

N-Butyl-4-chlorobenzamide

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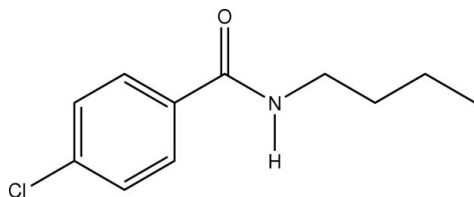
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Key indicators: single-crystal X-ray study; $T = 81$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.033; wR factor = 0.090; data-to-parameter ratio = 26.1.

In the title benzamide derivative, $\text{C}_{11}\text{H}_{14}\text{ClNO}$, the chlorobenzene and butylamine groups are each planar, with mean deviations from the planes of 0.013 and 0.030 Å, respectively, and a dihedral angle of 2.54 (9)° between the two planes. In the crystal structure, $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules in rows along a . Short intermolecular $\text{Cl}\cdots\text{Cl}$ interactions [3.4225 (5) Å] link these rows into sheets in the ac plane. Additional weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions generate a three-dimensional network.

Related literature

For details of the biological activity of benzanilides, see: Olsson *et al.*, (2002); Lindgren *et al.* (2001); Calderone *et al.* (2006). For the use of benzamides in organic synthesis, see: Reinaud *et al.* (1991); Zhichkin *et al.* (2007); Beccalli *et al.* (2005); For the fluorescence properties of benzanilides, see: Lewis & Long (1998). For related structures see: Saeed *et al.* (2008); Hempel *et al.* (2005). For reference structural data, see: Allen *et al.* (1987). For related literature, see: Vega-Noverola *et al.* (1989); Yoo *et al.* (2005).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{14}\text{ClNO}$
 $M_r = 211.68$
 Triclinic, $P\bar{1}$
 $a = 5.1702$ (4) Å
 $b = 7.8979$ (5) Å

$c = 13.2978$ (9) Å
 $\alpha = 89.275$ (3)°
 $\beta = 84.863$ (4)°
 $\gamma = 77.165$ (4)°
 $V = 527.29$ (6) Å³

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.33$ mm⁻¹

$T = 81$ (2) K
 $0.42 \times 0.30 \times 0.08$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2006)
 $T_{\min} = 0.820$, $T_{\max} = 0.974$
 6632 measured reflections
 3445 independent reflections
 3050 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.090$
 $S = 1.04$
 3445 reflections
 132 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.43$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{HN1}\cdots\text{O1}^{\text{i}}$	0.831 (15)	2.203 (15)	3.0164 (10)	166.3 (13)
$\text{C3}-\text{H3}\cdots\text{O1}^{\text{ii}}$	0.95	2.66	3.3146 (11)	127
$\text{C8}-\text{H8A}\cdots\text{Cg1}^{\text{iii}}$	0.99	2.84	3.697 (16)	145

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x, -y + 2, -z$; (iii) $-x + 1, -y, -z$. Cg1 is the centroid of the C2–C7 benzene ring.

Data collection: APEX2 (Bruker, 2006); cell refinement: APEX2 and SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and TITAN2000 (Hunter & Simpson, 1999); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97, enCIFer (Allen *et al.*, 2004), PLATON (Spek, 2003) and publCIF (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2272).

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supplementary materials

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Comment

The benzanilide core is present in compounds with such a wide range of biological activities that it has been called a privileged structure. N-substituted benzamides are well known anticancer compounds and the mechanism of action for N-substituted benzamide-induced apoptosis has been studied, using declopramide as a lead compound (Olsson *et al.*, 2002). N-substituted benzamides inhibit the activity of nuclear factor- κ B and nuclear factor of activated T cells activity while inducing activator protein 1 activity in T lymphocytes (Lindgren *et al.*, 2001). Various N-substituted benzamides exhibit potent antiemetic activity (Vega-noverola *et al.*, 1989), while heterocyclic analogs of benzanilide derivatives are potassium channel activators (Calderone *et al.*, 2006). *o*-Aryloxylation of N-substituted benzamides induced by the copper(II)/trimethylamine N-oxide system has been studied (Reinaud *et al.*, 1991). 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-disubstituted 3H-quinazoline-4-ones (Beccalli *et al.*, 2005). A one-pot conversion of 2-nitro-*n*-arylbenzamides to 2,3-dihydro-1H-quinazoline-4-ones has also been reported (Yoo *et al.*, 2005). The anomalous dual fluorescence of benzanilides has been assigned to the two lowest benzanilide singlet states (Lewis & Long, 1998)

As part of our work on the structure of benzanilides and related compounds, we report here the structure of the title benzamide derivative, I, Fig. 1. The C1 \cdots C7/Cl system is planar with a maximum deviation of 0.0161 (7) Å from the least squares plane. The carbonyl oxygen atom O1 is displaced by 0.6102 (10) Å from this plane. The butylamine N1/C8 \cdots C11 fragment is also planar, maximum deviation 0.0365 (7) Å for C9. The dihedral angle between these two planes is 2.54 (9)°. Bond distances within the molecule are normal (Allen *et al.*, 1987) and similar to those found in the structures of related 4-chlorobenzamide derivatives (Saeed *et al.*, 2008, Hempel *et al.*, 2005).

In the crystal structure N1—HN1 \cdots O1 hydrogen bonds, Table 1, link molecules into rows along *a*. C11 \cdots C11 interactions at 3.4225 (5) Å bridge these rows to form sheets in the *ac* plane, Fig. 2. The sheets are interconnected by weak C3—H3 \cdots O1 hydrogen bonds and C8—H8 \cdots π interactions involving the C2 \cdots C7 benzene ring to generate a three dimensional network, Fig. 3.

Experimental

2-Fluorobenzoyl chloride (1 mmol) in CHCl₃ was treated with cyclohexyl amine (3.5 mmol) under a nitrogen atmosphere at reflux for 5 h. Upon cooling, the reaction mixture was diluted with CHCl₃ and washed consecutively with 1 M aq HCl and saturated aq NaHCO₃. The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. Crystallization of the residue in ethanol afforded the title compound (79 %) as white needles: Anal. calcd. for C₁₁H₁₄ClNO: C 62.41, H 6.67, N 6.62%; found: C 62.34, H 7.16, N 6.57%.

Refinement

The H atom bound to N1 was located in a difference electron density map and refined freely with an isotropic displacement parameter. All other H-atoms were refined using a riding model with $d(\text{C—H}) = 0.95 \text{ \AA}$, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for aromatic, 0.99 \AA , $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for CH_2 , and 0.98 \AA , $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$ for CH_3 H atoms.

Figures

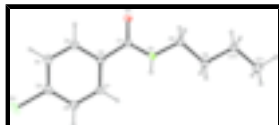


Fig. 1. The structure of I showing the atom numbering with displacement ellipsoids drawn at the 50% probability level.

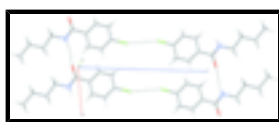


Fig. 2. Sheets of molecules of I formed in the *ac* plane by $\text{N—H}\cdots\text{O}$ hydrogen bonds and $\text{Cl}\cdots\text{Cl}$ interactions.

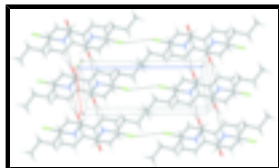


Fig. 3. Crystal packing of I viewed down the *b* axis.

N-Butyl-4-chlorobenzamide

Crystal data

$\text{C}_{11}\text{H}_{14}\text{ClNO}$

$M_r = 211.68$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.1702(4) \text{ \AA}$

$b = 7.8979(5) \text{ \AA}$

$c = 13.2978(9) \text{ \AA}$

$\alpha = 89.275(3)^\circ$

$\beta = 84.863(4)^\circ$

$\gamma = 77.165(4)^\circ$

$V = 527.29(6) \text{ \AA}^3$

$Z = 2$

$F_{000} = 224$

$D_x = 1.333 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3494 reflections

$\theta = 5.3\text{--}66.2^\circ$

$\mu = 0.33 \text{ mm}^{-1}$

$T = 81(2) \text{ K}$

Irregular fragment, colourless

$0.42 \times 0.30 \times 0.08 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 81(2) \text{ K}$

3445 independent reflections

3050 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\text{max}} = 33.1^\circ$

ω scans	$\theta_{\min} = 3.1^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2006)	$h = -7 \rightarrow 6$
$T_{\min} = 0.820$, $T_{\max} = 0.974$	$k = -11 \rightarrow 11$
6632 measured reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.090$	$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 0.125P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
3445 reflections	$(\Delta/\sigma)_{\max} = 0.002$
132 parameters	$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.51637 (16)	0.82511 (10)	-0.10915 (6)	0.01587 (15)
HN1	0.662 (3)	0.8260 (18)	-0.0876 (11)	0.026 (3)*
C1	0.31476 (17)	0.79695 (10)	-0.04475 (6)	0.01281 (15)
O1	0.08420 (13)	0.81551 (8)	-0.06861 (5)	0.01616 (14)
C2	0.38267 (17)	0.73835 (10)	0.05917 (6)	0.01257 (15)
C3	0.18549 (19)	0.78474 (11)	0.13885 (7)	0.01611 (17)
H3	0.0169	0.8545	0.1262	0.019*
C4	0.2331 (2)	0.73001 (12)	0.23650 (7)	0.01847 (18)
H4	0.0995	0.7631	0.2908	0.022*
C5	0.4800 (2)	0.62579 (11)	0.25329 (7)	0.01667 (17)
Cl1	0.54360 (5)	0.55693 (3)	0.375283 (17)	0.02530 (8)
C6	0.67765 (19)	0.57568 (12)	0.17528 (7)	0.01740 (17)

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H6	0.8439	0.5027	0.1879	0.021*
C7	0.62886 (18)	0.63406 (11)	0.07801 (7)	0.01532 (16)
H7	0.7642	0.6026	0.0241	0.018*
C8	0.47533 (19)	0.87979 (13)	-0.21289 (7)	0.01730 (17)
H8A	0.4281	1.0082	-0.2152	0.021*
H8B	0.3242	0.8363	-0.2352	0.021*
C9	0.72100 (18)	0.81319 (12)	-0.28494 (7)	0.01526 (16)
H9A	0.7664	0.6847	-0.2840	0.018*
H9B	0.8733	0.8549	-0.2623	0.018*
C10	0.67469 (19)	0.87506 (12)	-0.39261 (7)	0.01705 (17)
H10A	0.5112	0.8435	-0.4123	0.020*
H10B	0.6458	1.0032	-0.3943	0.020*
C11	0.9070 (2)	0.79692 (14)	-0.46899 (8)	0.02213 (19)
H11A	1.0696	0.8277	-0.4500	0.033*
H11B	0.8691	0.8425	-0.5363	0.033*
H11C	0.9317	0.6703	-0.4698	0.033*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0106 (3)	0.0251 (4)	0.0134 (3)	-0.0064 (3)	-0.0033 (3)	0.0048 (3)
C1	0.0120 (4)	0.0133 (3)	0.0131 (4)	-0.0024 (3)	-0.0018 (3)	0.0006 (3)
O1	0.0097 (3)	0.0217 (3)	0.0170 (3)	-0.0028 (2)	-0.0029 (2)	0.0024 (2)
C2	0.0115 (4)	0.0138 (3)	0.0131 (4)	-0.0038 (3)	-0.0025 (3)	0.0013 (3)
C3	0.0132 (4)	0.0188 (4)	0.0154 (4)	-0.0017 (3)	-0.0005 (3)	0.0009 (3)
C4	0.0185 (4)	0.0224 (4)	0.0140 (4)	-0.0043 (3)	0.0006 (3)	0.0006 (3)
C5	0.0213 (4)	0.0170 (4)	0.0138 (4)	-0.0074 (3)	-0.0055 (3)	0.0036 (3)
C11	0.03326 (15)	0.02986 (13)	0.01517 (12)	-0.00986 (10)	-0.00874 (9)	0.00705 (8)
C6	0.0160 (4)	0.0180 (4)	0.0184 (4)	-0.0030 (3)	-0.0058 (3)	0.0038 (3)
C7	0.0117 (4)	0.0177 (4)	0.0159 (4)	-0.0019 (3)	-0.0015 (3)	0.0016 (3)
C8	0.0121 (4)	0.0261 (4)	0.0137 (4)	-0.0040 (3)	-0.0022 (3)	0.0059 (3)
C9	0.0115 (4)	0.0202 (4)	0.0143 (4)	-0.0034 (3)	-0.0028 (3)	0.0018 (3)
C10	0.0138 (4)	0.0230 (4)	0.0139 (4)	-0.0032 (3)	-0.0020 (3)	0.0029 (3)
C11	0.0185 (5)	0.0300 (5)	0.0170 (4)	-0.0043 (4)	0.0006 (3)	-0.0014 (3)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.3446 (12)	C6—H6	0.9500
N1—C8	1.4598 (11)	C7—H7	0.9500
N1—HN1	0.831 (15)	C8—C9	1.5185 (13)
C1—O1	1.2378 (11)	C8—H8A	0.9900
C1—C2	1.4984 (12)	C8—H8B	0.9900
C2—C3	1.3952 (12)	C9—C10	1.5290 (12)
C2—C7	1.3955 (12)	C9—H9A	0.9900
C3—C4	1.3891 (13)	C9—H9B	0.9900
C3—H3	0.9500	C10—C11	1.5242 (13)
C4—C5	1.3918 (13)	C10—H10A	0.9900
C4—H4	0.9500	C10—H10B	0.9900
C5—C6	1.3848 (14)	C11—H11A	0.9800

C5—C11	1.7405 (9)	C11—H11B	0.9800
C6—C7	1.3931 (12)	C11—H11C	0.9800
C1—N1—C8	121.48 (8)	N1—C8—C9	112.18 (7)
C1—N1—HN1	119.5 (10)	N1—C8—H8A	109.2
C8—N1—HN1	118.4 (10)	C9—C8—H8A	109.2
O1—C1—N1	122.89 (8)	N1—C8—H8B	109.2
O1—C1—C2	120.60 (8)	C9—C8—H8B	109.2
N1—C1—C2	116.51 (8)	H8A—C8—H8B	107.9
C3—C2—C7	119.40 (8)	C8—C9—C10	111.15 (7)
C3—C2—C1	117.87 (8)	C8—C9—H9A	109.4
C7—C2—C1	122.67 (8)	C10—C9—H9A	109.4
C4—C3—C2	120.71 (8)	C8—C9—H9B	109.4
C4—C3—H3	119.6	C10—C9—H9B	109.4
C2—C3—H3	119.6	H9A—C9—H9B	108.0
C3—C4—C5	118.78 (9)	C11—C10—C9	112.75 (8)
C3—C4—H4	120.6	C11—C10—H10A	109.0
C5—C4—H4	120.6	C9—C10—H10A	109.0
C6—C5—C4	121.65 (8)	C11—C10—H10B	109.0
C6—C5—C11	118.96 (7)	C9—C10—H10B	109.0
C4—C5—C11	119.39 (7)	H10A—C10—H10B	107.8
C5—C6—C7	118.95 (8)	C10—C11—H11A	109.5
C5—C6—H6	120.5	C10—C11—H11B	109.5
C7—C6—H6	120.5	H11A—C11—H11B	109.5
C6—C7—C2	120.49 (9)	C10—C11—H11C	109.5
C6—C7—H7	119.8	H11A—C11—H11C	109.5
C2—C7—H7	119.8	H11B—C11—H11C	109.5
C8—N1—C1—O1	-0.14 (13)	C3—C4—C5—C11	179.68 (7)
C8—N1—C1—C2	179.00 (8)	C4—C5—C6—C7	1.22 (14)
O1—C1—C2—C3	-30.72 (12)	C11—C5—C6—C7	-178.56 (7)
N1—C1—C2—C3	150.11 (8)	C5—C6—C7—C2	-1.35 (13)
O1—C1—C2—C7	146.41 (9)	C3—C2—C7—C6	0.37 (13)
N1—C1—C2—C7	-32.76 (12)	C1—C2—C7—C6	-176.72 (8)
C7—C2—C3—C4	0.79 (13)	C1—N1—C8—C9	-149.00 (8)
C1—C2—C3—C4	178.01 (8)	N1—C8—C9—C10	-178.91 (7)
C2—C3—C4—C5	-0.92 (14)	C8—C9—C10—C11	-174.52 (8)
C3—C4—C5—C6	-0.10 (14)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—HN1...O1 ⁱ	0.831 (15)	2.203 (15)	3.0164 (10)	166.3 (13)
C3—H3...O1 ⁱⁱ	0.95	2.66	3.3146 (11)	127
C8—H8A...Cg1 ⁱⁱⁱ	0.99	2.84	3.697 (16)	145

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+2, -z$; (iii) $-x+1, -y, -z$.

Fig. 1

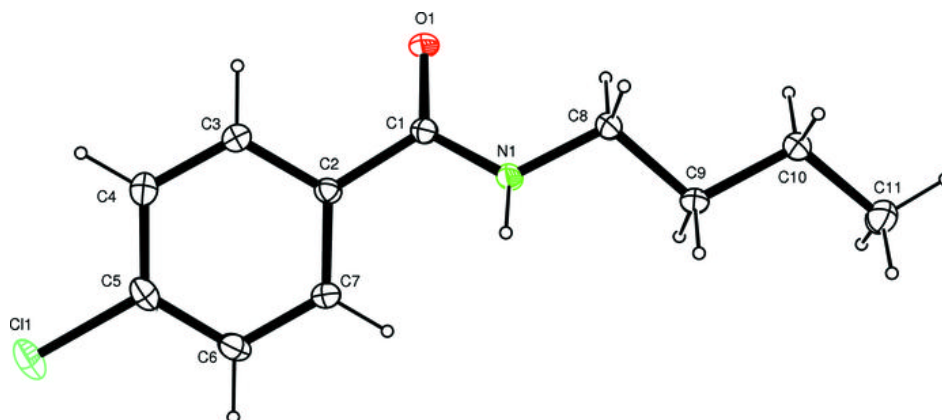


Fig. 2

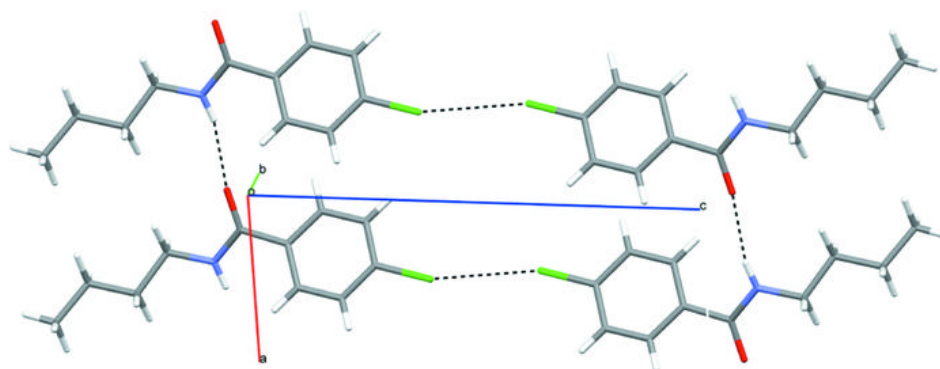


Fig. 3

