Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(3-Chlorophenyl)succinamic acid

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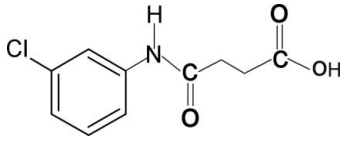
Received 7 March 2010; accepted 9 March 2010

Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.058; wR factor = 0.152; data-to-parameter ratio = 15.4.

In the title compound, $C_{10}H_{10}ClNO_3$, the N—H and C=O bonds in the amide segment are *trans* to each other. In the crystal structure, the molecules are linked into infinite chains through intermolecular N—H···O and O—H···O hydrogen bonds.

Related literature

For our study of the effect of ring and side-chain substitutions on the structures of anilides and for related structures, see: Gowda *et al.* (2009*a*,*b*; 2010); Jagannathan *et al.* (1994).



Experimental

Crystal data

$C_{10}H_{10}ClNO_3$
M_r = 227.64
Orthorhombic, *Pbca*
a = 10.0308 (8) Å
b = 11.1810 (9) Å
c = 19.036 (2) Å
V = 2135.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 299 K
0.24 × 0.20 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.922, T_{max} = 0.980
8200 measured reflections
2184 independent reflections
1137 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.152
S = 1.02
2184 reflections
142 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*O*···O1^{i}	0.82 (2)	1.92 (2)	2.693 (3)	158 (5)
N1—H1*N*···O2^{ii}	0.85 (2)	2.02 (2)	2.872 (4)	173 (3)

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

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*.

BSS thanks the University Grants Commission, Government of India, New Delhi, for the award of a research fellowship under its faculty improvement program.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5210).

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supplementary materials

Acta Cryst. (2010). E**66**, o842 [**doi:10.1107/S1600536810008949**]

N-(3-Chlorophenyl)succinamic acid

B. T. Gowda, S. Foro, B. S. Saraswathi and H. Fuess

Comment

As a part of studying the effect of ring and side chain substitutions on the structures of anilides (Gowda *et al.*, 2009*a,b*; 2010), the crystal structure of *N*-(3-chlorophenyl)succinamic acid (I) has been determined. The conformations of N—H and C=O bonds in the amide segment are *anti* to each other, similar to those observed in *N*-(2-chlorophenyl)succinamic acid (II)(Gowda *et al.*, 2009*b*) and *N*-(4-chlorophenyl)succinamic acid (III) (Gowda *et al.*, 2009*a*) and *N*-(3-methylphenyl)succinamic acid (IV)(Gowda *et al.*, 2010). But the conformation of the amide oxygen and the carbonyl oxygen of the acid segment are *syn* to each other, similar to that observed in (IV), but contrary contrary to the *anti* conformation observed in (II) and (III). Further, the conformation of both the C=O bonds are *anti* to the H atoms of their adjacent $–CH_2$ groups (Fig. 1) and the C=O and O—H bonds of the acid group are in *syn* position to each other, similar to that observed in (II), (III) and (IV).

The conformation of the amide hydrogen is *syn* to the *meta*- Cl group in the benzene ring, similar to that of the *ortho*-Cl in (II), but contrary to the *anti* conformation observed between the amide hydrogen and the *meta*-methyl group in (IV).

The N—H···O and O—H···O intermolecular hydrogen bonds pack the mpolecules into infinite chains in the structure (Table 1, Fig.2).

The packing of molecules involving dimeric hydrogen bonded association of each carboxyl group with a centrosymmetrically related neighbor has also been observed (Jagannathan *et al.*, 1994).

Experimental

The solution of succinic anhydride (0.01 mole) in toluene (25 ml) was treated dropwise with the solution of *m*-chloroaniline (0.01 mole) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about one h and set aside for an additional hour at room temperature for completion of the reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted *m*-chloroaniline. The resultant solid *N*-(3-chlorophenyl)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 by elemental analysis and characterized by its infrared and NMR spectra. The plate like colorless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

Refinement

The H atoms of the OH and NH group were located in a difference map and refined with a distance restraint of O—H = 0.82 (2) %A and N—H = 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model

with C—H = 0.93–0.97 Å. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.

Figures

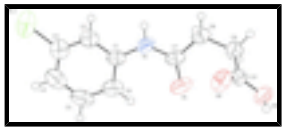

Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.

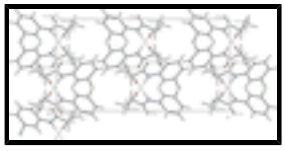

Fig. 2. Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

N-(3-Chlorophenyl)succinamic acid

Crystal data

$C_{10}H_{10}ClNO_3$	$F(000) = 944$
$M_r = 227.64$	$D_x = 1.416$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 2016 reflections
$a = 10.0308$ (8) Å	θ = 2.7–27.7°
$b = 11.1810$ (9) Å	μ = 0.34 mm^{-1}
$c = 19.036$ (2) Å	$T = 299$ K
$V = 2135.0$ (3) Å^3	Plate, colourless
$Z = 8$	0.24 × 0.20 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	2184 independent reflections
Radiation source: fine-focus sealed tube	1137 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.045$
Rotation method data acquisition using ω and φ scans.	$\theta_{max} = 26.4°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2009)	$h = -9 \rightarrow 12$
$T_{min} = 0.922$, $T_{max} = 0.980$	$k = -12 \rightarrow 13$
8200 measured reflections	$l = -22 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.058$	Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.152$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 1.1737P]$ where $P = (F_o^2 + 2F_c^2)/3$
2184 reflections	$(\Delta/\sigma)_{max} = 0.012$
142 parameters	$\Delta\rho_{max} = 0.30$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.39$ 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 esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
Cl1	0.25030 (11)	0.71276 (10)	0.20800 (6)	0.0858 (4)
O1	−0.0249 (2)	0.23883 (19)	0.02471 (13)	0.0655 (7)
O2	0.1845 (3)	0.0367 (2)	−0.03891 (14)	0.0673 (7)
O3	0.0091 (3)	−0.0318 (2)	−0.09646 (13)	0.0651 (7)
H3O	0.033 (5)	−0.097 (2)	−0.081 (2)	0.098*
N1	0.1200 (3)	0.3941 (2)	0.03276 (15)	0.0539 (8)
H1N	0.181 (3)	0.431 (3)	0.0101 (16)	0.065*
C1	0.0900 (3)	0.4445 (3)	0.09884 (18)	0.0484 (8)
C2	0.1706 (4)	0.5390 (3)	0.12010 (18)	0.0523 (9)
H2	0.2395	0.5654	0.0913	0.063*
C3	0.1482 (4)	0.5930 (3)	0.1835 (2)	0.0574 (10)
C4	0.0471 (5)	0.5568 (4)	0.2270 (2)	0.0714 (12)
H4	0.0329	0.5943	0.2700	0.086*
C5	−0.0325 (5)	0.4644 (4)	0.2058 (2)	0.0741 (12)
H5	−0.1015	0.4393	0.2349	0.089*
C6	−0.0129 (4)	0.4072 (3)	0.1420 (2)	0.0627 (10)
H6	−0.0682	0.3446	0.1284	0.075*
C7	0.0649 (3)	0.2997 (3)	−0.00050 (19)	0.0480 (8)
C8	0.1239 (3)	0.2756 (3)	−0.07174 (17)	0.0519 (9)
H8A	0.2183	0.2590	−0.0664	0.062*
H8B	0.1151	0.3468	−0.1004	0.062*
C9	0.0587 (4)	0.1716 (3)	−0.10939 (18)	0.0566 (9)
H9A	−0.0373	0.1824	−0.1086	0.068*

H9B	0.0870	0.1716	−0.1581	0.068*
C10	0.0920 (4)	0.0530 (3)	−0.07727 (17)	0.0444 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0739 (7)	0.0821 (8)	0.1014 (9)	0.0073 (6)	−0.0183 (7)	−0.0295 (6)
O1	0.0610 (15)	0.0445 (13)	0.091 (2)	−0.0112 (12)	0.0190 (14)	0.0046 (12)
O2	0.0650 (17)	0.0477 (14)	0.0894 (19)	0.0055 (13)	−0.0291 (16)	0.0059 (13)
O3	0.0739 (18)	0.0504 (14)	0.0712 (17)	−0.0174 (15)	−0.0159 (14)	0.0058 (13)
N1	0.0531 (18)	0.0452 (16)	0.063 (2)	−0.0146 (14)	0.0162 (15)	−0.0029 (14)
C1	0.049 (2)	0.0408 (18)	0.055 (2)	0.0040 (16)	0.0087 (18)	0.0059 (16)
C2	0.045 (2)	0.054 (2)	0.058 (2)	0.0041 (18)	0.0071 (17)	0.0025 (17)
C3	0.053 (2)	0.057 (2)	0.062 (2)	0.0130 (18)	−0.009 (2)	0.0002 (19)
C4	0.088 (3)	0.075 (3)	0.052 (3)	0.019 (3)	0.008 (2)	0.004 (2)
C5	0.083 (3)	0.074 (3)	0.066 (3)	0.007 (3)	0.032 (2)	0.018 (2)
C6	0.060 (2)	0.054 (2)	0.074 (3)	−0.0032 (19)	0.018 (2)	0.0106 (19)
C7	0.0447 (18)	0.0346 (16)	0.065 (2)	0.0046 (15)	0.0048 (19)	0.0118 (16)
C8	0.056 (2)	0.0356 (17)	0.064 (2)	0.0035 (16)	0.0013 (19)	0.0076 (16)
C9	0.065 (2)	0.0490 (19)	0.056 (2)	0.0029 (18)	−0.0134 (19)	0.0037 (17)
C10	0.047 (2)	0.0430 (19)	0.0430 (19)	0.0012 (16)	0.0024 (17)	−0.0039 (15)

Geometric parameters (Å, °)

Cl1—C3	1.749 (4)	C4—C5	1.366 (6)
O1—C7	1.226 (4)	C4—H4	0.9300
O2—C10	1.195 (4)	C5—C6	1.388 (5)
O3—C10	1.313 (4)	C5—H5	0.9300
O3—H3O	0.820 (19)	C6—H6	0.9300
N1—C7	1.349 (4)	C7—C8	1.504 (5)
N1—C1	1.411 (4)	C8—C9	1.515 (4)
N1—H1N	0.853 (18)	C8—H8A	0.9700
C1—C6	1.384 (5)	C8—H8B	0.9700
C1—C2	1.391 (5)	C9—C10	1.498 (4)
C2—C3	1.369 (5)	C9—H9A	0.9700
C2—H2	0.9300	C9—H9B	0.9700
C3—C4	1.371 (5)		
C10—O3—H3O	111 (3)	C1—C6—H6	120.4
C7—N1—C1	130.1 (3)	C5—C6—H6	120.4
C7—N1—H1N	116 (2)	O1—C7—N1	123.5 (3)
C1—N1—H1N	114 (2)	O1—C7—C8	122.8 (3)
C6—C1—C2	119.4 (3)	N1—C7—C8	113.7 (3)
C6—C1—N1	124.6 (3)	C7—C8—C9	113.2 (3)
C2—C1—N1	116.0 (3)	C7—C8—H8A	108.9
C3—C2—C1	119.8 (3)	C9—C8—H8A	108.9
C3—C2—H2	120.1	C7—C8—H8B	108.9
C1—C2—H2	120.1	C9—C8—H8B	108.9
C2—C3—C4	121.6 (4)	H8A—C8—H8B	107.7

C2—C3—Cl1	118.5 (3)	C10—C9—C8	112.9 (3)
C4—C3—Cl1	119.9 (3)	C10—C9—H9A	109.0
C5—C4—C3	118.5 (4)	C8—C9—H9A	109.0
C5—C4—H4	120.7	C10—C9—H9B	109.0
C3—C4—H4	120.7	C8—C9—H9B	109.0
C4—C5—C6	121.7 (4)	H9A—C9—H9B	107.8
C4—C5—H5	119.2	O2—C10—O3	123.5 (3)
C6—C5—H5	119.2	O2—C10—C9	123.9 (3)
C1—C6—C5	119.1 (4)	O3—C10—C9	112.6 (3)
C7—N1—C1—C6	−4.0 (6)	N1—C1—C6—C5	−179.8 (3)
C7—N1—C1—C2	176.7 (3)	C4—C5—C6—C1	0.2 (6)
C6—C1—C2—C3	0.6 (5)	C1—N1—C7—O1	−1.2 (5)
N1—C1—C2—C3	179.9 (3)	C1—N1—C7—C8	178.9 (3)
C1—C2—C3—C4	−0.4 (5)	O1—C7—C8—C9	1.7 (4)
C1—C2—C3—Cl1	−179.2 (3)	N1—C7—C8—C9	−178.4 (3)
C2—C3—C4—C5	0.0 (6)	C7—C8—C9—C10	−71.0 (4)
Cl1—C3—C4—C5	178.8 (3)	C8—C9—C10—O2	−18.4 (5)
C3—C4—C5—C6	0.1 (6)	C8—C9—C10—O3	162.3 (3)
C2—C1—C6—C5	−0.5 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···O1^{i}	0.82 (2)	1.92 (2)	2.693 (3)	158 (5)
N1—H1N···O2^{ii}	0.85 (2)	2.02 (2)	2.872 (4)	173 (3)

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

Fig. 1

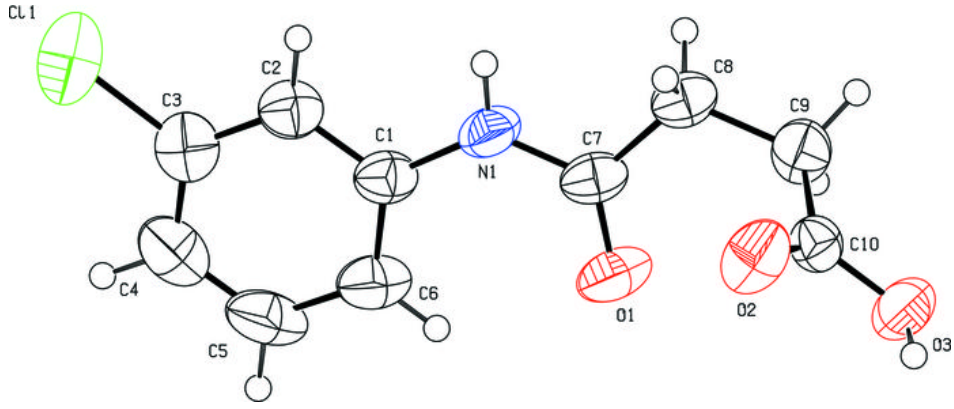

Fig. 2