Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(4-Chlorophenyl)-4-methylpiperazine-1-carboxamide

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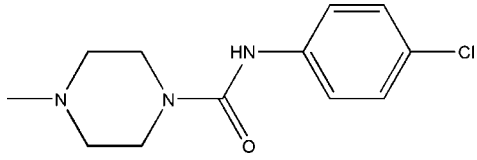
Received 6 August 2011; accepted 21 August 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.059; wR factor = 0.192; data-to-parameter ratio = 19.5.

In the title compound, $C_{12}H_{16}ClN_3O$, the piperazine ring has a chair conformation. Within this ring, the *N*-methyl nitrogen atom has a pyramidal geometry and the *N*-carboxamide nitrogen atom is almost planar (bond-angle sum = 359.8°). In the crystal, the molecules are linked by N—H···O hydrogen bonds into *C*(4) chains propagating in [010].

Related literature

For related structures, see: Arrieta *et al.* (20071); Li (2011*a*,*b*).



Experimental

Crystal data

$C_{12}H_{16}ClN_3O$ M_r = 253.73
Orthorhombic, *Pbcn*
a = 24.920 (5) Å
b = 9.5033 (19) Å
c = 11.064 (2) Å
V = 2620.3 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
23785 measured reflections
3006 independent reflections
2077 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.192
S = 1.11
3006 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1^{i}	0.86	2.26	3.001 (2)	144

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

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: HB6351).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o2453 [doi:10.1107/S1600536811034283]

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S1. Experimental

A mixture of 1-methylpiperazine (0.1 mol), and (4-chlorophenyl)carbamic chloride (0.1 mol) was stirred in refluxing ethanol (20 ml) for 4 h to afford the title compound (0.079 mol, yield 79%). Colourless blocks of the title compound were obtained by recrystallization from ethanol at room temperature.

S2. 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 = 0.86Å and with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C_{methyl}).

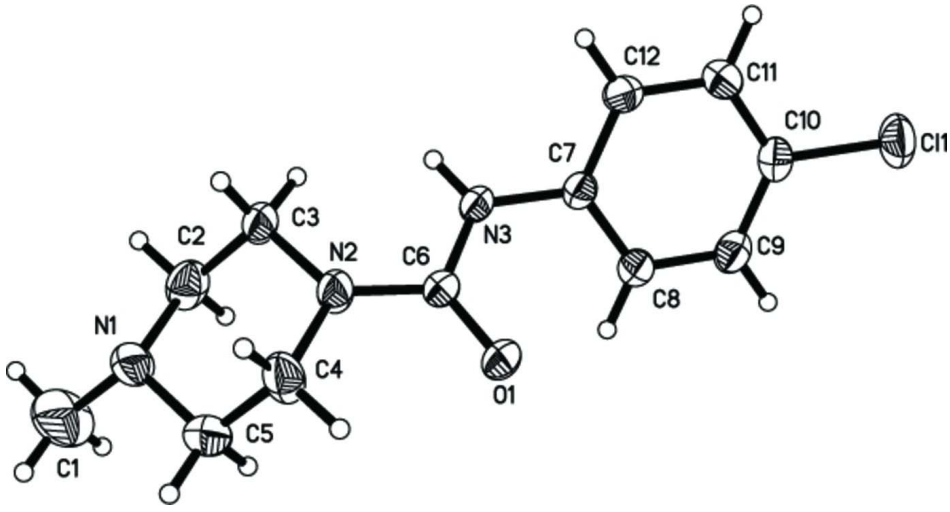

Figure 1

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

N-(4-Chlorophenyl)-4-methylpiperazine-1-carboxamide

Crystal data

$C_{12}H_{16}ClN_3O$
M_r = 253.73
Orthorhombic, *Pbcn*
a = 24.920 (5) Å
b = 9.5033 (19) Å
c = 11.064 (2) Å
V = 2620.3 (9) Å^3
Z = 8
F(000) = 1072
D_x = 1.286 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 2077 reflections
θ = 3.2–27.4°
μ = 0.28 mm^{-1}
T = 293 K
Block, colorless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
23785 measured reflections
3006 independent reflections
2077 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -31 \rightarrow 32$
$k = -12 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.192$
$S = 1.11$
3006 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.1054P)^2 + 0.5647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.07382 (3)	0.14060 (8)	0.66714 (8)	0.0750 (3)
O1	0.28704 (6)	0.11160 (14)	1.01352 (17)	0.0559 (5)
C6	0.28896 (7)	−0.0180 (2)	1.0143 (2)	0.0424 (5)
C7	0.21110 (7)	−0.0415 (2)	0.88098 (19)	0.0399 (5)
N3	0.25282 (6)	−0.09932 (17)	0.95154 (18)	0.0453 (5)
H3A	0.2558	−0.1894	0.9555	0.054*
C12	0.16012 (8)	−0.0996 (2)	0.8900 (2)	0.0479 (5)
H12A	0.1541	−0.1754	0.9415	0.058*
C11	0.11817 (8)	−0.0447 (3)	0.8224 (2)	0.0516 (6)
H11A	0.0840	−0.0836	0.8282	0.062*
C10	0.12738 (8)	0.0675 (2)	0.7469 (2)	0.0501 (5)
C9	0.17805 (9)	0.1239 (2)	0.7340 (2)	0.0522 (6)
H9A	0.1840	0.1981	0.6809	0.063*
C8	0.21988 (8)	0.0687 (2)	0.8010 (2)	0.0464 (5)
H8A	0.2542	0.1057	0.7925	0.056*
N2	0.32650 (7)	−0.08928 (19)	1.0782 (2)	0.0558 (6)
N1	0.43172 (7)	−0.1934 (2)	1.1165 (2)	0.0605 (6)

C3	0.33749 (9)	−0.2391 (2)	1.0715 (3)	0.0593 (7)
H3B	0.3139	−0.2824	1.0126	0.071*
H3C	0.3306	−0.2822	1.1495	0.071*
C4	0.36406 (10)	−0.0151 (3)	1.1554 (3)	0.0681 (8)
H4A	0.3592	−0.0450	1.2385	0.082*
H4B	0.3572	0.0852	1.1512	0.082*
C5	0.42085 (10)	−0.0446 (3)	1.1162 (3)	0.0719 (8)
H5A	0.4265	−0.0074	1.0356	0.086*
H5B	0.4456	0.0025	1.1705	0.086*
C2	0.39479 (11)	−0.2641 (3)	1.0361 (3)	0.0647 (7)
H2A	0.4021	−0.3643	1.0372	0.078*
H2B	0.4004	−0.2305	0.9542	0.078*
C1	0.48742 (12)	−0.2234 (5)	1.0819 (4)	0.1134 (15)
H1A	0.5114	−0.1767	1.1367	0.170*
H1B	0.4937	−0.1902	1.0012	0.170*
H1C	0.4936	−0.3231	1.0851	0.170*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0629 (4)	0.0773 (5)	0.0846 (6)	0.0211 (3)	−0.0201 (3)	0.0074 (4)
O1	0.0555 (9)	0.0286 (7)	0.0836 (13)	0.0032 (6)	−0.0119 (8)	−0.0023 (7)
C6	0.0424 (10)	0.0319 (9)	0.0529 (13)	0.0009 (7)	−0.0027 (8)	0.0008 (8)
C7	0.0433 (10)	0.0341 (9)	0.0422 (11)	0.0036 (8)	−0.0015 (8)	0.0007 (8)
N3	0.0477 (9)	0.0295 (8)	0.0587 (12)	0.0000 (7)	−0.0117 (8)	0.0055 (7)
C12	0.0493 (11)	0.0431 (11)	0.0514 (13)	−0.0051 (9)	−0.0025 (9)	0.0047 (9)
C11	0.0419 (10)	0.0536 (13)	0.0591 (15)	−0.0025 (9)	−0.0016 (9)	−0.0004 (11)
C10	0.0499 (12)	0.0490 (12)	0.0514 (13)	0.0120 (9)	−0.0052 (9)	−0.0021 (10)
C9	0.0592 (13)	0.0455 (11)	0.0520 (14)	0.0077 (9)	0.0019 (10)	0.0093 (10)
C8	0.0433 (10)	0.0436 (11)	0.0523 (13)	0.0014 (8)	0.0018 (9)	0.0060 (9)
N2	0.0542 (11)	0.0345 (9)	0.0787 (15)	0.0054 (8)	−0.0240 (10)	−0.0057 (9)
N1	0.0428 (10)	0.0548 (12)	0.0838 (16)	0.0033 (8)	−0.0014 (9)	−0.0012 (11)
C3	0.0574 (13)	0.0342 (11)	0.0862 (19)	0.0016 (9)	−0.0252 (12)	0.0045 (11)
C4	0.0611 (13)	0.0508 (14)	0.092 (2)	0.0102 (11)	−0.0304 (13)	−0.0211 (14)
C5	0.0595 (14)	0.0533 (14)	0.103 (2)	−0.0107 (11)	−0.0139 (14)	0.0008 (15)
C2	0.0767 (16)	0.0486 (13)	0.0689 (18)	0.0123 (12)	−0.0063 (13)	−0.0065 (12)
C1	0.0538 (18)	0.113 (3)	0.173 (4)	0.0106 (17)	0.023 (2)	−0.011 (3)

Geometric parameters (Å, º)

Cl1—C10	1.745 (2)	N2—C3	1.452 (3)
O1—C6	1.232 (2)	N1—C5	1.440 (3)
C6—N2	1.355 (3)	N1—C2	1.446 (3)
C6—N3	1.375 (3)	N1—C1	1.468 (3)
C7—C8	1.388 (3)	C3—C2	1.500 (4)
C7—C12	1.389 (3)	C3—H3B	0.9700
C7—N3	1.411 (2)	C3—H3C	0.9700
N3—H3A	0.8600	C4—C5	1.506 (4)

C12—C11	1.388 (3)	C4—H4A	0.9700
C12—H12A	0.9300	C4—H4B	0.9700
C11—C10	1.374 (3)	C5—H5A	0.9700
C11—H11A	0.9300	C5—H5B	0.9700
C10—C9	1.379 (3)	C2—H2A	0.9700
C9—C8	1.383 (3)	C2—H2B	0.9700
C9—H9A	0.9300	C1—H1A	0.9600
C8—H8A	0.9300	C1—H1B	0.9600
N2—C4	1.450 (3)	C1—H1C	0.9600

O1—C6—N2	122.02 (19)	N2—C3—C2	110.4 (2)
O1—C6—N3	122.22 (18)	N2—C3—H3B	109.6
N2—C6—N3	115.75 (17)	C2—C3—H3B	109.6
C8—C7—C12	119.35 (19)	N2—C3—H3C	109.6
C8—C7—N3	122.00 (17)	C2—C3—H3C	109.6
C12—C7—N3	118.63 (18)	H3B—C3—H3C	108.1
C6—N3—C7	122.87 (16)	N2—C4—C5	110.3 (2)
C6—N3—H3A	118.6	N2—C4—H4A	109.6
C7—N3—H3A	118.6	C5—C4—H4A	109.6
C11—C12—C7	120.0 (2)	N2—C4—H4B	109.6
C11—C12—H12A	120.0	C5—C4—H4B	109.6
C7—C12—H12A	120.0	H4A—C4—H4B	108.1
C10—C11—C12	119.6 (2)	N1—C5—C4	111.0 (2)
C10—C11—H11A	120.2	N1—C5—H5A	109.4
C12—C11—H11A	120.2	C4—C5—H5A	109.4
C11—C10—C9	121.2 (2)	N1—C5—H5B	109.4
C11—C10—Cl1	119.27 (17)	C4—C5—H5B	109.4
C9—C10—Cl1	119.57 (18)	H5A—C5—H5B	108.0
C10—C9—C8	119.2 (2)	N1—C2—C3	111.8 (2)
C10—C9—H9A	120.4	N1—C2—H2A	109.3
C8—C9—H9A	120.4	C3—C2—H2A	109.3
C9—C8—C7	120.6 (2)	N1—C2—H2B	109.3
C9—C8—H8A	119.7	C3—C2—H2B	109.3
C7—C8—H8A	119.7	H2A—C2—H2B	107.9
C6—N2—C4	120.68 (18)	N1—C1—H1A	109.5
C6—N2—C3	126.47 (18)	N1—C1—H1B	109.5
C4—N2—C3	112.64 (17)	H1A—C1—H1B	109.5
C5—N1—C2	109.6 (2)	N1—C1—H1C	109.5
C5—N1—C1	111.6 (2)	H1A—C1—H1C	109.5
C2—N1—C1	110.5 (3)	H1B—C1—H1C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1^{i}	0.86	2.26	3.001 (2)	144

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