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4-Chloro-3-nitrobenzamide

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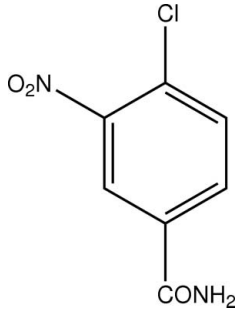
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.078; wR factor = 0.198; data-to-parameter ratio = 13.0.

In the crystal of the title compound, $C_7H_5ClN_2O_3$, the molecules are linked by N—H···O and C—H···O hydrogen bonds. The π–π contact between the benzene rings, [centroid–centroid distance = 3.803 (3) Å] may further stabilize the structure.

Related literature

For a related structure, see: Sun *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_7H_5ClN_2O_3$
M_r = 200.58
Monoclinic, $P2_1/n$
a = 8.8490 (18) Å
b = 7.5470 (15) Å
c = 12.374 (3) Å
β = 101.18 (3)°
V = 810.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.879, T_{max} = 0.957
1555 measured reflections
1459 independent reflections
1085 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.198
S = 1.01
1459 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O3^{i}	0.86	2.10	2.958 (6)	177
N2—H2C···O2^{ii}	0.86	2.26	3.067 (6)	155
C2—H2A···O3^{iii}	0.93	2.42	3.331 (6)	166

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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 *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2596).

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supporting information

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4-Chloro-3-nitrobenzamide

Bo-Nian Liu, Shi-Gui Tang, Hao-Yuan Li and Cheng Guo

S1. Comment

Some derivatives of pyridine are important chemical materials. We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Ring A (C1-C6) is, of course, planar. Atoms Cl, N1 and C7 are 0.021 (3), 0.029 (3) and -0.001 (3) Å away from the plane of the benzene ring. The intramolecular C-H···O hydrogen bond results in the formation of a five-membered ring B (O2/N1/C5/C6/H5A), having envelope conformation with O2 atom displaced by 0.278 (3) Å from the plane of the other ring atoms.

In the crystal structure, intermolecular N-H···O and C-H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure. The π-π contact between the benzene rings, Cg1—Cg1^{i} [symmetry code: (i) -x, -y, 1 - z, where Cg1 is centroid of the ring A (C1-C6)] may further stabilize the structure, with centroid-centroid distance of 3.803 (3) Å.

S2. Experimental

For the preparation of the title compound, 4-chloro-3-nitrobenzoic acid (60.3 g, 0.32 mol) was suspended in thionyl chloride (180 ml) and heated at reflux for 5 h, then concentrated in vacuum as far as possible, the oily substance obtained. Added ice ammonia water (300 ml) to the oil, cooling to room temperature, a precipitate formed, which was collected by filtration and washed with water. Pure title compound was obtained by crystallizing from methanol (Sun *et al.*, 2006). Crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

S3. Refinement

H atoms were positioned geometrically, with N-H = 0.86 (for NH_2) and C-H = 0.93 Å for aromatic H and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

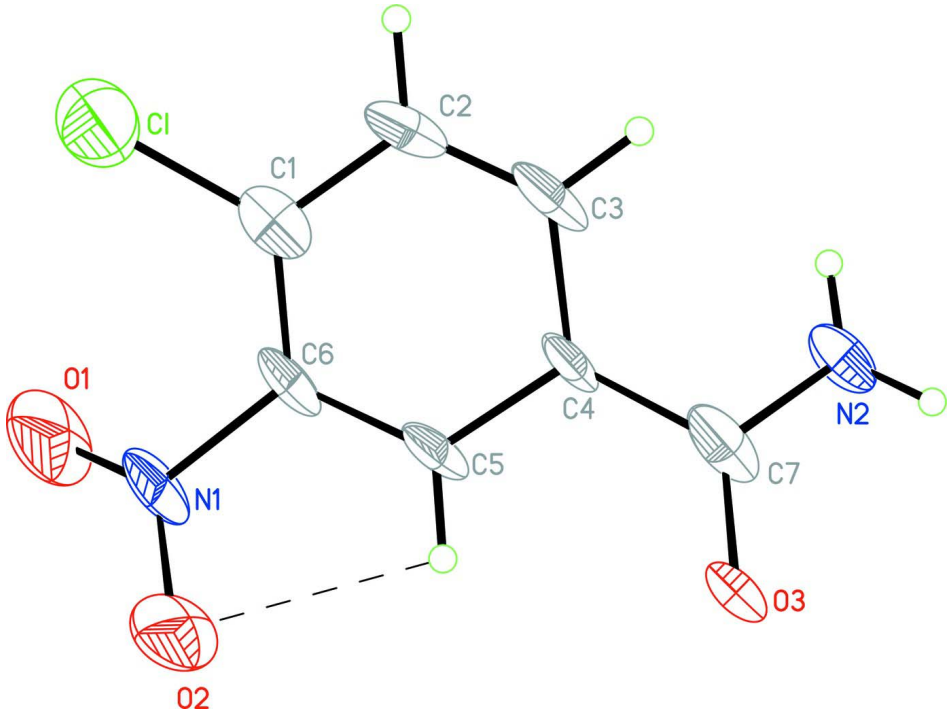

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bond is shown as dashed line.

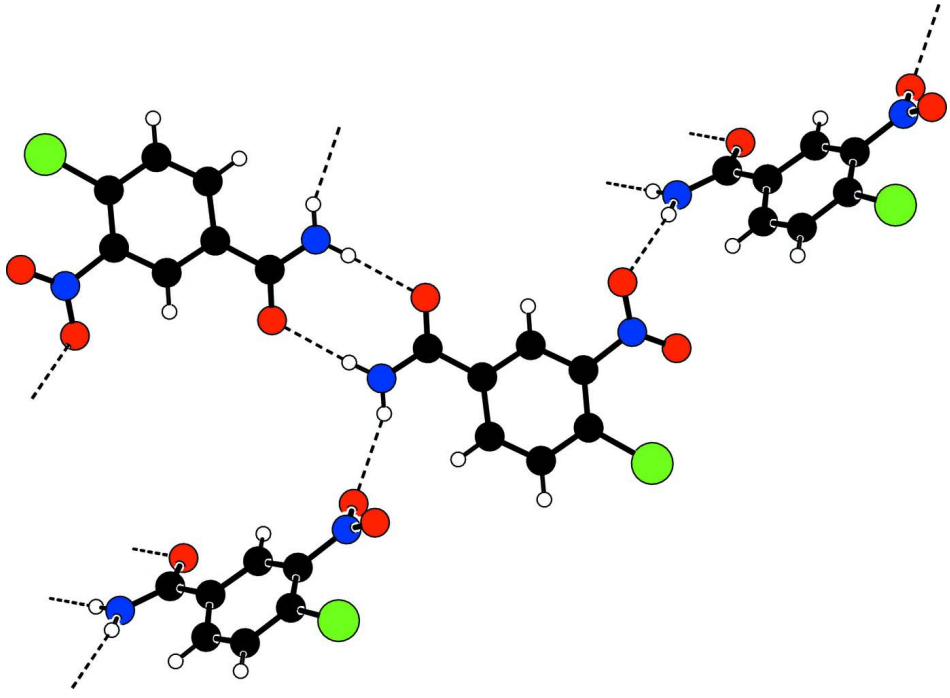

Figure 2

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

4-Chloro-3-nitrobenzamide

Crystal data

$C_7H_5ClN_2O_3$
$M_r = 200.58$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.8490$ (18) Å
$b = 7.5470$ (15) Å
$c = 12.374$ (3) Å
$\beta = 101.18$ (3)°
$V = 810.7$ (3) Å^3
$Z = 4$
$F(000) = 408$
$D_x = 1.643$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.44$ mm^{-1}
$T = 294$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.879$, $T_{max} = 0.957$
1555 measured reflections
1459 independent reflections
1085 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.6°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 14$
3 standard reflections every 120 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.198$
$S = 1.01$
1459 reflections
112 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 4.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ 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}
Cl	0.65545 (17)	0.0416 (2)	0.36001 (13)	0.0618 (5)
O1	0.7053 (5)	0.1130 (7)	0.5888 (4)	0.0749 (14)
O2	0.8610 (5)	0.3068 (6)	0.6677 (3)	0.0554 (11)
O3	1.3178 (4)	0.4669 (5)	0.5500 (2)	0.0390 (9)

N1	0.8103 (5)	0.2076 (5)	0.5896 (3)	0.0385 (10)
N2	1.3809 (5)	0.3507 (7)	0.3985 (4)	0.0502 (12)
H2B	1.4701	0.4002	0.4120	0.060*
H2C	1.3546	0.2856	0.3409	0.060*
C1	0.8373 (6)	0.1359 (7)	0.3967 (4)	0.0378 (11)
C2	0.9221 (6)	0.1454 (7)	0.3143 (4)	0.0401 (12)
H2A	0.8819	0.1004	0.2448	0.048*
C3	1.0637 (6)	0.2201 (7)	0.3346 (4)	0.0424 (12)
H3A	1.1188	0.2260	0.2779	0.051*
C4	1.1312 (5)	0.2902 (6)	0.4397 (3)	0.0295 (10)
C5	1.0412 (5)	0.2840 (6)	0.5196 (3)	0.0316 (10)
H5A	1.0790	0.3319	0.5888	0.038*
C6	0.8958 (5)	0.2076 (6)	0.4985 (4)	0.0308 (10)
C7	1.2837 (6)	0.3746 (7)	0.4668 (4)	0.0435 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0584 (9)	0.0679 (10)	0.0626 (10)	−0.0058 (7)	0.0208 (7)	−0.0114 (8)
O1	0.079 (3)	0.088 (3)	0.074 (3)	−0.027 (3)	0.057 (3)	−0.007 (3)
O2	0.072 (3)	0.068 (3)	0.035 (2)	−0.016 (2)	0.0311 (18)	−0.007 (2)
O3	0.0454 (18)	0.052 (2)	0.0267 (16)	−0.0091 (16)	0.0254 (14)	−0.0091 (15)
N1	0.054 (2)	0.038 (2)	0.036 (2)	0.000 (2)	0.0375 (19)	0.0044 (19)
N2	0.051 (2)	0.070 (3)	0.042 (2)	−0.007 (2)	0.038 (2)	−0.018 (2)
C1	0.049 (3)	0.035 (3)	0.035 (2)	0.008 (2)	0.020 (2)	0.004 (2)
C2	0.063 (3)	0.042 (3)	0.019 (2)	0.001 (2)	0.018 (2)	−0.004 (2)
C3	0.068 (3)	0.044 (3)	0.024 (2)	0.002 (2)	0.031 (2)	0.002 (2)
C4	0.043 (2)	0.030 (2)	0.022 (2)	−0.0015 (19)	0.0245 (18)	−0.0009 (18)
C5	0.050 (3)	0.032 (2)	0.020 (2)	0.002 (2)	0.0234 (18)	−0.0002 (18)
C6	0.046 (2)	0.028 (2)	0.027 (2)	0.0066 (19)	0.0275 (19)	0.0056 (18)
C7	0.061 (2)	0.038 (2)	0.038 (2)	0.009 (19)	0.034 (19)	0.006 (18)

Geometric parameters (Å, º)

Cl—C1	1.737 (5)	C1—C6	1.377 (7)
O3—C7	1.231 (6)	C2—C3	1.352 (7)
N1—O1	1.170 (6)	C2—H2A	0.9300
N1—O2	1.236 (5)	C3—C4	1.424 (7)
N1—C6	1.474 (5)	C3—H3A	0.9300
N2—C7	1.329 (6)	C4—C5	1.386 (6)
N2—H2B	0.8600	C4—C7	1.471 (7)
N2—H2C	0.8600	C5—C6	1.388 (7)
C1—C2	1.380 (6)	C5—H5A	0.9300
O1—N1—O2	122.9 (4)	C4—C3—H3A	118.9
O1—N1—C6	121.2 (4)	C3—C4—C7	124.9 (4)
O2—N1—C6	115.8 (4)	C5—C4—C3	116.2 (4)
C7—N2—H2B	120.0	C5—C4—C7	118.8 (4)

C7—N2—H2C	120.0	C4—C5—C6	121.3 (4)
H2B—N2—H2C	120.0	C4—C5—H5A	119.3
C2—C1—Cl	115.9 (4)	C6—C5—H5A	119.3
C6—C1—Cl	124.4 (4)	C1—C6—C5	120.4 (4)
C6—C1—C2	119.6 (5)	C1—C6—N1	122.8 (4)
C1—C2—H2A	119.9	C5—C6—N1	116.8 (4)
C3—C2—C1	120.1 (5)	O3—C7—N2	121.7 (5)
C3—C2—H2A	119.9	O3—C7—C4	120.0 (4)
C2—C3—C4	122.3 (4)	N2—C7—C4	118.3 (5)
C2—C3—H3A	118.9		

O1—N1—C6—C1	17.6 (7)	C2—C3—C4—C5	−2.5 (7)
O2—N1—C6—C1	−165.6 (5)	C2—C3—C4—C7	−179.3 (5)
O1—N1—C6—C5	−162.2 (5)	C3—C4—C5—C6	2.3 (7)
O2—N1—C6—C5	14.6 (6)	C7—C4—C5—C6	179.3 (4)
C6—C1—C2—C3	2.0 (8)	C5—C4—C7—O3	−13.8 (7)
Cl—C1—C2—C3	178.6 (4)	C3—C4—C7—O3	163.0 (5)
C2—C1—C6—C5	−2.2 (7)	C5—C4—C7—N2	167.3 (5)
Cl—C1—C6—C5	−178.5 (4)	C3—C4—C7—N2	−15.9 (8)
C2—C1—C6—N1	178.1 (4)	C4—C5—C6—C1	0.0 (7)
Cl—C1—C6—N1	1.8 (7)	C4—C5—C6—N1	179.7 (4)
C1—C2—C3—C4	0.4 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O3^{i}	0.86	2.10	2.958 (6)	177
N2—H2*C*···O2^{ii}	0.86	2.26	3.067 (6)	155
C2—H2*A*···O3^{iii}	0.93	2.42	3.331 (6)	166
C5—H5*A*···O2	0.93	2.33	2.658 (6)	100

Symmetry codes: (i) −*x*+3, −*y*+1, −*z*+1; (ii) *x*+1/2, −*y*+1/2, *z*−1/2; (iii) *x*−1/2, −*y*+1/2, *z*−1/2.