4-Chloro-N-cyclohexylbenzamide

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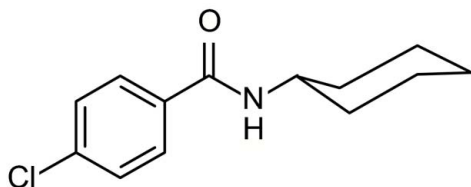Correspondence e-mail: aamersaeed@yahoo.com

In the title compound, $C_{13}H_{16}ClNO$, the cyclohexyl ring adopts a chair conformation, with puckering parameters $Q = 0.576$ (3) Å, $\theta = 0.1$ (3) and $\varphi = 8$ (15)°. In the crystal structure, intermolecular N—H···O hydrogen bonds link molecules into one-dimensional chains propagating in [010].

Related literature

For applications of N-substituted benzamides, see: Beccalli *et al.* (2005); Calderone *et al.* (2006); Vega-Noverola *et al.* (1989); Zhichkin *et al.* (2007); Lindgren *et al.* (2001); Olsson *et al.* (2002). For related crystal structures, see: Jones & Kuś (2004); Saeed *et al.* (2008). For puckering parameters, see: Cremer & Pople (1975). For a description of the Cambridge Structural Database, see: Allen (2002).

Experimental

Crystal data

$C_{13}H_{16}ClNO$	$c = 16.818$ (16) Å
$M_r = 237.72$	$\beta = 96.13$ (6)°
Monoclinic, $P2_1/c$	$V = 1244$ (2) Å^3
$a = 14.755$ (14) Å	$Z = 4$
$b = 5.043$ (7) Å	Mo $K\alpha$ radiation

$\mu = 0.29$ mm^{-1}	$0.12 \times 0.08 \times 0.06$ mm
$T = 173$ K	

Data collection

Bruker APEXII CCD diffractometer	3651 measured reflections
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	2388 independent reflections
$T_{min} = 0.967$, $T_{max} = 0.983$	1497 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$	145 parameters
$wR(F^2) = 0.151$	H-atom parameters constrained
$S = 1.06$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
2388 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.88	2.06	2.901 (5)	160

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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supporting information

4-Chloro-*N*-cyclohexylbenzamide

Aamer Saeed, Rasheed Ahmad Khera, Muhammad Latif and Masood Parvez

S1. Comment

N-Substituted benzamides, *e.g.*, declopramideare, are well known anticancer compounds and the mechanism of benzamide-induced apoptosis has been studied, (Olsson *et al.*, 2002). N-substituted benzamides inhibit the activity of nuclear factor-B and nuclear factor of activated T cells (Lindgren *et al.*, 2001). Various N-substituted benzamides exhibit potent antiemetic activity (Vega-Noverola *et al.*, 1989), while heterocyclic benzanilide are potassium channel activators (Calderone *et al.*, 2006). *N*-Alkylated 2-nitrobenzamides are intermediates in the synthesis of dibenzo[b,e][1,4]diazepines (Zhichkin *et al.*, 2007) and *N*-Acyl-2-nitrobenzamides are precursors of 2,3-disubstitued 3*H*-quinazoline-4-ones (Beccalli *et al.*, 2005). As part of our work on the structure of benzanilides and related compounds, in this paper, we report the crystal structure of the title compound, (I).

The molecular structure of (I) is presented in Fig. 1. The molecular dimensions in (I) are normal (CSD version 5.30; Allen, 2002). The six-membered ring adopts a chair conformation with puckering parameters: $Q = 0.576$ (3) Å, $\theta = 0.1$ (3)° and $\varphi = 8(15)$° (Cremer & Pople, 1975). The structure is stabilized by hydrogen bonding (N1–H1···O1) forming chains of molecules along the *b*-axis (details are in Table 1). The crystal structures of closely related compounds have been reported (Saeed *et al.*, 2008; Jones & Kuś, 2004).

S2. Experimental

4-Chlorobenzoyl chloride (5.4 mmol) in CHCl$_3$ was treated with cyclohexylamine (21.6 mmol) under a nitrogen atmosphere at reflux for 3 h. Upon cooling, the reaction mixture was diluted with CHCl$_3$ and washed consecutively with aq 1 *M* HCl and saturated aq NaHCO$_3$. The organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Crystallization of the residue in CHCl$_3$ afforded the title compound (87%) as colorless needles: Anal. calcd. for C$_{13}$H$_{16}$ClNO,: C, 65.68; H, 6.78; N, 5.89%; found: C, 65.61; H, 6.80; N, 5.91%.

S3. Refinement

All the H-atoms were visible in the difference Fourier maps, they were included in the refinements at geometrically idealized positions with N—H = 0.88 Å and C—H distances = 0.95 - 0.99 Å, and U_{iso} = 1.2 times U_{eq} of the atoms to which they were bonded. The final difference map was free of chemically significant features.

Figure 1
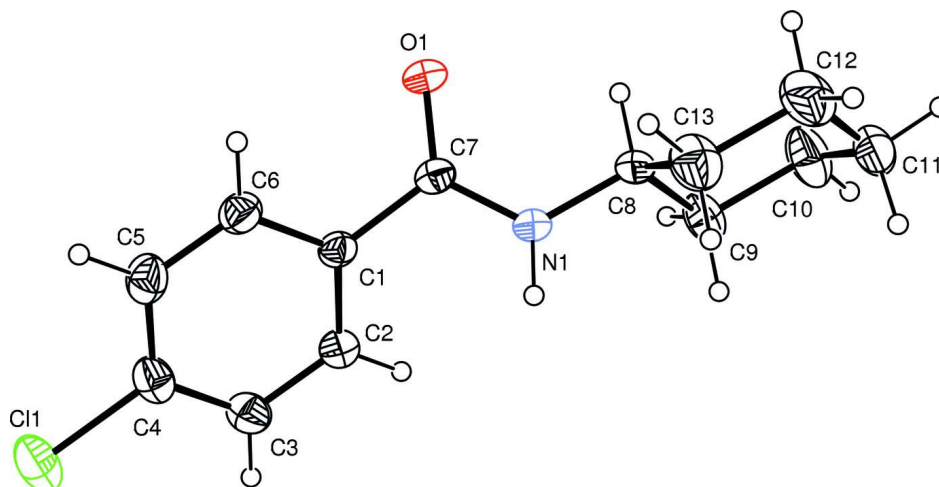
ORTEP-3 (Farrugia, 1997) drawing of (I) with displacement ellipsoids plotted at 30% probability level.

4-Chloro-*N*-cyclohexylbenzamide

Crystal data

$C_{13}H_{16}ClNO$	$F(000) = 504$
$M_r = 237.72$	$D_x = 1.269$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3651 reflections
$a = 14.755$ (14) Å	$\theta = 3.9–26.0°$
$b = 5.043$ (7) Å	$\mu = 0.29$ mm^{-1}
$c = 16.818$ (16) Å	$T = 173$ K
$\beta = 96.13$ (6)°	Needle, colorless
$V = 1244$ (2) Å^3	$0.12 \times 0.08 \times 0.06$ mm
$Z = 4$	

Data collection

Bruker APEXII CCD diffractometer	3651 measured reflections
Radiation source: fine-focus sealed tube	2388 independent reflections
Graphite monochromator	1497 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.034$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	$\theta_{max} = 26.0°$, $\theta_{min} = 3.9°$
	$h = -18 \rightarrow 17$
	$k = -6 \rightarrow 4$
$T_{min} = 0.967$, $T_{max} = 0.983$	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.051$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.151$	
$S = 1.06$	H-atom parameters constrained
2388 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.3764P]$ where $P = (F_o^2 + 2F_c^2)/3$
145 parameters	
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.16$ e Å^{-3}
	$\Delta\rho_{min} = -0.21$ 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}
Cl1	0.77571 (5)	0.29595 (19)	0.35603 (5)	0.0739 (3)
O1	0.35835 (12)	0.8003 (3)	0.37556 (11)	0.0489 (5)
N1	0.32675 (14)	0.3654 (4)	0.38965 (14)	0.0491 (6)
H1	0.3492	0.2036	0.3922	0.059*
C1	0.48155 (15)	0.4944 (5)	0.37540 (13)	0.0369 (6)
C2	0.52269 (17)	0.2853 (5)	0.41805 (15)	0.0462 (6)
H2	0.4884	0.1813	0.4512	0.055*
C3	0.61349 (18)	0.2254 (6)	0.41296 (16)	0.0529 (7)
H3	0.6419	0.0831	0.4431	0.063*
C4	0.66205 (17)	0.3746 (6)	0.36373 (15)	0.0489 (7)
C5	0.62262 (18)	0.5866 (6)	0.32108 (16)	0.0539 (7)
H5	0.6569	0.6895	0.2876	0.065*
C6	0.53255 (18)	0.6464 (5)	0.32789 (15)	0.0474 (6)
H6	0.5051	0.7938	0.2996	0.057*
C7	0.38370 (16)	0.5666 (5)	0.38013 (13)	0.0388 (6)
C8	0.22970 (16)	0.3992 (5)	0.39603 (16)	0.0484 (7)
H8	0.2181	0.5912	0.4058	0.058*
C9	0.20142 (17)	0.2430 (7)	0.46572 (16)	0.0564 (8)
H9A	0.2371	0.3025	0.5157	0.068*
H9B	0.2142	0.0524	0.4582	0.068*
C10	0.0997 (2)	0.2819 (9)	0.4724 (2)	0.0789 (11)
H10A	0.0816	0.1731	0.5171	0.095*
H10B	0.0881	0.4702	0.4845	0.095*
C11	0.04290 (19)	0.2051 (7)	0.3968 (2)	0.0735 (10)
H11A	−0.0221	0.2421	0.4022	0.088*
H11B	0.0494	0.0126	0.3875	0.088*
C12	0.0718 (2)	0.3567 (8)	0.3267 (2)	0.0819 (11)
H12A	0.0588	0.5476	0.3333	0.098*
H12B	0.0360	0.2949	0.2770	0.098*
C13	0.1742 (2)	0.3193 (7)	0.31917 (18)	0.0699 (9)
H13A	0.1864	0.1313	0.3072	0.084*
H13B	0.1920	0.4289	0.2746	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0443 (4)	0.0948 (7)	0.0840 (6)	0.0035 (4)	0.0128 (4)	−0.0220 (5)
O1	0.0535 (11)	0.0273 (9)	0.0662 (12)	0.0040 (8)	0.0080 (9)	0.0016 (8)
N1	0.0406 (11)	0.0278 (12)	0.0806 (15)	0.0040 (9)	0.0135 (11)	0.0015 (10)
C1	0.0413 (12)	0.0304 (12)	0.0393 (12)	−0.0013 (10)	0.0055 (10)	−0.0056 (10)
C2	0.0445 (13)	0.0408 (14)	0.0535 (15)	0.0020 (12)	0.0069 (11)	0.0053 (12)
C3	0.0475 (15)	0.0517 (17)	0.0589 (16)	0.0078 (13)	0.0025 (12)	0.0025 (14)
C4	0.0410 (13)	0.0589 (18)	0.0472 (14)	−0.0017 (13)	0.0063 (11)	−0.0165 (13)
C5	0.0550 (16)	0.0583 (18)	0.0510 (15)	−0.0079 (14)	0.0183 (13)	−0.0018 (14)
C6	0.0527 (15)	0.0415 (16)	0.0486 (14)	−0.0019 (12)	0.0086 (12)	0.0051 (12)
C7	0.0462 (14)	0.0305 (14)	0.0399 (13)	0.0006 (11)	0.0058 (10)	−0.0009 (10)
C8	0.0396 (13)	0.0292 (13)	0.0768 (18)	0.0047 (11)	0.0074 (13)	−0.0048 (13)
C9	0.0407 (14)	0.078 (2)	0.0518 (15)	0.0046 (14)	0.0100 (12)	−0.0119 (14)
C10	0.0473 (16)	0.113 (3)	0.080 (2)	0.0028 (18)	0.0212 (15)	−0.016 (2)
C11	0.0400 (15)	0.067 (2)	0.113 (3)	−0.0003 (14)	0.0070 (17)	−0.018 (2)
C12	0.0597 (19)	0.088 (3)	0.091 (2)	0.0080 (18)	−0.0240 (17)	−0.005 (2)
C13	0.0609 (18)	0.088 (2)	0.0589 (17)	0.0027 (17)	−0.0039 (14)	0.0122 (17)

Geometric parameters (Å, º)

Cl1—C4	1.742 (3)	C8—C13	1.510 (4)
O1—C7	1.236 (3)	C8—H8	1.0000
N1—C7	1.338 (3)	C9—C10	1.530 (4)
N1—C8	1.457 (3)	C9—H9A	0.9900
N1—H1	0.8800	C9—H9B	0.9900
C1—C2	1.379 (4)	C10—C11	1.496 (5)
C1—C6	1.386 (3)	C10—H10A	0.9900
C1—C7	1.499 (3)	C10—H10B	0.9900
C2—C3	1.385 (4)	C11—C12	1.505 (5)
C2—H2	0.9500	C11—H11A	0.9900
C3—C4	1.375 (4)	C11—H11B	0.9900
C3—H3	0.9500	C12—C13	1.541 (4)
C4—C5	1.381 (4)	C12—H12A	0.9900
C5—C6	1.379 (4)	C12—H12B	0.9900
C5—H5	0.9500	C13—H13A	0.9900
C6—H6	0.9500	C13—H13B	0.9900
C8—C9	1.508 (4)		
C7—N1—C8	123.7 (2)	C8—C9—C10	110.2 (2)
C7—N1—H1	118.1	C8—C9—H9A	109.6
C8—N1—H1	118.1	C10—C9—H9A	109.6
C2—C1—C6	119.1 (2)	C8—C9—H9B	109.6
C2—C1—C7	122.1 (2)	C10—C9—H9B	109.6
C6—C1—C7	118.8 (2)	H9A—C9—H9B	108.1
C1—C2—C3	120.6 (2)	C11—C10—C9	111.7 (3)
C1—C2—H2	119.7	C11—C10—H10A	109.3

C3—C2—H2	119.7	C9—C10—H10A	109.3
C4—C3—C2	119.2 (3)	C11—C10—H10B	109.3
C4—C3—H3	120.4	C9—C10—H10B	109.3
C2—C3—H3	120.4	H10A—C10—H10B	107.9
C3—C4—C5	121.2 (3)	C10—C11—C12	110.8 (3)
C3—C4—Cl1	119.2 (2)	C10—C11—H11A	109.5
C5—C4—Cl1	119.6 (2)	C12—C11—H11A	109.5
C6—C5—C4	118.8 (2)	C10—C11—H11B	109.5
C6—C5—H5	120.6	C12—C11—H11B	109.5
C4—C5—H5	120.6	H11A—C11—H11B	108.1
C5—C6—C1	121.0 (3)	C11—C12—C13	111.3 (3)
C5—C6—H6	119.5	C11—C12—H12A	109.4
C1—C6—H6	119.5	C13—C12—H12A	109.4
O1—C7—N1	122.7 (2)	C11—C12—H12B	109.4
O1—C7—C1	121.0 (2)	C13—C12—H12B	109.4
N1—C7—C1	116.3 (2)	H12A—C12—H12B	108.0
N1—C8—C9	110.6 (2)	C8—C13—C12	110.1 (3)
N1—C8—C13	110.7 (2)	C8—C13—H13A	109.6
C9—C8—C13	110.9 (2)	C12—C13—H13A	109.6
N1—C8—H8	108.2	C8—C13—H13B	109.6
C9—C8—H8	108.2	C12—C13—H13B	109.6
C13—C8—H8	108.2	H13A—C13—H13B	108.1
C6—C1—C2—C3	−0.7 (4)	C6—C1—C7—O1	−32.8 (3)
C7—C1—C2—C3	−179.3 (2)	C2—C1—C7—N1	−34.0 (3)
C1—C2—C3—C4	−1.0 (4)	C6—C1—C7—N1	147.4 (2)
C2—C3—C4—C5	1.7 (4)	C7—N1—C8—C9	−132.2 (3)
C2—C3—C4—Cl1	−178.9 (2)	C7—N1—C8—C13	104.5 (3)
C3—C4—C5—C6	−0.6 (4)	N1—C8—C9—C10	179.5 (2)
Cl1—C4—C5—C6	−180.0 (2)	C13—C8—C9—C10	−57.3 (3)
C4—C5—C6—C1	−1.2 (4)	C8—C9—C10—C11	56.9 (4)
C2—C1—C6—C5	1.8 (4)	C9—C10—C11—C12	−56.0 (4)
C7—C1—C6—C5	−179.5 (2)	C10—C11—C12—C13	55.5 (4)
C8—N1—C7—O1	−0.3 (4)	N1—C8—C13—C12	−179.8 (3)
C8—N1—C7—C1	179.5 (2)	C9—C8—C13—C12	57.0 (3)
C2—C1—C7—O1	145.9 (2)	C11—C12—C13—C8	−56.1 (4)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.88	2.06	2.901 (5)	160

Symmetry code: (i) x, y−1, z.