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4-[4-(Piperidin-1-yl)piperidin-1-yl]benzonitrile

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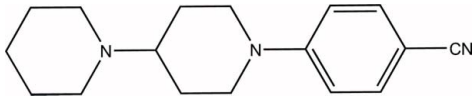
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.111; data-to-parameter ratio = 19.2.

In the title compound, $C_{17}H_{23}N_3$, both piperidine rings adopt chair conformations. In the crystal packing, intermolecular C—H···N hydrogen bonds and C—H···π interactions are present.

Related literature

For general background, see: Pevarello *et al.* (2006).



Experimental

Crystal data

$C_{17}H_{23}N_3$
M_r = 269.38
Monoclinic, $P2_1/c$
a = 10.090 (2) Å
b = 11.100 (2) Å
c = 13.446 (3) Å
β = 100.72 (3)°
V = 1479.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 113 K
0.26 × 0.25 × 0.20 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.981, T_{max} = 0.986
11970 measured reflections
3500 independent reflections
2749 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.111
S = 1.12
3500 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

Cg is the centroid of the C11–C16 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···Cg^{i}	1.00	2.99	3.9363 (14)	158
C16—H16···N3^{ii}	0.95	2.54	3.3442 (16)	143

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ChemBioDraw Ultra* CambridgeSoft (2008); software used to prepare material for publication: *SHELXL97*.

The authors thank Mr Zhi-Hua Mao of Sichuan University for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2240).

References

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Pevarello, P., Fancelli, D., Vulpetti, A., Amici, R., Villa, M., Pittalà, V., Vianello, P., Cameron, A., Ciomei, M., Mercurio, C., Bischoff, J. R., Roletto, F., Varasi, M. & Brasca, M. G. (2006). *Bioorg. Med. Chem. Lett.* **16**, 1084–1090.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o284 [https://doi.org/10.1107/S160053680905507X]

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S1. Comment

4-(4-(Piperidin-1-yl)piperidin-1-yl)benzonitrile are key intermediates which can be used to synthesize 3-aminopyrazole derivatives, which can be used as precursors for anticancer and anti-malarial agents. In the structure of the title molecule (Fig. 1) both piperidine rings are in a chair conformation. A crystal packing is dominated by van der Waals interactions (Fig. 2).

S2. Experimental

A DMSO solution of 1-(piperidin-4-yl)piperidine (4.37 g, 0.01 mol) with 4-fluorobenzonitrile (1.21 g, 0.01 mol) was heated to reflux for 3 h, then water (50 ml) was added into the solution. The mixture was extracted with CH_2Cl_2. After the solvent was removed a red crystalline powder was obtained; its recrystallisation from a methanol solution after 5 days yielded single crystals.

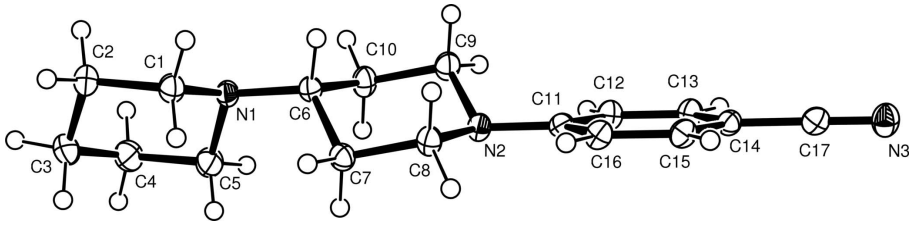

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

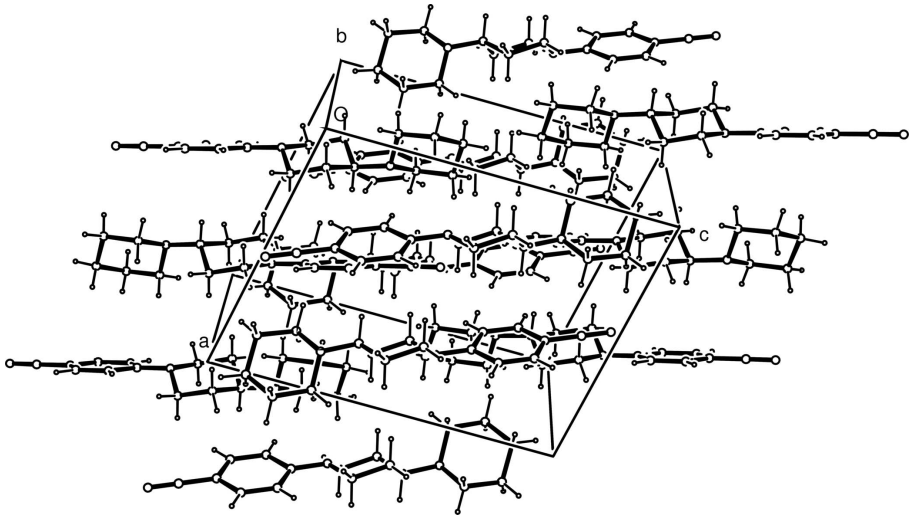

Figure 2

The crystal packing of I dominated by van der Waals interactions.

4-[4-(Piperidin-1-yl)piperidin-1-yl]benzonitrile

Crystal data

$C_{17}H_{23}N_3$
$M_r = 269.38$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.090$ (2) Å
$b = 11.100$ (2) Å
$c = 13.446$ (3) Å
$\beta = 100.72$ (3)°
$V = 1479.7$ (5) Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.209$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4488 reflections
$\theta = 2.7$–27.9°
$\mu = 0.07$ mm^{-1}
$T = 113$ K
Block, red
0.26 × 0.25 × 0.20 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.981$, $T_{max} = 0.986$
11970 measured reflections
3500 independent reflections
2749 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.8°$
$h = -13\rightarrow10$
$k = -14\rightarrow14$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.111$
$S = 1.12$
3500 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.0668P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.033 (7)

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}
N1	0.19321 (8)	0.59022 (8)	0.73578 (6)	0.0193 (2)
N2	0.26675 (8)	0.49828 (7)	0.43659 (6)	0.0187 (2)
N3	0.48688 (9)	0.35177 (8)	0.00345 (7)	0.0256 (2)
C1	0.21163 (10)	0.49931 (10)	0.81631 (8)	0.0225 (2)
H1A	0.3069	0.4727	0.8304	0.027*
H1B	0.1547	0.4283	0.7934	0.027*
C2	0.17411 (11)	0.54896 (10)	0.91273 (8)	0.0270 (3)
H2A	0.2363	0.6154	0.9391	0.032*
H2B	0.1839	0.4848	0.9647	0.032*
C3	0.02945 (11)	0.59541 (11)	0.89283 (9)	0.0310 (3)
H3A	−0.0339	0.5270	0.8768	0.037*
H3B	0.0102	0.6360	0.9542	0.037*
C4	0.00956 (11)	0.68349 (11)	0.80492 (9)	0.0307 (3)
H4A	0.0638	0.7569	0.8249	0.037*
H4B	−0.0865	0.7074	0.7881	0.037*
C5	0.05156 (10)	0.62790 (11)	0.71205 (8)	0.0274 (3)
H5A	−0.0062	0.5573	0.6894	0.033*
H5B	0.0392	0.6875	0.6563	0.033*
C6	0.24849 (10)	0.54949 (9)	0.64737 (8)	0.0182 (2)
H6	0.3448	0.5272	0.6733	0.022*
C7	0.18209 (10)	0.43925 (9)	0.59102 (8)	0.0205 (2)
H7A	0.1816	0.3721	0.6394	0.025*
H7B	0.0873	0.4584	0.5608	0.025*
C8	0.25693 (10)	0.40038 (9)	0.50799 (8)	0.0204 (2)
H8A	0.3488	0.3734	0.5391	0.024*
H8B	0.2093	0.3313	0.4708	0.024*
C9	0.32454 (11)	0.60897 (9)	0.48736 (8)	0.0224 (2)
H9A	0.3206	0.6742	0.4366	0.027*
H9B	0.4206	0.5948	0.5172	0.027*
C10	0.25036 (11)	0.64891 (9)	0.56997 (8)	0.0229 (2)
H10A	0.1566	0.6711	0.5394	0.027*
H10B	0.2951	0.7211	0.6042	0.027*

C11	0.31345 (9)	0.46754 (9)	0.34801 (7)	0.0179 (2)
C12	0.32252 (10)	0.55520 (9)	0.27405 (8)	0.0209 (2)
H12	0.2976	0.6360	0.2849	0.025*
C13	0.36689 (10)	0.52613 (9)	0.18606 (8)	0.0212 (2)
H13	0.3727	0.5870	0.1374	0.025*
C14	0.40341 (10)	0.40774 (9)	0.16801 (8)	0.0182 (2)
C15	0.39452 (10)	0.31974 (9)	0.24037 (8)	0.0203 (2)
H15	0.4191	0.2390	0.2289	0.024*
C16	0.35023 (10)	0.34886 (9)	0.32857 (8)	0.0205 (2)
H16	0.3445	0.2876	0.3770	0.025*
C17	0.45003 (10)	0.37726 (9)	0.07679 (8)	0.0200 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0189 (4)	0.0234 (5)	0.0162 (4)	0.0020 (3)	0.0049 (3)	−0.0006 (3)
N2	0.0253 (4)	0.0146 (4)	0.0169 (4)	−0.0026 (3)	0.0060 (3)	−0.0002 (3)
N3	0.0337 (5)	0.0195 (5)	0.0252 (5)	−0.0013 (4)	0.0101 (4)	−0.0025 (4)
C1	0.0240 (5)	0.0248 (5)	0.0194 (5)	0.0000 (4)	0.0053 (4)	0.0015 (4)
C2	0.0287 (6)	0.0343 (6)	0.0190 (6)	−0.0034 (5)	0.0071 (4)	−0.0014 (5)
C3	0.0272 (6)	0.0414 (7)	0.0273 (6)	−0.0046 (5)	0.0127 (5)	−0.0073 (5)
C4	0.0237 (5)	0.0400 (7)	0.0300 (6)	0.0065 (5)	0.0085 (5)	−0.0051 (5)
C5	0.0215 (5)	0.0370 (6)	0.0237 (6)	0.0071 (5)	0.0044 (4)	−0.0020 (5)
C6	0.0183 (5)	0.0197 (5)	0.0170 (5)	0.0005 (4)	0.0045 (4)	−0.0008 (4)
C7	0.0226 (5)	0.0200 (5)	0.0196 (5)	−0.0024 (4)	0.0058 (4)	0.0011 (4)
C8	0.0272 (5)	0.0160 (5)	0.0189 (5)	−0.0022 (4)	0.0069 (4)	0.0008 (4)
C9	0.0287 (5)	0.0187 (5)	0.0210 (5)	−0.0062 (4)	0.0076 (4)	−0.0031 (4)
C10	0.0315 (6)	0.0177 (5)	0.0212 (6)	−0.0024 (4)	0.0094 (4)	−0.0023 (4)
C11	0.0178 (5)	0.0183 (5)	0.0170 (5)	−0.0011 (4)	0.0018 (4)	−0.0009 (4)
C12	0.0263 (5)	0.0161 (5)	0.0209 (5)	0.0028 (4)	0.0059 (4)	0.0000 (4)
C13	0.0268 (5)	0.0187 (5)	0.0185 (5)	0.0012 (4)	0.0054 (4)	0.0027 (4)
C14	0.0196 (5)	0.0181 (5)	0.0171 (5)	−0.0009 (4)	0.0033 (4)	−0.0014 (4)
C15	0.0238 (5)	0.0164 (5)	0.0205 (6)	0.0007 (4)	0.0033 (4)	−0.0017 (4)
C16	0.0253 (5)	0.0170 (5)	0.0187 (5)	−0.0007 (4)	0.0031 (4)	0.0011 (4)
C17	0.0236 (5)	0.0145 (5)	0.0216 (6)	−0.0012 (4)	0.0038 (4)	0.0000 (4)

Geometric parameters (Å, º)

N1—C5	1.4663 (13)	C6—H6	1.0000
N1—C1	1.4664 (13)	C7—C8	1.5219 (14)
N1—C6	1.4749 (13)	C7—H7A	0.9900
N2—C11	1.4020 (13)	C7—H7B	0.9900
N2—C8	1.4656 (13)	C8—H8A	0.9900
N2—C9	1.4723 (13)	C8—H8B	0.9900
N3—C17	1.1520 (13)	C9—C10	1.5164 (14)
C1—C2	1.5200 (14)	C9—H9A	0.9900
C1—H1A	0.9900	C9—H9B	0.9900
C1—H1B	0.9900	C10—H10A	0.9900

C2—C3	1.5241 (16)	C10—H10B	0.9900
C2—H2A	0.9900	C11—C16	1.4063 (14)
C2—H2B	0.9900	C11—C12	1.4064 (14)
C3—C4	1.5183 (17)	C12—C13	1.3788 (14)
C3—H3A	0.9900	C12—H12	0.9500
C3—H3B	0.9900	C13—C14	1.3981 (14)
C4—C5	1.5220 (15)	C13—H13	0.9500
C4—H4A	0.9900	C14—C15	1.3933 (14)
C4—H4B	0.9900	C14—C17	1.4335 (14)
C5—H5A	0.9900	C15—C16	1.3811 (14)
C5—H5B	0.9900	C15—H15	0.9500
C6—C10	1.5195 (14)	C16—H16	0.9500
C6—C7	1.5269 (14)		

C5—N1—C1	109.98 (8)	C8—C7—H7A	109.4
C5—N1—C6	114.29 (8)	C6—C7—H7A	109.4
C1—N1—C6	111.67 (8)	C8—C7—H7B	109.4
C11—N2—C8	116.77 (8)	C6—C7—H7B	109.4
C11—N2—C9	115.49 (8)	H7A—C7—H7B	108.0
C8—N2—C9	112.59 (8)	N2—C8—C7	111.93 (8)
N1—C1—C2	111.27 (9)	N2—C8—H8A	109.2
N1—C1—H1A	109.4	C7—C8—H8A	109.2
C2—C1—H1A	109.4	N2—C8—H8B	109.2
N1—C1—H1B	109.4	C7—C8—H8B	109.2
C2—C1—H1B	109.4	H8A—C8—H8B	107.9
H1A—C1—H1B	108.0	N2—C9—C10	112.14 (8)
C1—C2—C3	110.81 (9)	N2—C9—H9A	109.2
C1—C2—H2A	109.5	C10—C9—H9A	109.2
C3—C2—H2A	109.5	N2—C9—H9B	109.2
C1—C2—H2B	109.5	C10—C9—H9B	109.2
C3—C2—H2B	109.5	H9A—C9—H9B	107.9
H2A—C2—H2B	108.1	C9—C10—C6	111.10 (8)
C4—C3—C2	109.76 (9)	C9—C10—H10A	109.4
C4—C3—H3A	109.7	C6—C10—H10A	109.4
C2—C3—H3A	109.7	C9—C10—H10B	109.4
C4—C3—H3B	109.7	C6—C10—H10B	109.4
C2—C3—H3B	109.7	H10A—C10—H10B	108.0
H3A—C3—H3B	108.2	N2—C11—C16	121.86 (9)
C3—C4—C5	111.16 (10)	N2—C11—C12	120.57 (9)
C3—C4—H4A	109.4	C16—C11—C12	117.55 (9)
C5—C4—H4A	109.4	C13—C12—C11	121.27 (9)
C3—C4—H4B	109.4	C13—C12—H12	119.4
C5—C4—H4B	109.4	C11—C12—H12	119.4
H4A—C4—H4B	108.0	C12—C13—C14	120.46 (9)
N1—C5—C4	110.24 (9)	C12—C13—H13	119.8
N1—C5—H5A	109.6	C14—C13—H13	119.8
C4—C5—H5A	109.6	C15—C14—C13	118.98 (9)
N1—C5—H5B	109.6	C15—C14—C17	120.39 (9)

C4—C5—H5B	109.6	C13—C14—C17	120.63 (9)
H5A—C5—H5B	108.1	C16—C15—C14	120.57 (9)
N1—C6—C10	112.59 (8)	C16—C15—H15	119.7
N1—C6—C7	116.67 (8)	C14—C15—H15	119.7
C10—C6—C7	107.60 (8)	C15—C16—C11	121.17 (9)
N1—C6—H6	106.4	C15—C16—H16	119.4
C10—C6—H6	106.4	C11—C16—H16	119.4
C7—C6—H6	106.4	N3—C17—C14	179.37 (11)
C8—C7—C6	111.06 (8)		

C5—N1—C1—C2	−60.67 (11)	N2—C9—C10—C6	56.38 (12)
C6—N1—C1—C2	171.35 (8)	N1—C6—C10—C9	172.41 (8)
N1—C1—C2—C3	56.80 (12)	C7—C6—C10—C9	−57.65 (11)
C1—C2—C3—C4	−52.73 (12)	C8—N2—C11—C16	−0.48 (13)
C2—C3—C4—C5	53.76 (12)	C9—N2—C11—C16	135.34 (10)
C1—N1—C5—C4	60.92 (12)	C8—N2—C11—C12	178.25 (9)
C6—N1—C5—C4	−172.56 (9)	C9—N2—C11—C12	−45.93 (12)
C3—C4—C5—N1	−58.25 (12)	N2—C11—C12—C13	−179.30 (9)
C5—N1—C6—C10	63.26 (11)	C16—C11—C12—C13	−0.52 (14)
C1—N1—C6—C10	−171.10 (8)	C11—C12—C13—C14	0.37 (15)
C5—N1—C6—C7	−61.87 (12)	C12—C13—C14—C15	−0.13 (15)
C1—N1—C6—C7	63.77 (11)	C12—C13—C14—C17	−179.78 (9)
N1—C6—C7—C8	−174.76 (8)	C13—C14—C15—C16	0.05 (15)
C10—C6—C7—C8	57.63 (11)	C17—C14—C15—C16	179.70 (9)
C11—N2—C8—C7	−169.71 (8)	C14—C15—C16—C11	−0.21 (15)
C9—N2—C8—C7	53.24 (11)	N2—C11—C16—C15	179.21 (9)
C6—C7—C8—N2	−56.30 (11)	C12—C11—C16—C15	0.44 (15)
C11—N2—C9—C10	169.02 (8)	C15—C14—C17—N3	38 (9)
C8—N2—C9—C10	−53.35 (11)	C13—C14—C17—N3	−142 (9)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C11–C16 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···*Cg*^{i}	1.00	2.99	3.9363 (14)	158
C16—H16···N3^{ii}	0.95	2.54	3.3442 (16)	143

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