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2,4-Bis(4-fluorophenyl)-3-azabicyclo-[3.3.1]nonan-9-one

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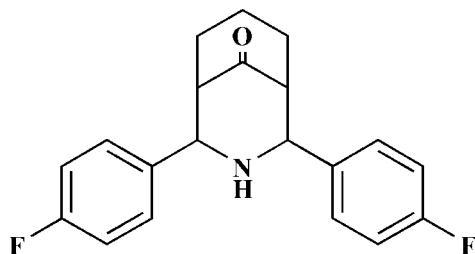
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.141; data-to-parameter ratio = 17.5.

In the title compound, $\text{C}_{20}\text{H}_{19}\text{F}_2\text{NO}$, a crystallographic mirror plane bisects the molecule, passing through the N, O and two C atoms of the central ring system. The molecule exists in a twin-chair conformation with equatorial dispositions of the 4-fluorophenyl groups on both sides of the secondary amino groups; the dihedral angle between the aromatic ring planes is 28.67 (3)°.

Related literature

For chemical background, see: Buxton & Roberts (1996); Evans & Seddon (1997); Ramachandran *et al.* (2007). For ring puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{19}\text{F}_2\text{NO}$
 $M_r = 327.36$
 Orthorhombic, $Pnma$
 $a = 7.6153$ (3) Å
 $b = 21.1392$ (9) Å
 $c = 10.0878$ (4) Å
 $V = 1623.95$ (11) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 298$ (2) K
 $0.35 \times 0.32 \times 0.30$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.967$, $T_{\max} = 0.971$
 11360 measured reflections
 2064 independent reflections
 1596 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.141$
 $S = 0.91$
 2064 reflections
 118 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97..

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2763).

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supporting information

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2,4-Bis(4-fluorophenyl)-3-azabicyclo[3.3.1]nonan-9-one

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S1. Comment

Fluorine substituted organic compounds are very important due to the significance of C—F bonds in some bioorganic systems (e.g. Evans & Seddon, 1997). The intermolecular and intramolecular hydrogen bonds involving fluorine atom have attracted much attention in various aspects (e.g. Ramachandran *et al.*, 2007). Moreover, the biological activities mainly depend on the stereochemistry of the synthesized compound (e.g. Buxton & Roberts, 1996). Hence, realising the importance of the investigation of the conformation, stereochemistry and the nature of bondings in the synthesized title fluorine substituted heterocycle, (I), we have carried out single-crystal X-ray diffraction studies.

An analysis of torsion angles, asymmetry parameters and least-squares plane calculation shows that the piperidine ring adopts a near ideal chair conformation with the deviation of ring atoms N1 and C5 from the C1/C1ⁱ/C2/C2ⁱ (*i* = x, 1/2-*y*, *z*) plane by -0.670 (3) Å and 0.693 (3) Å respectively, $Q_T = 0.6064$ (13) Å. The cyclohexane ring deviate from the ideal chair conformation by the deviation of ring atoms C4 and C5 from the C2/C2ⁱ/C3/C3ⁱ plane by 0.522 (4) Å and 0.734 (3) Å respectively, $Q_T = 0.5681$ (14) Å (Cremer & Pople, 1975).

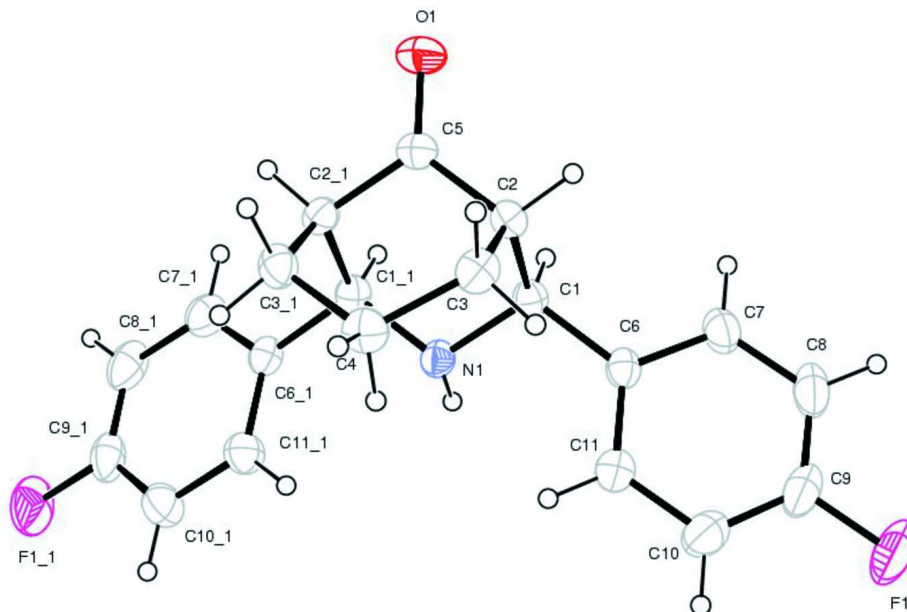
Compound (I) has a crystallographic mirror plane, which bisects the molecule passing through N1, C4, C5 and O1 of the central ring (Fig. 1) and exists in twin-chair conformation with equatorial orientations of the *para* fluoro phenyl groups on the heterocycle with the torsion angle of C5—C2—C1—C6 is 178.41 (6)°. The aryl groups are orientated at an angle of 28.67 (3)° to each other.

S2. Experimental

A mixture of cyclohexanone (0.05 mol) and *para* fluorobenzaldehyde (0.1 mol) was added to a warm solution of ammonium acetate (0.075 mol) in 50 ml of absolute ethanol. The mixture was very gently warmed on a hot plate till the yellow color formed during the mixing of the reactants and allowed to stir till the formation of the product. Thus, the formed azabicyclononane was separated by filtration and washed with a 1:5 v/v ethanol-ether mixture till the solid became colorless. Then, recrystallization of the compound from ethanol afforded colorless blocks of (I).

S3. Refinement

The nitrogen H atom was located in a difference Fourier map and refined isotropically. Other hydrogen atoms were fixed geometrically (C—H = 0.93-0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

**Figure 1**

The molecular structure of (I) with non-hydrogen atoms represented as 30% probability ellipsoids.

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Crystal data

$C_{20}H_{19}F_2NO$

$M_r = 327.36$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 7.6153$ (3) Å

$b = 21.1392$ (9) Å

$c = 10.0878$ (4) Å

$V = 1623.95$ (11) Å³

$Z = 4$

$F(000) = 688$

$D_x = 1.339$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4451 reflections

$\theta = 3.4$ – 28.0°

$\mu = 0.10$ mm⁻¹

$T = 298$ K

Block, colourless

$0.35 \times 0.32 \times 0.30$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2004)

$T_{\min} = 0.967$, $T_{\max} = 0.971$

11360 measured reflections

2064 independent reflections

1596 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 1.9^\circ$

$h = -8 \rightarrow 10$

$k = -28 \rightarrow 28$

$l = -13 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.141$

$S = 0.91$

2064 reflections

118 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
 $w = 1/[\sigma^2(F_o^2) + (0.0897P)^2 + 0.3733P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.22 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{Å}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.92315 (17)	0.30735 (5)	0.39201 (12)	0.0391 (3)
H1	0.8839	0.3055	0.2996	0.047*
C2	0.75726 (16)	0.30901 (5)	0.48153 (14)	0.0421 (3)
H2	0.6864	0.3460	0.4576	0.051*
C3	0.79266 (19)	0.31054 (6)	0.63140 (13)	0.0473 (3)
H3A	0.8701	0.3458	0.6508	0.057*
H3B	0.6827	0.3181	0.6773	0.057*
C4	0.8751 (3)	0.2500	0.68562 (18)	0.0492 (4)
H4A	0.9993	0.2500	0.6644	0.059*
H4B	0.8642	0.2500	0.7814	0.059*
C5	0.6527 (2)	0.2500	0.45348 (18)	0.0435 (4)
C6	1.03437 (17)	0.36590 (5)	0.40791 (12)	0.0400 (3)
C7	0.9916 (2)	0.41937 (6)	0.33533 (16)	0.0543 (4)
H7	0.8976	0.4179	0.2766	0.065*
C8	1.0856 (2)	0.47481 (7)	0.34848 (19)	0.0666 (5)
H8	1.0550	0.5107	0.3003	0.080*
C9	1.2238 (2)	0.47573 (7)	0.4334 (2)	0.0643 (5)
C10	1.2733 (2)	0.42414 (8)	0.50616 (19)	0.0641 (4)
H10	1.3688	0.4261	0.5634	0.077*
C11	1.1771 (2)	0.36882 (7)	0.49220 (15)	0.0519 (4)
H11	1.2091	0.3331	0.5404	0.062*
F1	1.31755 (17)	0.53023 (5)	0.44594 (17)	0.1030 (5)
H111	1.121 (3)	0.2500	0.369 (2)	0.047 (5)*
N1	1.0241 (2)	0.2500	0.41923 (15)	0.0384 (3)
O1	0.50237 (19)	0.2500	0.41536 (17)	0.0626 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0441 (7)	0.0372 (6)	0.0359 (6)	0.0013 (5)	-0.0009 (5)	0.0021 (4)
C2	0.0395 (6)	0.0368 (6)	0.0500 (7)	0.0045 (5)	-0.0010 (5)	0.0018 (5)

C3	0.0493 (7)	0.0465 (7)	0.0461 (7)	-0.0017 (6)	0.0077 (6)	-0.0072 (5)
C4	0.0529 (10)	0.0585 (10)	0.0361 (9)	0.000	0.0003 (8)	0.000
C5	0.0384 (9)	0.0483 (9)	0.0437 (9)	0.000	-0.0027 (7)	0.000
C6	0.0440 (7)	0.0352 (5)	0.0406 (6)	0.0016 (5)	0.0073 (5)	0.0021 (4)
C7	0.0567 (8)	0.0455 (7)	0.0605 (8)	0.0048 (6)	0.0044 (7)	0.0137 (6)
C8	0.0694 (10)	0.0390 (7)	0.0915 (12)	0.0051 (7)	0.0242 (10)	0.0157 (7)
C9	0.0586 (9)	0.0382 (7)	0.0961 (13)	-0.0091 (6)	0.0292 (9)	-0.0104 (7)
C10	0.0558 (9)	0.0579 (9)	0.0787 (11)	-0.0105 (7)	0.0011 (8)	-0.0110 (8)
C11	0.0529 (8)	0.0455 (7)	0.0572 (8)	-0.0038 (6)	-0.0029 (6)	0.0043 (6)
F1	0.0834 (8)	0.0474 (6)	0.1782 (14)	-0.0225 (5)	0.0323 (8)	-0.0170 (6)
N1	0.0377 (8)	0.0341 (7)	0.0434 (8)	0.000	0.0057 (6)	0.000
O1	0.0421 (8)	0.0693 (10)	0.0763 (11)	0.000	-0.0159 (7)	0.000

Geometric parameters (Å, °)

C1—N1	1.4613 (14)	C5—C2 ⁱ	1.5067 (15)
C1—C6	1.5083 (16)	C6—C11	1.381 (2)
C1—C2	1.5533 (18)	C6—C7	1.3856 (17)
C1—H1	0.9800	C7—C8	1.380 (2)
C2—C5	1.5067 (15)	C7—H7	0.9300
C2—C3	1.536 (2)	C8—C9	1.357 (3)
C2—H2	0.9800	C8—H8	0.9300
C3—C4	1.5266 (17)	C9—F1	1.3613 (17)
C3—H3A	0.9700	C9—C10	1.367 (3)
C3—H3B	0.9700	C10—C11	1.387 (2)
C4—C3 ⁱ	1.5266 (17)	C10—H10	0.9300
C4—H4A	0.9700	C11—H11	0.9300
C4—H4B	0.9700	N1—C1 ⁱ	1.4613 (14)
C5—O1	1.208 (2)	N1—H111	0.89 (2)
N1—C1—C6	111.44 (10)	O1—C5—C2	124.12 (7)
N1—C1—C2	109.70 (10)	O1—C5—C2 ⁱ	124.12 (7)
C6—C1—C2	112.11 (9)	C2—C5—C2 ⁱ	111.76 (14)
N1—C1—H1	107.8	C11—C6—C7	118.28 (12)
C6—C1—H1	107.8	C11—C6—C1	122.96 (11)
C2—C1—H1	107.8	C7—C6—C1	118.76 (12)
C5—C2—C3	107.16 (11)	C8—C7—C6	121.34 (15)
C5—C2—C1	107.58 (11)	C8—C7—H7	119.3
C3—C2—C1	115.47 (11)	C6—C7—H7	119.3
C5—C2—H2	108.8	C9—C8—C7	118.37 (14)
C3—C2—H2	108.8	C9—C8—H8	120.8
C1—C2—H2	108.8	C7—C8—H8	120.8
C4—C3—C2	114.02 (11)	C8—C9—F1	118.51 (16)
C4—C3—H3A	108.7	C8—C9—C10	122.75 (14)
C2—C3—H3A	108.7	F1—C9—C10	118.74 (18)
C4—C3—H3B	108.7	C9—C10—C11	118.19 (16)
C2—C3—H3B	108.7	C9—C10—H10	120.9
H3A—C3—H3B	107.6	C11—C10—H10	120.9

C3 ⁱ —C4—C3	113.91 (16)	C6—C11—C10	121.05 (14)
C3 ⁱ —C4—H4A	108.8	C6—C11—H11	119.5
C3—C4—H4A	108.8	C10—C11—H11	119.5
C3 ⁱ —C4—H4B	108.8	C1 ⁱ —N1—C1	112.11 (14)
C3—C4—H4B	108.8	C1 ⁱ —N1—H111	109.1 (7)
H4A—C4—H4B	107.7	C1—N1—H111	109.1 (7)
N1—C1—C2—C5	-58.02 (14)	C2—C1—C6—C7	-84.10 (15)
C6—C1—C2—C5	177.61 (10)	C11—C6—C7—C8	-1.5 (2)
N1—C1—C2—C3	61.57 (13)	C1—C6—C7—C8	178.21 (13)
C6—C1—C2—C3	-62.80 (13)	C6—C7—C8—C9	0.9 (2)
C5—C2—C3—C4	52.64 (16)	C7—C8—C9—F1	179.55 (15)
C1—C2—C3—C4	-67.17 (15)	C7—C8—C9—C10	0.0 (3)
C2—C3—C4—C3 ⁱ	-43.3 (2)	C8—C9—C10—C11	-0.2 (3)
C3—C2—C5—O1	113.4 (2)	F1—C9—C10—C11	-179.77 (15)
C1—C2—C5—O1	-121.84 (19)	C7—C6—C11—C10	1.3 (2)
C3—C2—C5—C2 ⁱ	-65.39 (17)	C1—C6—C11—C10	-178.44 (14)
C1—C2—C5—C2 ⁱ	59.35 (18)	C9—C10—C11—C6	-0.4 (2)
N1—C1—C6—C11	-27.77 (17)	C6—C1—N1—C1 ⁱ	-174.53 (8)
C2—C1—C6—C11	95.62 (15)	C2—C1—N1—C1 ⁱ	60.72 (16)
N1—C1—C6—C7	152.51 (13)		

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