8—C7	88.49 (10)
C1—C2—C3	120.61 (12)	N2—C9—C8	88.40 (10)
C1—C2—H2	119.7	N2—C9—H9A	113.9
C3—C2—H2	119.7	C8—C9—H9A	113.9
C4—C3—C2	119.53 (12)	N2—C9—H9B	113.9
C4—C3—C12	119.89 (12)	C8—C9—H9B	113.9
C2—C3—C12	120.49 (12)	H9A—C9—H9B	111.1
C5—C4—C3	120.22 (12)	O1—C10—C8	91.91 (10)
C5—C4—H4	119.9	O1—C10—H10A	113.3
C3—C4—H4	119.9	C8—C10—H10A	113.3
C4—C5—C6	120.45 (12)	O1—C10—H10B	113.3
C4—C5—H5	119.8	C8—C10—H10B	113.3
C6—C5—H5	119.8	H10A—C10—H10B	110.6
N2—C6—C5	119.95 (12)	O1—C11—C8	92.11 (10)
N2—C6—C1	121.00 (12)	O1—C11—H11A	113.3
C5—C6—C1	119.05 (12)	C8—C11—H11A	113.3
N2—C7—C8	88.47 (10)	O1—C11—H11B	113.3
N2—C7—H7A	113.9	C8—C11—H11B	113.3
C8—C7—H7A	113.9	H11A—C11—H11B	110.6
N2—C7—H7B	113.9	N1—C12—C3	179.26 (15)
C8—C7—H7B	113.9		
C6—C1—C2—C3	0.67 (19)	N2—C7—C8—C11	130.78 (12)
C1—C2—C3—C4	−0.17 (18)	N2—C7—C8—C10	−125.31 (12)
C1—C2—C3—C12	176.30 (12)	N2—C7—C8—C9	3.04 (9)
C2—C3—C4—C5	−0.45 (19)	C6—N2—C9—C8	154.66 (13)
C12—C3—C4—C5	−176.95 (12)	C7—N2—C9—C8	3.21 (10)
C3—C4—C5—C6	0.57 (19)	C11—C8—C9—N2	−128.76 (12)
C7—N2—C6—C5	−18.4 (2)	C10—C8—C9—N2	123.99 (12)
C9—N2—C6—C5	−161.23 (13)	C7—C8—C9—N2	−3.04 (9)
C7—N2—C6—C1	162.08 (12)	C11—O1—C10—C8	−2.22 (10)
C9—N2—C6—C1	19.3 (2)	C11—C8—C10—O1	2.12 (10)
C4—C5—C6—N2	−179.56 (12)	C9—C8—C10—O1	128.42 (12)
C4—C5—C6—C1	−0.07 (19)	C7—C8—C10—O1	−120.66 (12)
C2—C1—C6—N2	178.93 (12)	C10—O1—C11—C8	2.22 (10)
C2—C1—C6—C5	−0.55 (19)	C10—C8—C11—O1	−2.12 (10)
C6—N2—C7—C8	−155.71 (13)	C9—C8—C11—O1	−128.56 (12)
C9—N2—C7—C8	−3.21 (10)	C7—C8—C11—O1	121.54 (12)

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C4—H4⋯O1[i]	0.95	2.47	3.371 (2)	159

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