Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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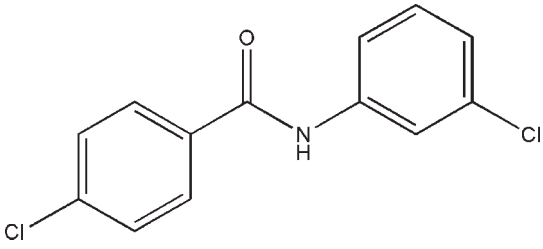
Received 11 August 2009; accepted 27 August 2009

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.104; data-to-parameter ratio = 15.5.

The title compound, $C_{13}H_9Cl_2N$, has an intramolecular C—H···O close contact, and presents the NH group *syn* to the *meta*-chloro group in the aniline ring and *trans* to the C=O group. The crystal packing is formed by infinite chains of N—H···O hydrogen bonds along the *c* axis. Cl···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 Cl···Cl interactions, see: Bui *et al.* (2009). For the program *ROTAX*, used to determine the twin law, see: Pearson & Gould (2003).



Experimental

Crystal data

$C_{13}H_9Cl_2NO$
M_r = 266.11
Monoclinic, $P2_1/c$
a = 12.8696 (15) Å
b = 9.7485 (10) Å
c = 9.8243 (12) Å
β = 90.265 (11)°
V = 1232.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 292 K
0.42 × 0.28 × 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_{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···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1^{i}	0.86	2.05	2.883 (2)	163
C13—H13···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).

We thank DST FIST (level II) for funding the XCalibur E Mova diffraction system. SKN thanks CSIR (SRF), INDIA, for financial support. We thank the referee for his valuable comments on handling the twinned crystal.

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

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supporting information

Acta Cryst. (2009). E**65**, o2434 [doi:10.1107/S1600536809034308]

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

Susanta K. Nayak, M. Kishore Reddy and T. N. Guru Row

S1. 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···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)° with the aniline and benzoyl rings respectively. The two rings are nearly coplanar, with dihedral angle of 3.7 (2)°. The crystal packing is formed by infinite chains with N—H···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···Cl1^{i} contact (i): -*x* + 1, -*y* + 2, -*z,* (3.47 (1) Å, [Type-I, θ_1=θ_2=171.1 (2)°] (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*$_1$···*Cg*$_1^{ii}$= 3.71 (2)Å, *Cg*$_2$···*Cg*$_2^{iii}$ = 3.77 (2)Å] ; (ii): 1-x,1-y,1-z ; (iii): -x,2-y,1-z; *Cg*1: centroid of the C1—>C6 ring; Cg2: centroid of the C8—>C13 ring).

S2. 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.

S3. Refinement

All H atoms were positioned geometrically, (C—H = 0.93 Å, N—H = 0.86 Å) and refined using a riding model with U_{iso}(H)= 1.2 U_{eq}(C, 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).

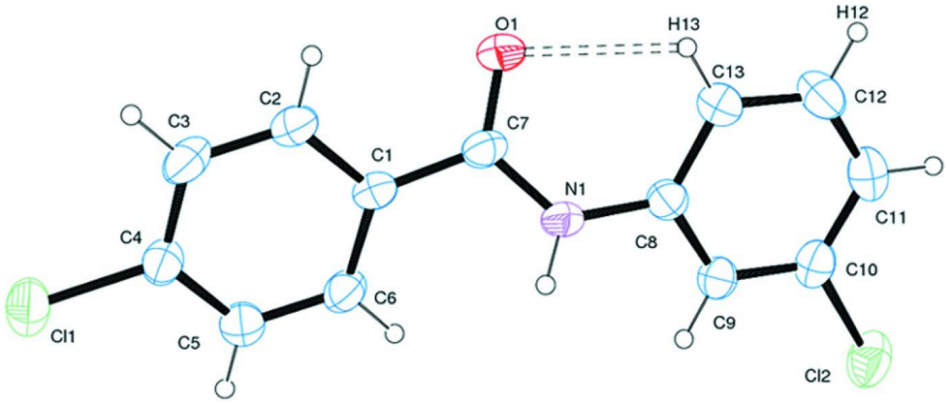

Figure 1

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

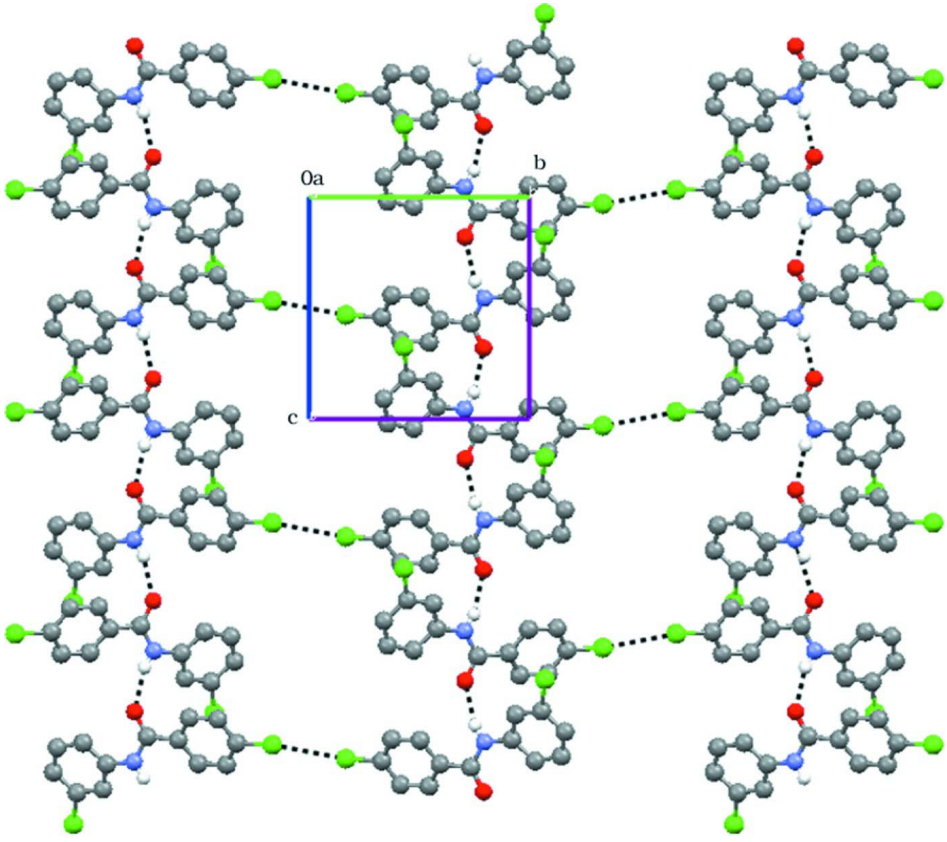

Figure 2

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
$\beta = 90.265\ (11)°$
$V = 1232.5\ (2)$ Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.434$ 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 × 0.28 × 0.19 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with an Eos (Nova) detector
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0839 pixels mm^{-1}
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 $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.2°$
$h = -15\rightarrow15$
$k = -12\rightarrow12$
$l = -12\rightarrow12$

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
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ 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}
Cl2	−0.01097 (5)	1.07010 (7)	0.17348 (7)	0.0830 (3)
Cl1	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*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0831 (5)	0.0747 (5)	0.0911 (5)	0.0067 (3)	−0.0221 (4)	0.0229 (3)
Cl1	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 (Å, º)

Cl2—C10	1.738 (2)	C6—H6	0.9300
Cl1—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—Cl1	119.41 (17)
C6—C1—C7	124.01 (17)	C5—C4—Cl1	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—Cl2	119.93 (18)
C1—C6—H6	119.4	C9—C10—Cl2	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—Cl1	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)	Cl1—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—Cl2	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—Cl2	−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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.86	2.05	2.883 (2)	163
C13—H13···O1	0.93	2.34	2.868 (3)	116

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