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N-(4-Chloro-3-nitrophenyl)succinamic acid

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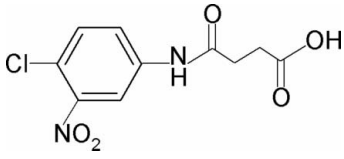
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.065; wR factor = 0.169; data-to-parameter ratio = 13.6.

In the title compound, $C_{10}H_9ClN_2O_5$, the nitro group is significantly twisted out of the plane of the benzene ring to which it is attached [dihedral angle = 27.4 (6)°]. In the crystal, molecules are linked into centrosymmetric dimers *via* pairs of O—H···O hydrogen bonds. These dimers are further linked by N—H···O hydrogen bonds into double chains running along the *a* axis.

Related literature

For our studies on the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Gowda *et al.* (2000); Chaithanya *et al.* (2012), on *N*-(aryl)-methanesulfonamides, see: Gowda *et al.* (2007), on *N*-chloroarylamides, see: Gowda *et al.* (2003); Jyothi & Gowda (2004) and on *N*-bromoarylsulfonamides, see: Usha & Gowda (2006).



Experimental

Crystal data

$C_{10}H_9ClN_2O_5$
M_r = 272.64
Monoclinic, $P2_1/n$
a = 4.8089 (8) Å
b = 10.278 (1) Å
c = 23.062 (3) Å
β = 90.69 (2)°
V = 1139.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 293 K
0.44 × 0.12 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.861, T_{max} = 0.966
4342 measured reflections
2305 independent reflections
1601 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.169
S = 1.05
2305 reflections
169 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 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}	0.85 (2)	2.28 (3)	3.006 (3)	144 (3)
O3—H3*O*···O2^{ii}	0.83 (2)	1.84 (2)	2.667 (3)	176 (4)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5832).

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supplementary materials

Acta Cryst. (2012). E**68**, o951 [doi:10.1107/S1600536812008720]

N-(4-Chloro-3-nitrophenyl)succinamic acid

U. Chaithanya, Sabine Foro and B. Thimme Gowda

Comment

As part of our studies on the substituent effects on the structures and other aspects of *N*-(aryl)-amides (Gowda *et al.*, 2000; Chaithanya *et al.*, 2012), *N*-(aryl)-methanesulfonamides (Gowda *et al.*, 2007); *N*-chloroarylsulfonamides (Gowda *et al.*, 2003; Jyothi & Gowda, 2004) and *N*-bromoaryl- sulfonamides (Usha & Gowda, 2006), in the present work, the crystal structure of *N*-(4-Chloro-3-nitrophenyl)succinamic acid has been determined (Fig. 1). The conformations of the N —H and the C=O bonds in the amide segment are *anti* to each other. But the N—H bond is *syn* to the *meta*–nitro group. The conformations of the amide C═O and the carboxyl C═O of the acid segment are *anti* to each other and both are *anti* to the H atoms on the adjacent –CH_2 groups. Furthermore, the C═O and O—H bonds of the acid group are in *syn* position to each other, in contrast to the *anti* positions observed in *N*-(4-Chloro-3-nitro- phenyl)maleamic acid (I) (Chaithanya *et al.*, 2012).

The dihedral angle between the phenyl ring and the amide group in the title compound is 31.8 (2)°, compared to the value of 11.5 (3)° in (I).

In the structure, the O—H···O and N—H···O intermolecular hydrogen bonds link the molecules into double chains running along the a axis (Table 1, Fig. 2).

Experimental

Succinic anhydride (0.025 mol) in toluene (25 ml) was treated dropwise with 4-chloro-3-nitroaniline (0.025 mol) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about 30 min and set aside for an additional 30 min at room temperature for the completion of reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 4-chloro-3-nitroaniline. The resultant solid *N*-(4-Chloro-3-nitrophenyl)succinamic acid was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. It was recrystallized to constant melting point from ethanol. The purity of the compound was checked and characterized by its infrared spectra.

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

Refinement

All H atoms were located in a difference map Those bonded to C H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å and methylene C—H = 0.97 Å. The coordinates of the H atoms bonded to N and O were refined with the N—H and O—H distance restrained to 0.86 (2) Å and 0.82 (2)Å, respectively. All H atoms were refined with isotropic displacement parameters set at 1.2 U_{eq} of the parent atom.

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); 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: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

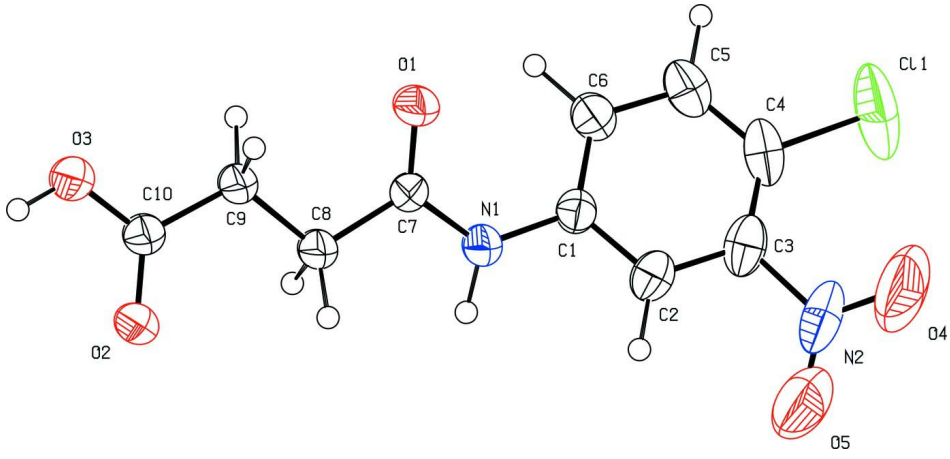

Figure 1

Molecular structure of the title compound, showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

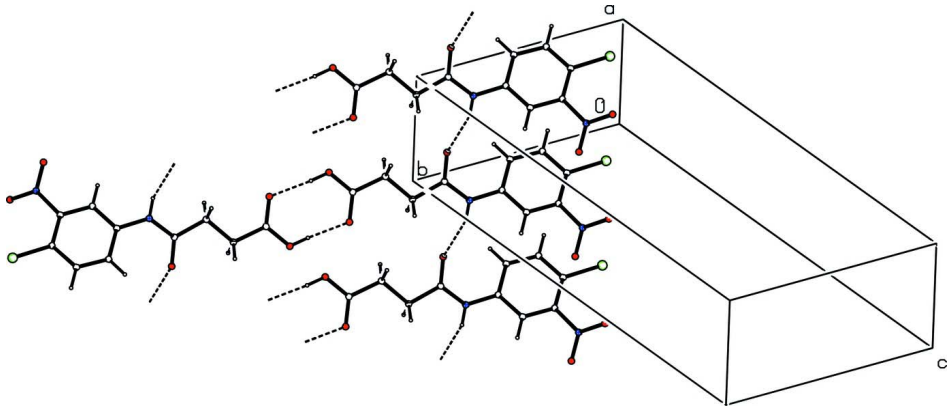

Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(4-Chloro-3-nitrophenyl)succinamic acid

Crystal data

$C_{10}H_9ClN_2O_5$
$M_r = 272.64$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.8089$ (8) Å
$b = 10.278$ (1) Å
$c = 23.062$ (3) Å
$\beta = 90.69$ (2)°
$V = 1139.8$ (3) Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.589$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1280 reflections
$\theta = 2.6$–27.7°
$\mu = 0.35$ mm^{-1}

T = 293 K
Rod, colourless

0.44 × 0.12 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω and phi scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.861, T_{max} = 0.966

4342 measured reflections
2305 independent reflections
1601 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 26.4°, θ_{min} = 2.7°
h = −3→6
k = −12→11
l = −28→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.169
S = 1.05
2305 reflections
169 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 1.4622P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.011
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}
C1	0.0737 (6)	0.7722 (3)	0.13397 (13)	0.0366 (7)
C2	−0.0598 (7)	0.7250 (3)	0.18230 (14)	0.0438 (8)
H2	−0.1913	0.7759	0.2009	0.053*
C3	0.0015 (8)	0.6024 (3)	0.20300 (14)	0.0503 (9)
C4	0.1914 (9)	0.5237 (3)	0.17540 (17)	0.0574 (10)
C5	0.3217 (8)	0.5714 (3)	0.12722 (17)	0.0556 (9)
H5	0.4498	0.5194	0.1082	0.067*
C6	0.2671 (7)	0.6949 (3)	0.10641 (14)	0.0451 (8)
H6	0.3597	0.7258	0.0740	0.054*
C7	0.1739 (6)	0.9830 (3)	0.08795 (13)	0.0355 (7)
C8	0.0419 (6)	1.1125 (3)	0.07321 (15)	0.0430 (8)

H8A	−0.1226	1.0980	0.0493	0.052*
H8B	−0.0156	1.1550	0.1087	0.052*
C9	0.2393 (6)	1.1998 (3)	0.04144 (15)	0.0433 (8)
H9A	0.4022	1.2140	0.0659	0.052*
H9B	0.3000	1.1551	0.0068	0.052*
C10	0.1234 (6)	1.3291 (3)	0.02419 (14)	0.0389 (7)
N1	0.0022 (5)	0.8974 (3)	0.11358 (12)	0.0427 (7)
H1N	−0.163 (4)	0.922 (3)	0.1198 (15)	0.051*
N2	−0.1337 (9)	0.5654 (4)	0.25738 (15)	0.0712 (11)
O1	0.4145 (4)	0.9578 (2)	0.07676 (12)	0.0550 (7)
O2	−0.1033 (5)	1.3682 (2)	0.04216 (12)	0.0557 (7)
O3	0.2758 (5)	1.3960 (2)	−0.00944 (13)	0.0588 (7)
H3O	0.219 (8)	1.470 (2)	−0.0181 (18)	0.071*
O4	−0.0219 (9)	0.4891 (5)	0.28923 (18)	0.1385 (19)
O5	−0.3572 (10)	0.6136 (4)	0.26831 (16)	0.1106 (14)
Cl1	0.2654 (4)	0.36516 (11)	0.19545 (7)	0.1142 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0353 (16)	0.0357 (16)	0.0387 (16)	−0.0019 (13)	−0.0005 (13)	0.0035 (13)
C2	0.0455 (18)	0.0432 (18)	0.0428 (18)	−0.0068 (14)	0.0034 (14)	−0.0003 (14)
C3	0.066 (2)	0.047 (2)	0.0379 (17)	−0.0164 (18)	−0.0092 (16)	0.0070 (15)
C4	0.081 (3)	0.0361 (18)	0.055 (2)	−0.0019 (18)	−0.020 (2)	0.0122 (16)
C5	0.065 (2)	0.0406 (19)	0.061 (2)	0.0128 (17)	−0.0061 (18)	−0.0025 (17)
C6	0.0497 (19)	0.0431 (18)	0.0426 (18)	0.0047 (15)	0.0023 (14)	0.0036 (15)
C7	0.0315 (15)	0.0367 (16)	0.0382 (16)	0.0010 (12)	0.0023 (12)	0.0074 (13)
C8	0.0339 (16)	0.0375 (17)	0.058 (2)	0.0063 (13)	0.0102 (14)	0.0103 (15)
C9	0.0362 (16)	0.0367 (17)	0.057 (2)	0.0063 (13)	0.0090 (15)	0.0100 (15)
C10	0.0334 (16)	0.0382 (17)	0.0453 (18)	−0.0002 (13)	0.0020 (13)	0.0058 (14)
N1	0.0332 (13)	0.0367 (14)	0.0585 (17)	0.0061 (11)	0.0110 (12)	0.0114 (13)
N2	0.086 (3)	0.072 (2)	0.055 (2)	−0.028 (2)	−0.0038 (19)	0.0273 (19)
O1	0.0343 (12)	0.0480 (14)	0.0831 (18)	0.0081 (10)	0.0141 (11)	0.0201 (13)
O2	0.0425 (13)	0.0464 (14)	0.0786 (18)	0.0124 (10)	0.0180 (12)	0.0196 (12)
O3	0.0525 (15)	0.0407 (14)	0.0837 (19)	0.0100 (11)	0.0238 (13)	0.0218 (13)
O4	0.126 (3)	0.192 (5)	0.098 (3)	−0.007 (3)	0.001 (2)	0.094 (3)
O5	0.144 (4)	0.111 (3)	0.079 (2)	−0.002 (3)	0.042 (2)	0.026 (2)
Cl1	0.1907 (17)	0.0468 (6)	0.1045 (11)	0.0161 (8)	−0.0287 (10)	0.0271 (6)

Geometric parameters (Å, º)

C1—C2	1.381 (4)	C7—C8	1.511 (4)
C1—C6	1.384 (4)	C8—C9	1.503 (4)
C1—N1	1.411 (4)	C8—H8A	0.9700
C2—C3	1.378 (5)	C8—H8B	0.9700
C2—H2	0.9300	C9—C10	1.493 (4)
C3—C4	1.381 (6)	C9—H9A	0.9700
C3—N2	1.470 (5)	C9—H9B	0.9700
C4—C5	1.373 (5)	C10—O2	1.238 (4)
C4—Cl1	1.729 (4)	C10—O3	1.275 (4)

C5—C6	1.381 (5)	N1—H1N	0.847 (18)
C5—H5	0.9300	N2—O4	1.198 (5)
C6—H6	0.9300	N2—O5	1.213 (5)
C7—O1	1.216 (3)	O3—H3O	0.834 (19)
C7—N1	1.348 (4)		
C2—C1—C6	119.3 (3)	C9—C8—H8A	109.3
C2—C1—N1	118.4 (3)	C7—C8—H8A	109.3
C6—C1—N1	122.2 (3)	C9—C8—H8B	109.3
C3—C2—C1	120.1 (3)	C7—C8—H8B	109.3
C3—C2—H2	119.9	H8A—C8—H8B	107.9
C1—C2—H2	119.9	C10—C9—C8	115.2 (3)
C2—C3—C4	121.1 (3)	C10—C9—H9A	108.5
C2—C3—N2	115.9 (4)	C8—C9—H9A	108.5
C4—C3—N2	122.9 (3)	C10—C9—H9B	108.5
C5—C4—C3	118.3 (3)	C8—C9—H9B	108.5
C5—C4—Cl1	117.3 (3)	H9A—C9—H9B	107.5
C3—C4—Cl1	124.3 (3)	O2—C10—O3	122.9 (3)
C4—C5—C6	121.5 (4)	O2—C10—C9	121.8 (3)
C4—C5—H5	119.2	O3—C10—C9	115.3 (3)
C6—C5—H5	119.2	C7—N1—C1	126.4 (3)
C5—C6—C1	119.6 (3)	C7—N1—H1N	118 (2)
C5—C6—H6	120.2	C1—N1—H1N	116 (2)
C1—C6—H6	120.2	O4—N2—O5	122.2 (4)
O1—C7—N1	122.9 (3)	O4—N2—C3	119.5 (5)
O1—C7—C8	122.5 (3)	O5—N2—C3	118.3 (4)
N1—C7—C8	114.6 (2)	C10—O3—H3O	117 (3)
C9—C8—C7	111.7 (2)		
C6—C1—C2—C3	0.6 (5)	O1—C7—C8—C9	2.6 (5)
N1—C1—C2—C3	179.1 (3)	N1—C7—C8—C9	−176.4 (3)
C1—C2—C3—C4	−1.5 (5)	C7—C8—C9—C10	178.9 (3)
C1—C2—C3—N2	175.0 (3)	C8—C9—C10—O2	10.1 (5)
C2—C3—C4—C5	1.1 (5)	C8—C9—C10—O3	−170.8 (3)
N2—C3—C4—C5	−175.1 (3)	O1—C7—N1—C1	3.8 (5)
C2—C3—C4—Cl1	−175.8 (3)	C8—C7—N1—C1	−177.2 (3)
N2—C3—C4—Cl1	8.1 (5)	C2—C1—N1—C7	147.1 (3)
C3—C4—C5—C6	0.1 (6)	C6—C1—N1—C7	−34.5 (5)
Cl1—C4—C5—C6	177.2 (3)	C2—C3—N2—O4	−151.7 (4)
C4—C5—C6—C1	−1.0 (5)	C4—C3—N2—O4	24.7 (6)
C2—C1—C6—C5	0.6 (5)	C2—C3—N2—O5	28.7 (5)
N1—C1—C6—C5	−177.8 (3)	C4—C3—N2—O5	−155.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.85 (2)	2.28 (3)	3.006 (3)	144 (3)
O3—H3*O*···O2^{ii}	0.83 (2)	1.84 (2)	2.667 (3)	176 (4)

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