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4-Chloro-*N*-(3-chlorophenyl)benzamide

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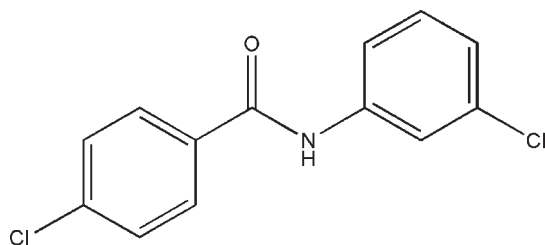
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Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.104; data-to-parameter ratio = 15.5.

The title compound, $\text{C}_{13}\text{H}_9\text{Cl}_2\text{N}$, has an intramolecular $\text{C}-\text{H}\cdots\text{O}$ close contact, and presents the NH group *syn* to the *meta*-chloro group in the aniline ring and *trans* to the $\text{C}=\text{O}$ group. The crystal packing is formed by infinite chains of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds along the c axis. $\text{Cl}\cdots\text{Cl}$ [3.474 (1) Å] contacts link chains. The crystal used for data collection was a twin, the domains related by the twin law 0.948 (1)/0.052 (1).

Related literature

For halogen interactions in the benzanilide series, see: Chopra & Guru Row (2005, 2008); Saeed *et al.* (2008); Gowda *et al.* (2008). For $\text{Cl}\cdots\text{Cl}$ interactions, see: Bui *et al.* (2009). For the program *ROTAX*, used to determine the twin law, see: Pearson & Gould (2003).



Experimental

Crystal data

$\text{C}_{13}\text{H}_9\text{Cl}_2\text{NO}$
 $M_r = 266.11$
 Monoclinic, $P2_1/c$
 $a = 12.8696$ (15) Å
 $b = 9.7485$ (10) Å
 $c = 9.8243$ (12) Å
 $\beta = 90.265$ (11)°

$V = 1232.5$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.51$ mm⁻¹
 $T = 292$ K
 $0.42 \times 0.28 \times 0.19$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with an Eos (Nova) detector
 Absorption correction: multi-scan (*CrysAlis Pro*; Oxford)

Diffraction, 2009)
 $T_{\min} = 0.815$, $T_{\max} = 0.910$
 13416 measured reflections
 2407 independent reflections
 1678 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.104$
 $S = 1.03$
 2407 reflections

155 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.16$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.86	2.05	2.883 (2)	163
$\text{C13}-\text{H13}\cdots\text{O1}$	0.93	2.34	2.868 (3)	116

Symmetry code: (i) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: *CrysAlis Pro* (Oxford Diffraction, 2009); cell refinement: *CrysAlis Pro*; data reduction: *CrysAlis Pro*; 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) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2292).

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supplementary materials

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4-Chloro-*N*-(3-chlorophenyl)benzamide

S. K. Nayak, M. K. Reddy and T. N. Guru Row

Comment

The Structure of 4-chloro-*N*-(3-chlorophenyl) benzamide is an extension of our previous work to evaluate the importance of interactions involving halogens in the benzanilide series (Chopra & Guru Row, 2005, 2008). The molecular structure prefers the N—H group to be *trans* to the CO group resulting in the formation of a C—H \cdots O intramolecular interaction (Fig. 1, Table 1) similar to those found in the flourine compounds (Chopra & Guru Row, 2008; Saeed *et al.*, 2008) (Figure 1). The NHCO group forms dihedral angles of 20.2 (2) and 21.5 (1) $^\circ$ with the aniline and benzoyl rings respectively. The two rings are nearly coplanar, with dihedral angle of 3.7 (2) $^\circ$. The crystal packing is formed by infinite chains with N—H \cdots O hydrogen bonds along the *c* axis (Figure 2, Table 1). Similar interactions were observed in the analogous chloro substituted benzanilides (Gowda *et al.*, 2008; Saeed *et al.*, 2008). There is a halogen Cl1 \cdots Cl1ⁱ contact (i): $-x + 1, -y + 2, -z, (3.47 (1) \text{ \AA}, [\text{Type-I}, \theta_1=\theta_2=171.1 (2)^\circ]$ (Bui *et al.*, 2009) which links chains across an inversion centre, (Figure 2). In addition $\pi\cdots\pi$ stacking enhance the stability of the packing across the centres of symmetry $(Cg_1\cdots Cg_1^{ii}=3.71 (2)\text{ \AA}, Cg_2\cdots Cg_2^{iii}=3.77 (2)\text{ \AA})$; (ii): $1-x, 1-y, 1-z$; (iii): $-x, 2-y, 1-z$; Cg1: centroid of the C1 \rightarrow C6 ring; Cg2: centroid of the C8 \rightarrow C13 ring).

Experimental

The title compound (Scheme) was prepared according to the literature method (Chopra & Guru Row, 2005). The purity of the compound was confirmed by infrared and NMR spectra. Single crystals were grown from ethanol at room temperature and used for X-ray diffraction study.

Refinement

All H atoms were positioned geometrically, (C—H = 0.93 \AA , N—H = 0.86 \AA) and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$. The crystal used for data collection was a twin, with twin law $1\ 0\ 0, 0\ \bar{1}\ 0, 0\ 0\ \bar{1}$, as disclosed by ROTAX, Pearson & Gould (2003), and confirmed by refinement with the TWIN instruction in SHELXL97, Sheldrick (2008), leading to a distribution (BASF parameter) of 0.948/0.052 (1).

Figures

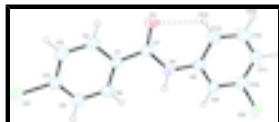


Fig. 1. : Molecular structure shows the atom labelling Scheme with displacement ellipsoids for non-H atoms at 50% probability level, hydrogen atoms are arbitrary circle. The dotted line shows the C—H \cdots O intramolecular interactions.

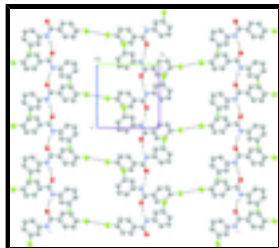


Fig. 2. : The molecular packing shows the infinite chain of N—H...O hydrogen bonds along *c* axis and Cl...Cl interactions as a linker between the chains.

4-Chloro-*N*-(3-chlorophenyl)benzamide

Crystal data

$C_{13}H_9Cl_2NO$

$M_r = 266.11$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.8696$ (15) Å

$b = 9.7485$ (10) Å

$c = 9.8243$ (12) Å

$\beta = 90.265$ (11)°

$V = 1232.5$ (2) Å³

$Z = 4$

$F_{000} = 544$

$D_x = 1.434$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å

Cell parameters from 350 reflections

$\theta = 1.0$ – 28.0°

$\mu = 0.51$ mm⁻¹

$T = 292$ K

Plate, colorless

$0.42 \times 0.28 \times 0.19$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with an Eos (Nova) detector

Radiation source: Enhance (Mo) X-ray Source

Monochromator: graphite

Detector resolution: 16.0839 pixels mm⁻¹

$T = 292$ K

ω scans

Absorption correction: multi-scan (CrysAlis Pro; Oxford Diffraction, 2009)

$T_{\min} = 0.815$, $T_{\max} = 0.910$

13416 measured reflections

2407 independent reflections

1678 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$

$\theta_{\max} = 26.0^\circ$

$\theta_{\min} = 3.2^\circ$

$h = -15 \rightarrow 15$

$k = -12 \rightarrow 12$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.104$

$S = 1.03$

2407 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.16$ e Å⁻³

155 parameters

$$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C12	-0.01097 (5)	1.07010 (7)	0.17348 (7)	0.0830 (3)
C11	0.45841 (6)	0.16654 (6)	0.53117 (7)	0.0811 (3)
O1	0.27955 (14)	0.79207 (16)	0.68009 (14)	0.0682 (5)
N1	0.24298 (14)	0.79496 (18)	0.45618 (16)	0.0512 (4)
H1N	0.2531	0.7516	0.3812	0.061*
C7	0.28063 (16)	0.7347 (2)	0.56910 (19)	0.0485 (5)
C1	0.32291 (15)	0.5931 (2)	0.55447 (18)	0.0459 (5)
C9	0.12551 (16)	0.9361 (2)	0.3325 (2)	0.0513 (5)
H9	0.1205	0.8662	0.2683	0.062*
C8	0.18891 (16)	0.9205 (2)	0.44516 (19)	0.0464 (5)
C6	0.29668 (18)	0.5044 (2)	0.4485 (2)	0.0561 (6)
H6	0.2510	0.5339	0.3810	0.067*
C2	0.39013 (18)	0.5444 (2)	0.6539 (2)	0.0599 (6)
H2	0.4077	0.6010	0.7267	0.072*
C13	0.19596 (19)	1.0256 (2)	0.5399 (2)	0.0602 (6)
H13	0.2385	1.0167	0.6162	0.072*
C3	0.43165 (19)	0.4141 (2)	0.6475 (2)	0.0634 (6)
H3	0.4773	0.3836	0.7147	0.076*
C4	0.40478 (17)	0.3300 (2)	0.5410 (2)	0.0559 (6)
C5	0.33695 (18)	0.3742 (2)	0.4418 (2)	0.0602 (6)
H5	0.3185	0.3162	0.3705	0.072*
C11	0.0749 (2)	1.1604 (2)	0.4082 (3)	0.0699 (7)
H11	0.0364	1.2403	0.3963	0.084*
C10	0.06962 (18)	1.0554 (2)	0.3151 (2)	0.0577 (6)
C12	0.1386 (2)	1.1438 (3)	0.5190 (3)	0.0748 (7)
H12	0.1434	1.2143	0.5825	0.090*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C12	0.0831 (5)	0.0747 (5)	0.0911 (5)	0.0067 (3)	-0.0221 (4)	0.0229 (3)
C11	0.0923 (5)	0.0617 (4)	0.0892 (5)	0.0194 (3)	-0.0156 (4)	0.0071 (3)
O1	0.1067 (13)	0.0635 (9)	0.0343 (8)	0.0055 (9)	0.0007 (8)	-0.0038 (7)
N1	0.0644 (11)	0.0550 (10)	0.0343 (9)	0.0082 (9)	-0.0023 (8)	-0.0031 (7)
C7	0.0550 (13)	0.0561 (13)	0.0344 (11)	-0.0050 (10)	0.0026 (9)	0.0013 (10)
C1	0.0491 (12)	0.0533 (13)	0.0351 (10)	-0.0028 (10)	-0.0013 (9)	0.0045 (9)
C9	0.0556 (13)	0.0462 (12)	0.0520 (13)	-0.0034 (10)	0.0010 (10)	0.0026 (9)
C8	0.0492 (12)	0.0462 (12)	0.0439 (11)	-0.0008 (9)	0.0080 (9)	0.0015 (9)
C6	0.0640 (14)	0.0567 (14)	0.0475 (12)	0.0033 (11)	-0.0149 (11)	0.0039 (10)
C2	0.0721 (15)	0.0671 (15)	0.0402 (12)	0.0010 (12)	-0.0125 (11)	0.0003 (10)
C13	0.0698 (15)	0.0545 (14)	0.0562 (13)	-0.0013 (12)	-0.0024 (12)	-0.0046 (11)
C3	0.0696 (15)	0.0708 (16)	0.0497 (13)	0.0078 (12)	-0.0142 (12)	0.0146 (11)
C4	0.0595 (14)	0.0525 (13)	0.0557 (13)	0.0053 (10)	-0.0036 (11)	0.0106 (10)
C5	0.0723 (15)	0.0546 (14)	0.0536 (13)	0.0021 (12)	-0.0141 (11)	-0.0016 (11)
C11	0.0802 (17)	0.0479 (14)	0.0816 (18)	0.0108 (12)	0.0045 (15)	0.0070 (12)
C10	0.0582 (14)	0.0512 (13)	0.0638 (14)	0.0002 (11)	0.0019 (11)	0.0132 (11)
C12	0.096 (2)	0.0515 (15)	0.0774 (17)	0.0021 (14)	0.0097 (16)	-0.0137 (13)

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

C12—C10	1.738 (2)	C6—H6	0.9300
C11—C4	1.740 (2)	C2—C3	1.380 (3)
O1—C7	1.226 (2)	C2—H2	0.9300
N1—C7	1.344 (2)	C13—C12	1.384 (3)
N1—C8	1.412 (3)	C13—H13	0.9300
N1—H1N	0.8600	C3—C4	1.373 (3)
C7—C1	1.491 (3)	C3—H3	0.9300
C1—C2	1.385 (3)	C4—C5	1.375 (3)
C1—C6	1.394 (3)	C5—H5	0.9300
C9—C10	1.378 (3)	C11—C12	1.369 (4)
C9—C8	1.381 (3)	C11—C10	1.374 (3)
C9—H9	0.9300	C11—H11	0.9300
C8—C13	1.386 (3)	C12—H12	0.9300
C6—C5	1.372 (3)		
C7—N1—C8	128.18 (17)	C12—C13—C8	118.8 (2)
C7—N1—H1N	115.9	C12—C13—H13	120.6
C8—N1—H1N	115.9	C8—C13—H13	120.6
O1—C7—N1	121.9 (2)	C4—C3—C2	119.22 (19)
O1—C7—C1	120.94 (18)	C4—C3—H3	120.4
N1—C7—C1	117.11 (17)	C2—C3—H3	120.4
C2—C1—C6	117.5 (2)	C3—C4—C5	120.7 (2)
C2—C1—C7	118.44 (18)	C3—C4—C11	119.41 (17)
C6—C1—C7	124.01 (17)	C5—C4—C11	119.90 (18)
C10—C9—C8	119.9 (2)	C6—C5—C4	119.6 (2)

C10—C9—H9	120.1	C6—C5—H5	120.2
C8—C9—H9	120.1	C4—C5—H5	120.2
C9—C8—C13	119.6 (2)	C12—C11—C10	117.9 (2)
C9—C8—N1	116.49 (18)	C12—C11—H11	121.0
C13—C8—N1	123.91 (19)	C10—C11—H11	121.0
C5—C6—C1	121.30 (19)	C11—C10—C9	121.5 (2)
C5—C6—H6	119.4	C11—C10—C12	119.93 (18)
C1—C6—H6	119.4	C9—C10—C12	118.53 (18)
C3—C2—C1	121.6 (2)	C11—C12—C13	122.3 (2)
C3—C2—H2	119.2	C11—C12—H12	118.9
C1—C2—H2	119.2	C13—C12—H12	118.9
C8—N1—C7—O1	-6.8 (3)	C9—C8—C13—C12	0.2 (3)
C8—N1—C7—C1	172.39 (18)	N1—C8—C13—C12	-179.4 (2)
O1—C7—C1—C2	-20.6 (3)	C1—C2—C3—C4	-0.8 (4)
N1—C7—C1—C2	160.2 (2)	C2—C3—C4—C5	-0.3 (4)
O1—C7—C1—C6	157.4 (2)	C2—C3—C4—C11	179.05 (18)
N1—C7—C1—C6	-21.8 (3)	C1—C6—C5—C4	-0.2 (3)
C10—C9—C8—C13	-0.2 (3)	C3—C4—C5—C6	0.8 (4)
C10—C9—C8—N1	179.50 (18)	C11—C4—C5—C6	-178.57 (18)
C7—N1—C8—C9	-156.2 (2)	C12—C11—C10—C9	0.6 (4)
C7—N1—C8—C13	23.4 (3)	C12—C11—C10—C12	179.3 (2)
C2—C1—C6—C5	-0.8 (3)	C8—C9—C10—C11	-0.3 (3)
C7—C1—C6—C5	-178.9 (2)	C8—C9—C10—C12	-178.96 (16)
C6—C1—C2—C3	1.3 (3)	C10—C11—C12—C13	-0.6 (4)
C7—C1—C2—C3	179.5 (2)	C8—C13—C12—C11	0.1 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O1 ⁱ	0.86	2.05	2.883 (2)	163
C13—H13 \cdots O1	0.93	2.34	2.868 (3)	116

Symmetry codes: (i) $x, -y+3/2, z-1/2$.

Fig. 1

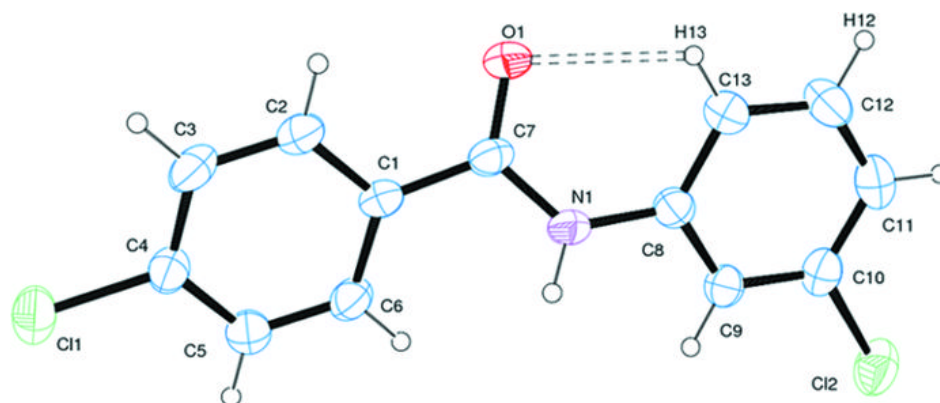


Fig. 2

