

3-Chloro-*N*-(3-methylphenyl)benzamideVinola Z. Rodrigues,^a Lenka Kucková,^b B. Thimme Gowda^{a*} and Jozef Kožisek^b^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Physical Chemistry and Chemical Physics, Slovak University of Technology, Radlinského 9, SK-812 37 Bratislava, Slovak Republic
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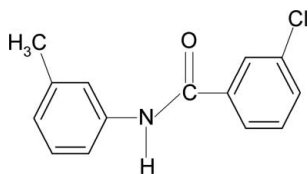
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.063; wR factor = 0.191; data-to-parameter ratio = 22.3.

In the molecular structure of the title compound, $\text{C}_{14}\text{H}_{12}\text{ClNO}$, the *meta*-Cl atom in the benzoyl ring is positioned *syn* to the $\text{C}=\text{O}$ bond, while the *meta*-methyl group in the aniline ring is positioned *anti* to the $\text{N}-\text{H}$ bond. The two aromatic rings make a dihedral angle of $77.4(1)^\circ$. In the crystal, the molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming $C(4)$ chains propagating in $[010]$.

Related literature

For preparation of the title compound, see: Gowda *et al.* (2003). For our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Bhat & Gowda (2000); Bowes *et al.* (2003); Gowda *et al.* (2008); Saeed *et al.* (2010), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007), on *N*-(aryl)-arylsulfonamides, see: Shetty & Gowda (2005) and on *N*-chloro-amides, see: Gowda & Weiss (1994).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClNO}$ $V = 2467.46(11)$ Å³
 $M_r = 245.70$ $Z = 8$
Orthorhombic, *Pbcn* $\text{Mo K}\alpha$ radiation
 $a = 9.4032(3)$ Å $\mu = 0.29$ mm⁻¹
 $b = 10.0963(2)$ Å $T = 298$ K
 $c = 25.9904(7)$ Å $0.38 \times 0.24 \times 0.04$ mm

Data collection

Oxford Diffraction Xcalibur Ruby
Gemini diffractometer
Absorption correction: analytical
[*CrysAlis RED* (Oxford
Diffraction, 2009), based on
expressions derived by Clark &
Reid (1995)]
 $T_{\min} = 0.921$, $T_{\max} = 0.988$
39014 measured reflections
3440 independent reflections
1666 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.191$
 $S = 1.02$
3440 reflections
154 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.86	2.10	2.938 (3)	163

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2002); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5712).

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supplementary materials

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3-Chloro-*N*-(3-methylphenyl)benzamide

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Comment

The amide and sulfonamide moieties are the constituents of many biologically important compounds. As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Bhat & Gowda, 2000; Bowes *et al.*, 2003; Gowda *et al.*, 2008; Saeed *et al.*, 2010), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007), *N*-(aryl)-arylsulfonamides (Shetty & Gowda, 2005) and *N*-chloro-arylamides (Gowda & Weiss, 1994), in the present work, the crystal structure of 3-Chloro-*N*-(3-methylphenyl)benzamide (I) has been determined (Fig.1).

In (I), the *meta*-Cl atom in the benzoyl ring is positioned *syn* to the C=O bond, while the *meta*-methyl group in the anilino ring is positioned *anti* to the N—H bond, the N—H and C=O bonds in the C—NH—C(O)—C segment being *anti* to each other. Further, the two aromatic rings make the dihedral angle of 77.4 (1)°, compared to the values of 9.1 (2)° and 7.3 (3)° in the two independent molecules of 3-chloro-*N*-(3-chlorophenyl)benzamide (Gowda *et al.*, 2008).

In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into infinite chains running along the *b*-axis. Part of the crystal structure is shown in Fig. 2.

Experimental

The title compound was prepared according to the method described by Gowda *et al.* (2003). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra.

Plate like colorless single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of an ethanol solution of the compound (0.5 g in about 30 ml of ethanol) at room temperature.

Refinement

All H atoms were visible in difference maps and then treated as riding atoms with C—H distances of 0.93 Å (C-aromatic), 0.96 Å (C-methyl) and N—H = 0.86 Å. The $U_{\text{iso}}(\text{H})$ values were set at 1.2 $U_{\text{eq}}(\text{C-aromatic, N})$ and 1.5 $U_{\text{eq}}(\text{C-methyl})$.

Figures

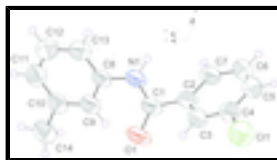


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

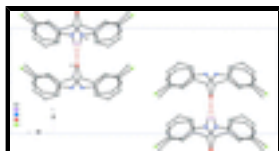


Fig. 2. Part of the crystal structure of the title compound. Molecular chains are generated by N—H...O hydrogen bonds which are shown by dashed lines.

3-Chloro-*N*-(3-methylphenyl)benzamide

Crystal data

$C_{14}H_{12}ClNO$	$F(000) = 1024$
$M_r = 245.70$	$D_x = 1.323 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2n 2ab	Cell parameters from 6149 reflections
$a = 9.4032 (3) \text{ \AA}$	$\theta = 2.2\text{--}29.5^\circ$
$b = 10.0963 (2) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$c = 25.9904 (7) \text{ \AA}$	$T = 298 \text{ K}$
$V = 2467.46 (11) \text{ \AA}^3$	Plate, colourless
$Z = 8$	$0.38 \times 0.24 \times 0.04 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer	3440 independent reflections
Radiation source: fine-focus sealed tube graphite	1666 reflections with $I > 2\sigma(I)$
Detector resolution: $10.4340 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.072$
ω scans	$\theta_{\text{max}} = 29.5^\circ$, $\theta_{\text{min}} = 2.7^\circ$
Absorption correction: analytical [<i>Crys.Alis RED</i> (Oxford Diffraction, 2009), based on expressions derived by Clark & Reid (1995)]	$h = -13 \rightarrow 13$
$T_{\text{min}} = 0.921$, $T_{\text{max}} = 0.988$	$k = -14 \rightarrow 14$
39014 measured reflections	$l = -36 \rightarrow 36$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.063$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.191$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2 + 0.6744P]$
3440 reflections	where $P = (F_o^2 + 2F_c^2)/3$
154 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived (Clark & Reid, 1995).

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2146 (3)	0.4686 (2)	0.23782 (10)	0.0575 (6)
C2	0.1595 (3)	0.5148 (2)	0.18676 (10)	0.0559 (6)
C3	0.1825 (3)	0.4345 (3)	0.14430 (10)	0.0615 (7)
H3A	0.2272	0.3530	0.1483	0.074*
C4	0.1393 (3)	0.4751 (3)	0.09637 (10)	0.0657 (7)
C5	0.0675 (3)	0.5920 (3)	0.08959 (11)	0.0743 (8)
H5A	0.0374	0.6179	0.0570	0.089*
C6	0.0407 (3)	0.6699 (3)	0.13170 (13)	0.0764 (8)
H6A	-0.0099	0.7484	0.1277	0.092*
C7	0.0881 (3)	0.6333 (2)	0.18017 (11)	0.0657 (7)
H7A	0.0718	0.6884	0.2082	0.079*
C8	0.3209 (3)	0.5467 (2)	0.31904 (10)	0.0546 (6)
C9	0.2617 (3)	0.4605 (2)	0.35419 (10)	0.0588 (6)
H9A	0.1822	0.4109	0.3451	0.071*
C10	0.3201 (3)	0.4473 (2)	0.40297 (10)	0.0601 (7)
C11	0.4389 (3)	0.5208 (3)	0.41499 (11)	0.0719 (8)
H11A	0.4797	0.5128	0.4474	0.086*
C12	0.4979 (3)	0.6061 (3)	0.37956 (13)	0.0770 (8)
H12A	0.5782	0.6550	0.3883	0.092*
C13	0.4390 (3)	0.6196 (3)	0.33154 (11)	0.0665 (7)
H13A	0.4788	0.6775	0.3077	0.080*
C14	0.2552 (4)	0.3530 (3)	0.44195 (12)	0.0796 (9)
H14C	0.3088	0.3565	0.4734	0.095*
H14B	0.2572	0.2644	0.4285	0.095*
H14A	0.1586	0.3784	0.4486	0.095*
N1	0.2606 (2)	0.56520 (19)	0.26937 (8)	0.0600 (6)
H1N	0.2529	0.6453	0.2585	0.072*
O1	0.2191 (2)	0.35053 (16)	0.24849 (7)	0.0776 (6)
Cl1	0.17750 (10)	0.37730 (9)	0.04296 (3)	0.0932 (4)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0787 (16)	0.0375 (13)	0.0563 (15)	0.0021 (11)	0.0064 (13)	0.0007 (10)
C2	0.0672 (15)	0.0420 (12)	0.0585 (15)	-0.0051 (12)	0.0003 (12)	0.0033 (11)
C3	0.0768 (17)	0.0486 (14)	0.0591 (16)	0.0002 (12)	-0.0029 (13)	0.0033 (12)
C4	0.0783 (17)	0.0599 (16)	0.0590 (17)	-0.0098 (14)	-0.0038 (13)	0.0027 (12)
C5	0.087 (2)	0.0708 (18)	0.0650 (18)	-0.0045 (16)	-0.0159 (15)	0.0150 (15)
C6	0.0796 (19)	0.0596 (16)	0.090 (2)	0.0089 (14)	-0.0155 (17)	0.0102 (16)
C7	0.0780 (18)	0.0488 (14)	0.0702 (18)	-0.0001 (13)	-0.0028 (14)	0.0026 (12)
C8	0.0717 (16)	0.0376 (12)	0.0546 (15)	0.0096 (11)	0.0009 (12)	0.0013 (10)
C9	0.0708 (16)	0.0442 (13)	0.0615 (16)	0.0051 (12)	0.0008 (13)	0.0018 (11)
C10	0.0785 (18)	0.0481 (14)	0.0538 (15)	0.0142 (13)	0.0031 (13)	-0.0001 (11)
C11	0.0861 (19)	0.0670 (17)	0.0626 (17)	0.0130 (16)	-0.0118 (15)	-0.0082 (14)
C12	0.085 (2)	0.0655 (18)	0.081 (2)	-0.0042 (15)	-0.0085 (16)	-0.0060 (16)
C13	0.0766 (18)	0.0520 (15)	0.0708 (18)	-0.0030 (13)	0.0031 (15)	-0.0007 (13)
C14	0.100 (2)	0.0742 (19)	0.0640 (18)	0.0066 (16)	0.0039 (17)	0.0155 (15)
N1	0.0853 (15)	0.0348 (10)	0.0599 (13)	0.0008 (10)	-0.0027 (11)	0.0053 (9)
O1	0.1386 (18)	0.0328 (9)	0.0615 (12)	0.0018 (10)	-0.0036 (11)	0.0041 (8)
Cl1	0.1290 (8)	0.0918 (7)	0.0588 (5)	-0.0005 (5)	-0.0013 (4)	-0.0071 (4)

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

C1—O1	1.225 (3)	C8—C9	1.379 (3)
C1—N1	1.345 (3)	C8—N1	1.422 (3)
C1—C2	1.499 (4)	C9—C10	1.388 (4)
C2—C7	1.383 (3)	C9—H9A	0.9300
C2—C3	1.386 (4)	C10—C11	1.377 (4)
C3—C4	1.373 (4)	C10—C14	1.518 (4)
C3—H3A	0.9300	C11—C12	1.377 (4)
C4—C5	1.371 (4)	C11—H11A	0.9300
C4—Cl1	1.741 (3)	C12—C13	1.372 (4)
C5—C6	1.371 (4)	C12—H12A	0.9300
C5—H5A	0.9300	C13—H13A	0.9300
C6—C7	1.386 (4)	C14—H14C	0.9600
C6—H6A	0.9300	C14—H14B	0.9600
C7—H7A	0.9300	C14—H14A	0.9600
C8—C13	1.371 (4)	N1—H1N	0.8600
O1—C1—N1	123.8 (2)	C8—C9—C10	120.4 (3)
O1—C1—C2	121.0 (2)	C8—C9—H9A	119.8
N1—C1—C2	115.2 (2)	C10—C9—H9A	119.8
C7—C2—C3	118.9 (2)	C11—C10—C9	118.4 (3)
C7—C2—C1	123.1 (2)	C11—C10—C14	120.8 (3)
C3—C2—C1	118.0 (2)	C9—C10—C14	120.7 (3)
C4—C3—C2	120.2 (2)	C10—C11—C12	120.8 (3)
C4—C3—H3A	119.9	C10—C11—H11A	119.6
C2—C3—H3A	119.9	C12—C11—H11A	119.6

C5—C4—C3	121.3 (3)	C13—C12—C11	120.5 (3)
C5—C4—C11	119.2 (2)	C13—C12—H12A	119.8
C3—C4—C11	119.6 (2)	C11—C12—H12A	119.8
C4—C5—C6	118.8 (3)	C8—C13—C12	119.3 (3)
C4—C5—H5A	120.6	C8—C13—H13A	120.4
C6—C5—H5A	120.6	C12—C13—H13A	120.4
C5—C6—C7	120.9 (3)	C10—C14—H14C	109.5
C5—C6—H6A	119.6	C10—C14—H14B	109.5
C7—C6—H6A	119.6	H14C—C14—H14B	109.5
C2—C7—C6	119.9 (3)	C10—C14—H14A	109.5
C2—C7—H7A	120.0	H14C—C14—H14A	109.5
C6—C7—H7A	120.0	H14B—C14—H14A	109.5
C13—C8—C9	120.6 (2)	C1—N1—C8	125.9 (2)
C13—C8—N1	117.9 (2)	C1—N1—H1N	117.1
C9—C8—N1	121.5 (2)	C8—N1—H1N	117.1
O1—C1—C2—C7	147.5 (3)	C13—C8—C9—C10	0.7 (4)
N1—C1—C2—C7	-34.1 (4)	N1—C8—C9—C10	-178.0 (2)
O1—C1—C2—C3	-32.8 (4)	C8—C9—C10—C11	-0.8 (4)
N1—C1—C2—C3	145.6 (2)	C8—C9—C10—C14	179.8 (2)
C7—C2—C3—C4	2.4 (4)	C9—C10—C11—C12	0.4 (4)
C1—C2—C3—C4	-177.3 (2)	C14—C10—C11—C12	179.8 (3)
C2—C3—C4—C5	-2.9 (4)	C10—C11—C12—C13	0.1 (4)
C2—C3—C4—C11	176.50 (19)	C9—C8—C13—C12	-0.1 (4)
C3—C4—C5—C6	0.9 (4)	N1—C8—C13—C12	178.6 (2)
C11—C4—C5—C6	-178.5 (2)	C11—C12—C13—C8	-0.3 (4)
C4—C5—C6—C7	1.5 (5)	O1—C1—N1—C8	0.1 (4)
C3—C2—C7—C6	0.0 (4)	C2—C1—N1—C8	-178.2 (2)
C1—C2—C7—C6	179.7 (2)	C13—C8—N1—C1	136.5 (3)
C5—C6—C7—C2	-2.0 (4)	C9—C8—N1—C1	-44.7 (4)

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—H1N...O1 ⁱ	0.86	2.10	2.938 (3)	163.

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

Fig. 1

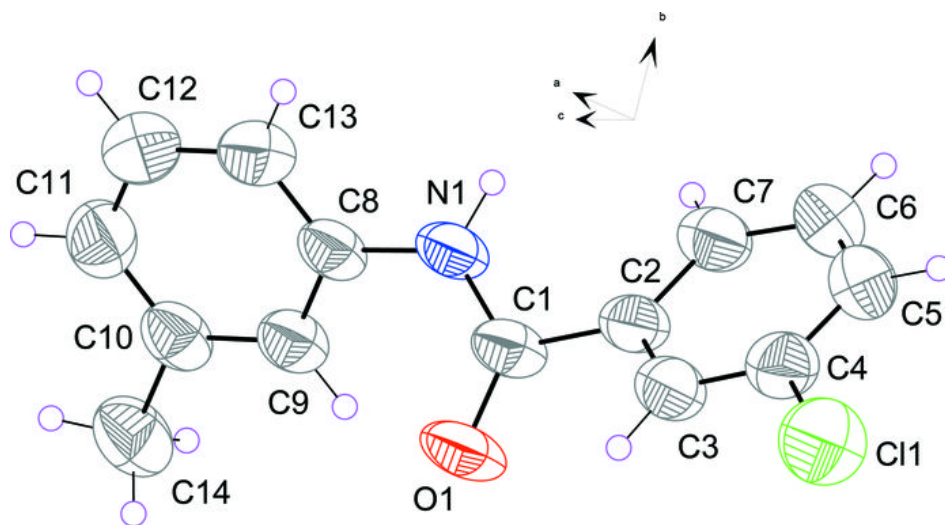


Fig. 2

