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2-Amino-3-chloro-5-nitrobenzamide

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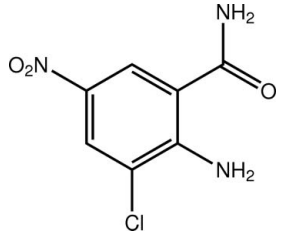
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.063; wR factor = 0.180; data-to-parameter ratio = 13.9.

The amide group in the title compound, $C_7H_6ClN_3O_3$, is significantly twisted out of the plane of the benzene ring [C—C—C—O = 34.2 (5)°] whereas the nitro group is almost co-planar [O—N—C—C = 4.0 (5)°] with the ring. Intramolecular N—H···O and N—H···Cl hydrogen bonds occur. In the crystal, the molecules are linked by N—H···O hydrogen bonds, generating layers propagating in the *ab* plane.

Related literature

For crystal engineering studies on related molecules, see: Wardell & Tiekink (2011).



Experimental

Crystal data

$C_7H_6ClN_3O_3$
M_r = 215.60
Triclinic, $P\overline{1}$
a = 4.891 (9) Å
b = 6.363 (13) Å
c = 14.61 (3) Å
α = 83.54 (11)°
β = 82.37 (11)°
γ = 73.64 (9)°
V = 431.1 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.43 mm^{-1}
T = 100 K
0.18 × 0.08 × 0.01 mm

Data collection

Rigaku Saturn724+ diffractometer
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2011)
T_{min} = 0.826, T_{max} = 1.000
3828 measured reflections
1936 independent reflections
1104 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.180
S = 0.95
1936 reflections
139 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H3n···O1	0.88 (3)	2.07 (2)	2.755 (7)	134 (3)
N2—H4n···Cl1	0.88 (2)	2.51 (3)	2.970 (7)	113 (3)
N1—H1n···O1^{i}	0.89 (3)	2.40 (4)	3.148 (8)	143 (3)
N1—H1n···O3^{ii}	0.89 (3)	2.55 (3)	3.130 (8)	124 (3)
N1—H2n···O1^{iii}	0.88 (2)	2.05 (3)	2.881 (7)	158 (4)
N2—H3n···O3^{iv}	0.88 (3)	2.44 (4)	3.076 (8)	130 (3)
N2—H4n···O2^{iv}	0.88 (2)	2.46 (4)	3.003 (8)	121 (3)

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2011); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6665).

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supporting information

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S1. Comment

The crystal structure determination on the impurity found from the recrystallization of a commercially available title compound (I) was performed as a part of a programme of crystal engineering studies with small molecule acids with amine and nitro substituents (Wardell & Tiekink, 2011).

In (I), Fig. 1, the amide group is twisted out of the plane of the benzene ring to which it is connected as seen in the value of the C2—C1—C7—O1 torsion angle of 34.2 (5)°. By contrast, the nitro group is co-planar with the ring with the O3—N3—C5—C6 torsion angle being 4.0 (5)°. Both amine-H atoms form intramolecular hydrogen bonds, one to the carbonyl-O and the other the chloride substituent, Table 1. The amine-H atoms also form intermolecular interactions with each connected to a nitro-O of the same molecule for form a six-membered $\{\cdots HNH\cdots ONO\}_2$ synthon. Pairs of amide groups self-associate *via* the familiar eight-membered centrosymmetric $\{\cdots HNCO\}_2$ synthon with this amide-H atom also connected to a translationally related amide-O atom. The second amide-H forms a hydrogen bond to a nitro-O3 atom. Thus, three of the N—H atoms form hydrogen bonds and two of the O donor atoms are bifurcated. This observation accounts for the deviations from linearity of the hydrogen bonds, Table 1. The hydrogen bonding scheme leads to the formation of layers in the *ab* plane. The layers stack along the *c* axis with no specific intermolecular interactions between them, Fig. 2.

S2. Experimental

The title compound was present as an impurity in a commercial batch of 2-amino-3-chloro-5-nitrobenzoic acid. It was isolated as extremely thin yellow plates from an ethanolic solution of the commercial 2-amino-3-chloro-5-nitrobenzoic acid and sodium hydroxide. IR: 3429 (*s*), 3325(*s*) and 3123(br) [NH], 1630–1586 (s, br, with maxima at 1629, 1607 and 1586) [CO], 1501(*s*) and 1317 (*s*) [NO_2].

S3. Refinement

The C-bound H atoms were geometrically placed (C—H = 0.95 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). The N-bound H-atoms were located in a difference Fourier map and refined with an N—H restraint of 0.88±0.01 Å, and with U_{iso}(H) = 1.2U_{eq}(N).

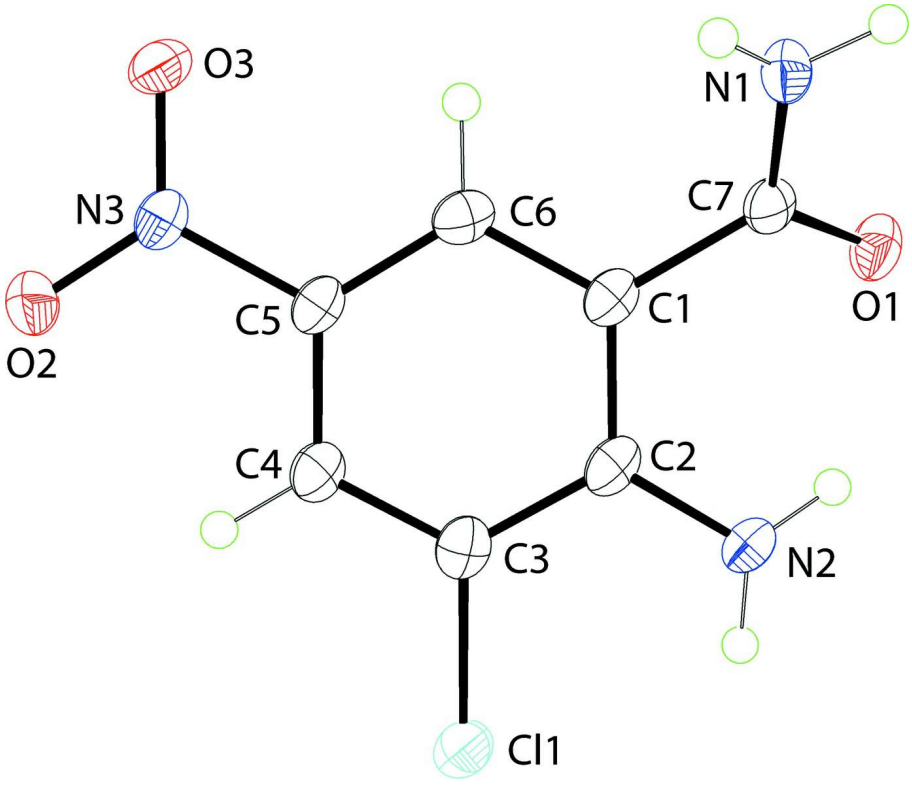

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

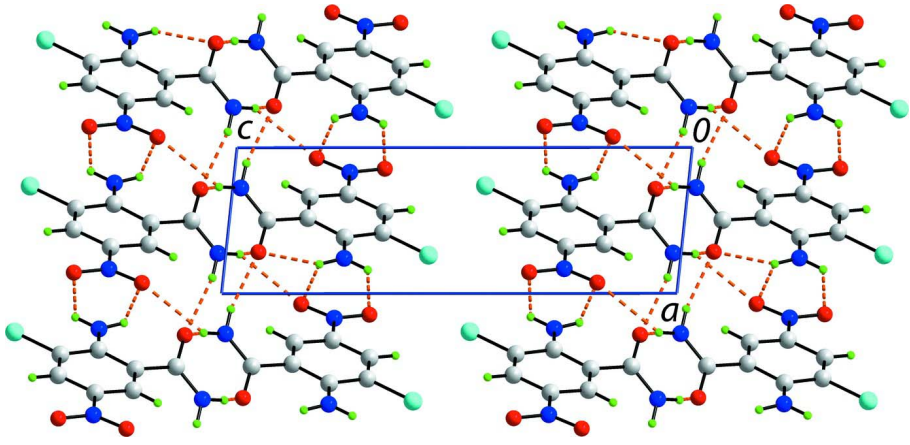

Figure 2

A view in projection down the *b* axis of the packing of supramolecular layers in (I). The N—H···O hydrogen bonds are shown as orange dashed lines.

2-Amino-3-chloro-5-nitrobenzamide

Crystal data

$C_7H_6ClN_3O_3$
$M_r = 215.60$
Triclinic, $P\overline{1}$

Hall symbol: -P 1
$a = 4.891$ (9) Å
$b = 6.363$ (13) Å

c = 14.61 (3) Å
α = 83.54 (11)°
β = 82.37 (11)°
γ = 73.64 (9)°
V = 431.1 (15) Å^3
Z = 2
F(000) = 220
D_x = 1.661 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1104 reflections
θ = 2.8–30.7°
μ = 0.43 mm^{-1}
T = 100 K
Plate, yellow
0.18 × 0.08 × 0.01 mm

Data collection

Rigaku Saturn724+ diffractometer
Radiation source: Rotating Anode
Confocal monochromator
Detector resolution: 28.5714 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2011)
T_{min} = 0.826, T_{max} = 1.000

3828 measured reflections
1936 independent reflections
1104 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 27.5°, θ_{min} = 2.8°
h = −4→6
k = −7→8
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.180
S = 0.95
1936 reflections
139 parameters
4 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0912P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
Cl1	0.2620 (2)	0.75410 (16)	0.44557 (7)	0.0290 (3)
O1	0.2845 (5)	0.6916 (4)	0.07122 (19)	0.0270 (7)
O2	0.8552 (6)	1.3190 (4)	0.32947 (19)	0.0265 (7)
O3	0.9263 (5)	1.3326 (4)	0.17747 (18)	0.0258 (7)
N1	0.7241 (7)	0.7345 (5)	0.0176 (2)	0.0224 (7)
H1N	0.731 (8)	0.659 (6)	−0.0305 (18)	0.027*
H2N	0.892 (4)	0.753 (6)	0.024 (3)	0.027*
N2	0.2414 (7)	0.6089 (5)	0.2610 (2)	0.0230 (7)

H3N	0.201 (8)	0.579 (6)	0.2079 (15)	0.028*
H4N	0.162 (8)	0.570 (6)	0.3155 (14)	0.028*
N3	0.8307 (6)	1.2566 (5)	0.2533 (2)	0.0203 (7)
C1	0.5172 (7)	0.8433 (6)	0.1718 (3)	0.0198 (8)
C2	0.3850 (8)	0.7644 (6)	0.2572 (3)	0.0218 (8)
C3	0.4161 (8)	0.8510 (6)	0.3398 (3)	0.0230 (9)
C4	0.5587 (8)	1.0104 (6)	0.3399 (3)	0.0224 (8)
H4	0.5744	1.0662	0.3961	0.027*
C5	0.6798 (7)	1.0873 (6)	0.2546 (3)	0.0197 (8)
C6	0.6623 (8)	1.0049 (6)	0.1714 (3)	0.0221 (8)
H6	0.7485	1.0581	0.1148	0.026*
C7	0.5010 (8)	0.7488 (6)	0.0829 (3)	0.0219 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0357 (6)	0.0244 (6)	0.0280 (5)	−0.0122 (4)	0.0008 (4)	−0.0015 (4)
O1	0.0219 (14)	0.0255 (15)	0.0363 (16)	−0.0106 (12)	0.0018 (12)	−0.0093 (12)
O2	0.0323 (15)	0.0245 (15)	0.0253 (15)	−0.0098 (12)	−0.0041 (12)	−0.0064 (12)
O3	0.0286 (15)	0.0220 (15)	0.0270 (15)	−0.0104 (12)	0.0033 (12)	−0.0004 (12)
N1	0.0213 (16)	0.0209 (18)	0.0262 (17)	−0.0059 (14)	−0.0019 (14)	−0.0071 (14)
N2	0.0253 (17)	0.0171 (17)	0.0272 (18)	−0.0077 (14)	−0.0025 (15)	0.0006 (14)
N3	0.0173 (15)	0.0145 (16)	0.0282 (17)	−0.0025 (12)	−0.0022 (13)	−0.0027 (13)
C1	0.0157 (17)	0.0131 (18)	0.028 (2)	−0.0002 (14)	−0.0007 (15)	−0.0016 (15)
C2	0.0187 (18)	0.0106 (18)	0.032 (2)	0.0006 (14)	0.0001 (16)	−0.0020 (15)
C3	0.0200 (18)	0.0158 (19)	0.031 (2)	0.0000 (15)	−0.0067 (16)	−0.0017 (16)
C4	0.0234 (19)	0.0141 (18)	0.028 (2)	−0.0018 (15)	−0.0011 (16)	−0.0040 (16)
C5	0.0162 (17)	0.0134 (18)	0.028 (2)	−0.0022 (14)	−0.0029 (15)	−0.0008 (15)
C6	0.0216 (18)	0.0149 (19)	0.026 (2)	−0.0007 (15)	0.0015 (16)	0.0003 (15)
C7	0.0241 (19)	0.0147 (18)	0.025 (2)	−0.0037 (15)	0.0018 (16)	−0.0041 (15)

Geometric parameters (Å, º)

Cl1—C3	1.753 (5)	N3—C5	1.464 (5)
O1—C7	1.250 (5)	C1—C6	1.403 (5)
O2—N3	1.254 (4)	C1—C2	1.433 (5)
O3—N3	1.244 (4)	C1—C7	1.511 (6)
N1—C7	1.340 (5)	C2—C3	1.425 (6)
N1—H1N	0.887 (10)	C3—C4	1.383 (6)
N1—H2N	0.881 (10)	C4—C5	1.406 (6)
N2—C2	1.358 (5)	C4—H4	0.9500
N2—H3N	0.881 (10)	C5—C6	1.398 (6)
N2—H4N	0.881 (10)	C6—H6	0.9500
C7—N1—H1N	118 (3)	C4—C3—C2	122.8 (4)
C7—N1—H2N	127 (3)	C4—C3—Cl1	118.7 (3)
H1N—N1—H2N	112 (4)	C2—C3—Cl1	118.4 (3)
C2—N2—H3N	117 (3)	C3—C4—C5	118.1 (4)

C2—N2—H4N	118 (3)	C3—C4—H4	121.0
H3N—N2—H4N	124 (4)	C5—C4—H4	121.0
O3—N3—O2	123.2 (3)	C6—C5—C4	121.7 (4)
O3—N3—C5	119.0 (3)	C6—C5—N3	119.4 (3)
O2—N3—C5	117.8 (3)	C4—C5—N3	118.9 (3)
C6—C1—C2	120.0 (4)	C5—C6—C1	119.9 (4)
C6—C1—C7	120.7 (3)	C5—C6—H6	120.0
C2—C1—C7	119.3 (3)	C1—C6—H6	120.0
N2—C2—C3	120.4 (4)	O1—C7—N1	122.2 (4)
N2—C2—C1	122.2 (4)	O1—C7—C1	120.6 (3)
C3—C2—C1	117.4 (4)	N1—C7—C1	117.2 (3)
C6—C1—C2—N2	179.8 (3)	O3—N3—C5—C6	4.0 (5)
C7—C1—C2—N2	−1.0 (5)	O2—N3—C5—C6	−176.4 (3)
C6—C1—C2—C3	−2.0 (5)	O3—N3—C5—C4	−176.6 (3)
C7—C1—C2—C3	177.3 (3)	O2—N3—C5—C4	2.9 (5)
N2—C2—C3—C4	−179.6 (3)	C4—C5—C6—C1	1.0 (5)
C1—C2—C3—C4	2.1 (5)	N3—C5—C6—C1	−179.7 (3)
N2—C2—C3—Cl1	−0.6 (5)	C2—C1—C6—C5	0.5 (5)
C1—C2—C3—Cl1	−178.9 (3)	C7—C1—C6—C5	−178.7 (3)
C2—C3—C4—C5	−0.7 (6)	C6—C1—C7—O1	−146.6 (4)
Cl1—C3—C4—C5	−179.7 (3)	C2—C1—C7—O1	34.2 (5)
C3—C4—C5—C6	−0.9 (5)	C6—C1—C7—N1	31.9 (5)
C3—C4—C5—N3	179.8 (3)	C2—C1—C7—N1	−147.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H3n···O1	0.88 (3)	2.07 (2)	2.755 (7)	134 (3)
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N1—H1n···O1^{i}	0.89 (3)	2.40 (4)	3.148 (8)	143 (3)
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