Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Methyl-*N*-*p*-tolylpiperidine-1-carboxamide

Yu-Feng Li

Microscale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: liyufeng8111@163.com

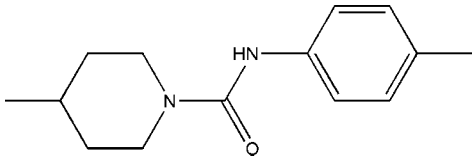
Received 26 March 2012; accepted 14 April 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.072; wR factor = 0.220; data-to-parameter ratio = 15.5.

In the title molecule, $C_{14}H_{20}N_2O$, the piperidine ring has a chair conformation and its N atom is close to planar (bond-angle sum = 357.5°). The dihedral angle between the amide group and the aromatic ring is 47.43 (19)°. In the crystal, molecules are linked into [100] $C(4)$ chains by N—H···O hydrogen bonds.

Related literature

For the medicinal properties of related compounds, see: Yang *et al.* (1997). For a related structure, see: Li (2011).



Experimental

Crystal data

$C_{14}H_{20}N_2O$ M_r = 232.32
Orthorhombic, *Pbca*
a = 9.6192 (19) Å
b = 11.127 (2) Å
c = 26.574 (5) Å
V = 2844.3 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
21306 measured reflections
2571 independent reflections
1219 reflections with $I > 2\sigma(I)$
R_{int} = 0.115

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.220
S = 1.03
2571 reflections
166 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.89 (3)	2.08 (3)	2.935 (3)	162 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6706).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS, Inc., Madison, Wisconsin, USA.
Li, Y.-F. (2011). *Acta Cryst.* E**67**, o1792.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yang, D., Soulier, J. L., Sicsic, S., Mathe-Allainmat, M., Bremont, B., Croci, T., Cardamone, R., Aureggi, G. & Langlois, M. (1997). *J. Med. Chem.* **40**, 608–621.

supporting information

Acta Cryst. (2012). E**68**, o1472 [doi:10.1107/S1600536812016248]

4-Methyl-*N*-*p*-tolylpiperidine-1-carboxamide

Yu-Feng Li

S1. Comment

Some compounds which are related to the title compound have been shown to have medicinal properties (Yang *et al.*, 1997). The structure of the title compound is shown in Fig. 1. The six-membered ring (C2—C6/N1) has a chair conformation. The bond lengths and angles can be compared to those within a related structure (Li, 2011). In the crystal, the molecules are linked into [100] chains by way of N—H···O hydrogen bonds.

S2. Experimental

A mixture of 4-methylpiperidine (0.08 mol), and *p*-tolylcarbamic chloride (0.08 mol) was stirred in refluxing ethanol (18 ml) for 4 h to afford the title compound (0.056 mol, yield 70%). Colourless blocks of the title compound were obtained by recrystallization from ethanol at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93–0.97 Å; N—H = 0.86 Å and with U_{iso}(H) = 1.2U_{eq}(C,*N*).

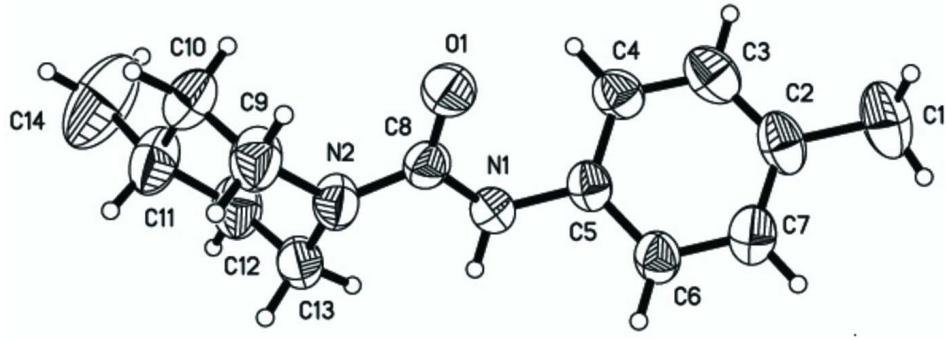


Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids.

4-Methyl-*N*-*p*-tolylpiperidine-1-carboxamide

Crystal data

$C_{14}H_{20}N_2O$
M_r = 232.32
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 9.6192 (19) Å
b = 11.127 (2) Å
c = 26.574 (5) Å
V = 2844.3 (9) Å^3
Z = 8
F(000) = 1008
D_x = 1.085 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1979 reflections
θ = 3.1–27.5°

μ = 0.07 mm^{-1}
T = 293 K

Block, colorless
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
21306 measured reflections
2571 independent reflections

1219 reflections with $I > 2\sigma(I)$
R_{int} = 0.115
θ_{max} = 25.3°, θ_{min} = 3.1°
h = −11→10
k = −13→13
l = −31→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.220
S = 1.03
2571 reflections
166 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
$w = 1/[\sigma^2(F_o^2) + (0.1032P)^2 + 0.1575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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}
N2	0.3590 (3)	0.2180 (2)	0.24983 (9)	0.0723 (7)
O1	0.1457 (2)	0.2820 (2)	0.27689 (8)	0.0937 (8)
N1	0.3387 (3)	0.3504 (2)	0.31682 (9)	0.0835 (8)
C8	0.3092 (3)	0.1515 (3)	0.20795 (11)	0.0692 (8)
C7	0.2741 (3)	0.2829 (3)	0.28129 (11)	0.0713 (8)
C13	0.3740 (3)	0.1655 (3)	0.16191 (12)	0.0774 (9)
H13A	0.4486	0.2180	0.1586	0.093*
C12	0.3270 (4)	0.1007 (3)	0.12054 (12)	0.0900 (10)
H12A	0.3714	0.1109	0.0897	0.108*
C9	0.2002 (3)	0.0699 (3)	0.21213 (12)	0.0828 (10)
H9A	0.1574	0.0577	0.2431	0.099*
C11	0.2162 (4)	0.0217 (3)	0.12365 (14)	0.0927 (11)
C5	0.4786 (4)	0.3297 (5)	0.33540 (14)	0.0885 (11)
C10	0.1556 (4)	0.0073 (3)	0.17043 (16)	0.0964 (11)

H10A	0.0822	−0.0464	0.1739	0.116*
C14	0.1648 (5)	−0.0461 (4)	0.07747 (15)	0.1356 (17)
H14A	0.2195	−0.0238	0.0488	0.203*
H14B	0.0691	−0.0264	0.0714	0.203*
H14C	0.1732	−0.1310	0.0832	0.203*
C4	0.2562 (4)	0.4264 (4)	0.35029 (14)	0.1071 (13)
H4A	0.1637	0.4367	0.3364	0.129*
H4B	0.2990	0.5051	0.3529	0.129*
C2	0.3901 (5)	0.3450 (4)	0.42415 (14)	0.1255 (15)
H2B	0.4366	0.4221	0.4299	0.151*
C6	0.4752 (4)	0.2737 (4)	0.38700 (14)	0.1118 (13)
H6A	0.4370	0.1933	0.3844	0.134*
H6B	0.5696	0.2667	0.3995	0.134*
C3	0.2461 (4)	0.3699 (4)	0.40226 (15)	0.1223 (15)
H3A	0.1945	0.2951	0.4000	0.147*
H3B	0.1957	0.4234	0.4245	0.147*
C1	0.3789 (9)	0.2780 (8)	0.47512 (19)	0.254 (4)
H1A	0.4703	0.2653	0.4886	0.381*
H1B	0.3341	0.2019	0.4701	0.381*
H1C	0.3254	0.3254	0.4983	0.381*
H2A	0.448 (3)	0.241 (3)	0.2491 (11)	0.088 (10)*
H5A	0.532 (3)	0.280 (3)	0.3091 (13)	0.101 (10)*
H5B	0.525 (4)	0.399 (4)	0.3365 (12)	0.115 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0617 (16)	0.0876 (18)	0.0677 (16)	−0.0074 (14)	−0.0034 (13)	−0.0075 (14)
O1	0.0612 (14)	0.136 (2)	0.0839 (16)	0.0009 (13)	−0.0030 (11)	−0.0044 (13)
N1	0.0641 (16)	0.114 (2)	0.0720 (17)	0.0103 (14)	−0.0053 (13)	−0.0237 (16)
C8	0.0735 (19)	0.0647 (19)	0.069 (2)	−0.0026 (16)	−0.0056 (15)	0.0013 (15)
C7	0.0629 (19)	0.085 (2)	0.0656 (19)	−0.0019 (17)	0.0031 (16)	0.0086 (17)
C13	0.093 (2)	0.0668 (19)	0.073 (2)	−0.0078 (17)	−0.0045 (17)	−0.0032 (16)
C12	0.124 (3)	0.075 (2)	0.071 (2)	0.006 (2)	−0.012 (2)	−0.0020 (18)
C9	0.086 (2)	0.075 (2)	0.087 (2)	−0.0111 (18)	−0.0064 (18)	0.0081 (18)
C11	0.113 (3)	0.067 (2)	0.099 (3)	0.004 (2)	−0.034 (2)	−0.0082 (19)
C5	0.071 (2)	0.117 (3)	0.078 (2)	−0.007 (2)	0.0001 (17)	−0.025 (2)
C10	0.100 (3)	0.068 (2)	0.121 (3)	−0.0100 (18)	−0.027 (2)	−0.002 (2)
C14	0.170 (4)	0.111 (3)	0.126 (3)	−0.005 (3)	−0.058 (3)	−0.031 (3)
C4	0.090 (2)	0.139 (3)	0.093 (3)	0.023 (2)	−0.002 (2)	−0.031 (2)
C2	0.142 (4)	0.166 (4)	0.069 (2)	0.028 (3)	0.000 (2)	−0.010 (2)
C6	0.113 (3)	0.130 (3)	0.092 (3)	0.020 (3)	−0.017 (2)	−0.014 (2)
C3	0.108 (3)	0.161 (4)	0.097 (3)	0.006 (3)	0.029 (2)	−0.031 (3)
C1	0.344 (11)	0.333 (10)	0.084 (4)	0.090 (8)	0.026 (5)	0.042 (5)

Geometric parameters (Å, º)

N2—C7	1.374 (4)	C5—H5B	0.89 (4)
N2—C8	1.420 (4)	C10—H10A	0.9300
N2—H2A	0.89 (3)	C14—H14A	0.9600
O1—C7	1.241 (3)	C14—H14B	0.9600
N1—C7	1.357 (4)	C14—H14C	0.9600
N1—C4	1.462 (4)	C4—C3	1.521 (5)
N1—C5	1.452 (4)	C4—H4A	0.9700
C8—C13	1.382 (4)	C4—H4B	0.9700
C8—C9	1.391 (4)	C2—C6	1.508 (5)
C13—C12	1.390 (4)	C2—C3	1.528 (6)
C13—H13A	0.9300	C2—C1	1.550 (6)
C12—C11	1.384 (5)	C2—H2B	0.9800
C12—H12A	0.9300	C6—H6A	0.9700
C9—C10	1.378 (4)	C6—H6B	0.9700
C9—H9A	0.9300	C3—H3A	0.9700
C11—C10	1.382 (5)	C3—H3B	0.9700
C11—C14	1.524 (5)	C1—H1A	0.9600
C5—C6	1.506 (5)	C1—H1B	0.9600
C5—H5A	1.03 (3)	C1—H1C	0.9600
C7—N2—C8	123.4 (3)	H14A—C14—H14B	109.5
C7—N2—H2A	116 (2)	C11—C14—H14C	109.5
C8—N2—H2A	117 (2)	H14A—C14—H14C	109.5
C7—N1—C4	119.7 (3)	H14B—C14—H14C	109.5
C7—N1—C5	125.0 (3)	N1—C4—C3	110.4 (3)
C4—N1—C5	112.8 (3)	N1—C4—H4A	109.6
C13—C8—C9	119.0 (3)	C3—C4—H4A	109.6
C13—C8—N2	118.9 (3)	N1—C4—H4B	109.6
C9—C8—N2	122.1 (3)	C3—C4—H4B	109.6
O1—C7—N1	121.7 (3)	H4A—C4—H4B	108.1
O1—C7—N2	122.0 (3)	C6—C2—C3	109.8 (3)
N1—C7—N2	116.2 (3)	C6—C2—C1	110.9 (4)
C8—C13—C12	119.7 (3)	C3—C2—C1	110.9 (4)
C8—C13—H13A	120.2	C6—C2—H2B	108.4
C12—C13—H13A	120.2	C3—C2—H2B	108.4
C13—C12—C11	122.2 (3)	C1—C2—H2B	108.4
C13—C12—H12A	118.9	C2—C6—C5	112.9 (3)
C11—C12—H12A	118.9	C2—C6—H6A	109.0
C10—C9—C8	120.0 (3)	C5—C6—H6A	109.0
C10—C9—H9A	120.0	C2—C6—H6B	109.0
C8—C9—H9A	120.0	C5—C6—H6B	109.0
C10—C11—C12	116.9 (3)	H6A—C6—H6B	107.8
C10—C11—C14	122.0 (4)	C4—C3—C2	111.3 (3)
C12—C11—C14	121.1 (4)	C4—C3—H3A	109.4
N1—C5—C6	110.8 (3)	C2—C3—H3A	109.4
N1—C5—H5A	108.7 (18)	C4—C3—H3B	109.4

C6—C5—H5A	113.7 (18)	C2—C3—H3B	109.4
N1—C5—H5B	110 (2)	H3A—C3—H3B	108.0
C6—C5—H5B	110 (2)	C2—C1—H1A	109.5
H5A—C5—H5B	104 (3)	C2—C1—H1B	109.5
C9—C10—C11	122.2 (3)	H1A—C1—H1B	109.5
C9—C10—H10A	118.9	C2—C1—H1C	109.5
C11—C10—H10A	118.9	H1A—C1—H1C	109.5
C11—C14—H14A	109.5	H1B—C1—H1C	109.5
C11—C14—H14B	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.89 (3)	2.08 (3)	2.935 (3)	162 (3)

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