organic compounds

Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2,5-Dichlorophenyl)succinamic acid

B. S. Saraswathi,[a] Sabine Foro[b] and B. Thimme Gowda[a]*

[a]Department of Chemistry, Mangalore University, Mangalagangotri 574 199, Mangalore, India, and [b]Institute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
Correspondence e-mail: gowdabt@yahoo.com

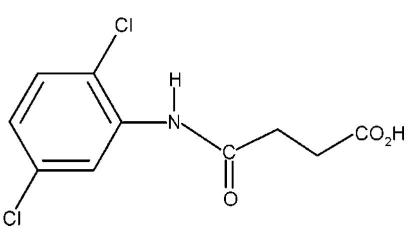
Received 5 July 2011; accepted 14 July 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.077; wR factor = 0.171; data-to-parameter ratio = 11.1.

In the title compound, $C_{10}H_9Cl_2NO_3$, the conformation of the N—H bond in the amide segment is *syn* with respect to the *ortho*-Cl atom and *anti* to the *meta*-Cl atom of the benzene ring. In the crystal, intermolecular O—H···O and N—H···O hydrogen bonds pack the molecules into two types of chains along the a and b axes, respectively, leading to an overall sheet structure. The acid group in the side chain is disordered and was refined using a split model with site-occupation factors of 0.60:0.40.

Related literature

For our studies of the effects of substituents on the structures and other aspects of *N*-(aryl)-amides, see: Bhat & Gowda (2000); Gowda *et al.* (2007); Saraswathi *et al.* (2011*a*,*b*), on *N*-(aryl)-methanesulfonamides, see: Jayalakshmi & Gowda (2004) and on *N*-chloro-arylsulfonamides, see: Gowda *et al.* (2003). For the modes of interlinking carboxylic acids by hydrogen bonds, see: Leiserowitz (1976). For the packing of molecules involving dimeric hydrogen-bonding associations of each carboxyl group with a centrosymmetrically related neighbor, see: Jagannathan *et al.* (1994).



Experimental

Crystal data

$C_{10}H_9Cl_2NO_3$
M_r = 262.08
Monoclinic, $P2_1/c$
a = 5.726 (1) Å
b = 4.787 (1) Å
c = 41.583 (6) Å
β = 91.93 (2)°
V = 1139.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 293 K
0.44 × 0.16 × 0.09 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.791, T_{max} = 0.951
3375 measured reflections
2046 independent reflections
1552 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.171
S = 1.14
2046 reflections
185 parameters
54 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.75 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
O2A—H2A···O3A^{i}	0.82	1.90	2.687 (15)	162
O2B—H2B···O3B^{i}	0.82	1.90	2.64 (2)	150
N1—H1N···O1^{ii}	0.85 (2)	2.07 (2)	2.901 (6)	167 (5)

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

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: VM2110).

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supporting information

Acta Cryst. (2011). E**67**, o2093 [doi:10.1107/S1600536811028297]

N-(2,5-Dichlorophenyl)succinamic acid

B. S. Saraswathi, Sabine Foro and B. Thimme Gowda

S1. Comment

The amide and sulfonamide moieties are important constituents of many biologically important compounds. As a part of our studies on the effects of substituents on the structures and other aspects of this class of compounds (Bhat & Gowda, 2000; Gowda *et al.*, 2003, 2007; Jayalakshmi & Gowda, 2004; Saraswathi *et al.*, 2011*a,b*), in the present work, the crystal structure of *N*-(2,5-dichlorophenyl)-succinamic acid (I) has been determined (Fig. 1). The conformation of the N—H bond in the amide segment is *syn* to the *ortho*–chloro atom and *anti* to the *meta*–chloro atom of the benzene ring, similar to the *syn* conformation observed between the amide hydrogen and the *ortho*- methyl group and *anti* conformation between the amide hydrogen and the *meta*-methyl group in the benzene ring of *N*-(2,5-dimethylphenyl)-succinamic acid monohydrate (II) (Saraswathi *et al.*, 2011*a*).

Further, the conformations of the amide oxygen and the carboxyl oxygen of the acid segment are *syn* to each other. But the conformation of the amide C═O is *anti* to the H atoms on the adjacent –CH_2 group, while the carboxyl C═O is *syn* to the H atoms on the adjacent –CH_2 group.

The C═O and O—H bonds of the acid group are in *syn* position to each other, similar to that observed in (II) and in *N*-(2,6-dichlorophenyl)-succinamic acid (Saraswathi *et al.*, 2011*b*).

The intermolecular O—H···O and N—H···O hydrogen bonds, along *a*- and *b*-axes, respectively, pack the molecules into infinite chains in the structure (Table 1, Fig.2).

The modes of interlinking carboxylic acids by hydrogen bonds is described elsewhere (Leiserowitz, 1976). 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).

S2. Experimental

The solution of succinic anhydride (0.01 mole) in toluene (25 ml) was treated dropwise with the solution of 2,5-dichloro-aniline (0.01 mole) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about one hour 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 2,5-dichloroaniline. The resultant title compound 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 and NMR spectra.

Needle like colorless single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and its position refined with N—H = 0.86 (2) Å. The other H atoms were positioned with idealized geometry using a riding model with the aromatic C—H = 0.93 Å, methylene C—H = 0.97 Å and O—H = 0.82 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

The atoms C9, C10, O2 and O3 are disordered and were refind using a split model. The corresponding site-occupation factors were fixed to 0.60:0.40 and their corresponding bond distances in the disordered groups were restrained to be equal. The U_{eq} of these atoms were restrained to approximate isotropic behavoir.

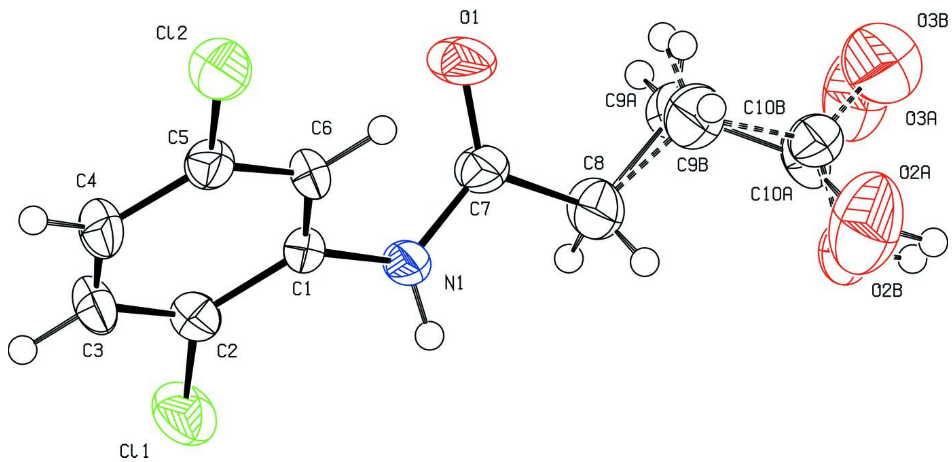

Figure 1

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

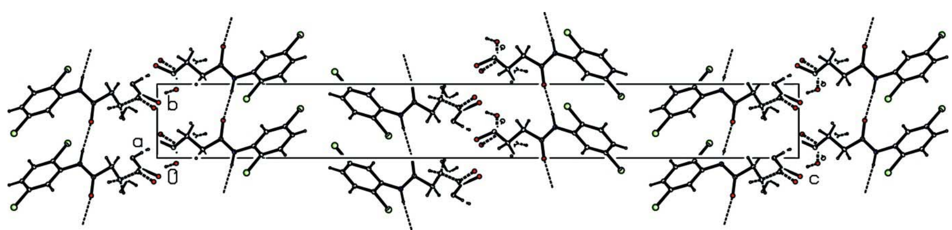

Figure 2

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

N-(2,5-Dichlorophenyl)succinamic acid

Crystal data

$C_{10}H_9Cl_2NO_3$
$M_r = 262.08$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.726$ (1) Å
$b = 4.787$ (1) Å
$c = 41.583$ (6) Å
$\beta = 91.93$ (2)°
$V = 1139.2$ (4) Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.528$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 987 reflections
$\theta = 2.9$–27.7°
$\mu = 0.56$ mm^{-1}
$T = 293$ K
Needle, colourless
0.44 × 0.16 × 0.09 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 scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{min} = 0.791$, $T_{max} = 0.951$
3375 measured reflections
2046 independent reflections
1552 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.9°$
$h = -5 \rightarrow 6$
$k = -5 \rightarrow 2$
$l = -50 \rightarrow 37$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.171$
$S = 1.14$
2046 reflections
185 parameters
54 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
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 4.544P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.048$
$\Delta\rho_{max} = 0.75$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ 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}	Occ. (<1)
Cl1	0.3416 (2)	−0.1995 (3)	0.13931 (4)	0.0513 (4)	
Cl2	0.8927 (3)	0.6671 (3)	0.22318 (3)	0.0460 (4)	
O1	0.8848 (9)	0.5091 (8)	0.10285 (10)	0.0599 (13)	
O2A	1.4182 (19)	−0.004 (3)	0.0382 (2)	0.088 (4)	0.60
H2A	1.4940	−0.0747	0.0239	0.106*	0.60
O2B	1.325 (3)	−0.072 (3)	0.0293 (4)	0.061 (4)	0.40
H2B	1.3972	−0.1171	0.0134	0.074*	0.40
O3A	1.259 (2)	0.248 (3)	−0.0021 (3)	0.082 (4)	0.60
O3B	1.357 (4)	0.322 (4)	0.0053 (5)	0.096 (7)	0.40
N1	0.7824 (8)	0.0816 (9)	0.12040 (10)	0.0342 (10)	
H1N	0.791 (9)	−0.091 (5)	0.1164 (13)	0.041*	
C1	0.6994 (8)	0.1621 (10)	0.15067 (11)	0.0296 (11)	
C2	0.4967 (9)	0.0438 (11)	0.16233 (12)	0.0342 (12)	
C3	0.4144 (9)	0.1202 (12)	0.19205 (13)	0.0402 (13)	
H3	0.2780	0.0402	0.1994	0.048*	

C4	0.5332 (9)	0.3132 (12)	0.21069 (13)	0.0412 (13)	
H4	0.4777	0.3662	0.2305	0.049*	
C5	0.7367 (9)	0.4276 (11)	0.19951 (12)	0.0334 (12)	
C6	0.8201 (8)	0.3543 (11)	0.17001 (11)	0.0321 (12)	
H6	0.9576	0.4337	0.1630	0.039*	
C7	0.8798 (10)	0.2569 (11)	0.09932 (13)	0.0369 (13)	
C8	0.9872 (12)	0.1158 (13)	0.07101 (14)	0.0516 (16)	
H8A	1.1120	−0.0047	0.0791	0.062*	
H8B	0.8695	−0.0031	0.0608	0.062*	
C9A	1.082 (2)	0.297 (3)	0.0464 (2)	0.044 (3)	0.60
H9A	1.1601	0.4541	0.0568	0.053*	0.60
H9B	0.9545	0.3688	0.0329	0.053*	0.60
C9B	1.172 (3)	0.293 (5)	0.0582 (5)	0.058 (5)	0.40
H9C	1.2859	0.3277	0.0756	0.069*	0.40
H9D	1.1028	0.4709	0.0523	0.069*	0.40
C10A	1.253 (3)	0.145 (3)	0.0256 (4)	0.057 (4)	0.60
C10B	1.302 (4)	0.187 (4)	0.0298 (5)	0.043 (6)	0.40

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0426 (8)	0.0500 (9)	0.0613 (9)	−0.0162 (7)	0.0020 (7)	−0.0020 (8)
Cl2	0.0553 (9)	0.0455 (8)	0.0376 (7)	−0.0051 (7)	0.0053 (6)	−0.0069 (7)
O1	0.106 (4)	0.026 (2)	0.050 (3)	−0.007 (2)	0.040 (2)	−0.0065 (19)
O2A	0.092 (7)	0.118 (8)	0.055 (5)	0.027 (6)	0.018 (5)	−0.009 (5)
O2B	0.068 (6)	0.056 (5)	0.062 (6)	0.002 (4)	0.031 (4)	−0.004 (4)
O3A	0.075 (6)	0.109 (8)	0.065 (6)	0.019 (6)	0.017 (5)	−0.005 (6)
O3B	0.110 (10)	0.081 (9)	0.099 (10)	0.001 (8)	0.038 (8)	0.009 (8)
N1	0.044 (2)	0.024 (2)	0.037 (2)	−0.004 (2)	0.0166 (19)	−0.004 (2)
C1	0.029 (2)	0.025 (3)	0.035 (3)	0.003 (2)	0.011 (2)	0.001 (2)
C2	0.031 (3)	0.030 (3)	0.042 (3)	−0.003 (2)	0.005 (2)	0.001 (2)
C3	0.032 (3)	0.045 (3)	0.044 (3)	−0.003 (3)	0.016 (2)	0.008 (3)
C4	0.042 (3)	0.046 (3)	0.037 (3)	0.004 (3)	0.020 (2)	0.005 (3)
C5	0.037 (3)	0.031 (3)	0.032 (3)	0.004 (2)	0.007 (2)	0.002 (2)
C6	0.028 (3)	0.033 (3)	0.037 (3)	−0.001 (2)	0.013 (2)	0.005 (2)
C7	0.050 (3)	0.026 (3)	0.035 (3)	−0.002 (2)	0.012 (2)	−0.006 (2)
C8	0.069 (4)	0.043 (3)	0.045 (3)	−0.007 (3)	0.027 (3)	−0.012 (3)
C9A	0.062 (6)	0.039 (5)	0.032 (5)	0.007 (5)	0.013 (4)	0.002 (5)
C9B	0.064 (9)	0.050 (8)	0.061 (9)	−0.001 (8)	0.018 (7)	−0.011 (8)
C10A	0.061 (7)	0.041 (7)	0.073 (8)	−0.009 (5)	0.043 (6)	0.002 (6)
C10B	0.048 (8)	0.037 (8)	0.045 (7)	−0.001 (5)	0.009 (4)	−0.004 (5)

Geometric parameters (Å, º)

Cl1—C2	1.733 (5)	C3—H3	0.9300
Cl2—C5	1.739 (5)	C4—C5	1.382 (7)
O1—C7	1.217 (6)	C4—H4	0.9300
O2A—C10A	1.282 (15)	C5—C6	1.377 (7)

O2A—H2A	0.8200	C6—H6	0.9300
O2B—C10B	1.246 (17)	C7—C8	1.507 (7)
O2B—H2B	0.8200	C8—C9A	1.458 (12)
O3A—C10A	1.254 (12)	C8—C9B	1.47 (2)
O3B—C10B	1.256 (15)	C8—H8A	0.9700
N1—C7	1.348 (7)	C8—H8B	0.9700
N1—C1	1.414 (6)	C9A—C10A	1.516 (12)
N1—H1N	0.85 (2)	C9A—H9A	0.9700
C1—C6	1.391 (7)	C9A—H9B	0.9700
C1—C2	1.393 (7)	C9B—C10B	1.505 (16)
C2—C3	1.386 (7)	C9B—H9C	0.9700
C3—C4	1.372 (8)	C9B—H9D	0.9700

C10A—O2A—H2A	109.5	C9A—C8—C7	117.0 (6)
C10B—O2B—H2B	109.5	C9B—C8—C7	110.1 (8)
C7—N1—C1	124.6 (4)	C9A—C8—H8A	108.1
C7—N1—H1N	117 (4)	C9B—C8—H8A	86.4
C1—N1—H1N	118 (4)	C7—C8—H8A	108.1
C6—C1—C2	118.1 (4)	C9A—C8—H8B	108.1
C6—C1—N1	121.3 (4)	C9B—C8—H8B	132.7
C2—C1—N1	120.5 (4)	C7—C8—H8B	108.1
C3—C2—C1	121.0 (5)	H8A—C8—H8B	107.3
C3—C2—Cl1	119.1 (4)	C8—C9A—C10A	112.3 (10)
C1—C2—Cl1	119.9 (4)	C8—C9A—H9A	109.1
C4—C3—C2	120.3 (5)	C10A—C9A—H9A	109.1
C4—C3—H3	119.8	C8—C9A—H9B	109.1
C2—C3—H3	119.8	C10A—C9A—H9B	109.1
C3—C4—C5	119.0 (5)	H9A—C9A—H9B	107.9
C3—C4—H4	120.5	C8—C9B—C10B	118.2 (17)
C5—C4—H4	120.5	C8—C9B—H9C	107.8
C6—C5—C4	121.4 (5)	C10B—C9B—H9C	107.7
C6—C5—Cl2	119.1 (4)	C8—C9B—H9D	107.8
C4—C5—Cl2	119.6 (4)	C10B—C9B—H9D	107.8
C5—C6—C1	120.2 (4)	H9C—C9B—H9D	107.1
C5—C6—H6	119.9	O3A—C10A—O2A	123.5 (12)
C1—C6—H6	119.9	O3A—C10A—C9A	112.0 (11)
O1—C7—N1	123.3 (5)	O2A—C10A—C9A	121.0 (14)
O1—C7—C8	122.0 (5)	O2B—C10B—O3B	117.8 (19)
N1—C7—C8	114.7 (5)	O2B—C10B—C9B	113.7 (18)
C9A—C8—C9B	27.7 (8)	O3B—C10B—C9B	127.6 (18)

C7—N1—C1—C6	−39.9 (8)	C1—N1—C7—O1	−7.7 (9)
C7—N1—C1—C2	142.0 (5)	C1—N1—C7—C8	171.2 (5)
C6—C1—C2—C3	1.5 (8)	O1—C7—C8—C9A	−4.8 (11)
N1—C1—C2—C3	179.7 (5)	N1—C7—C8—C9A	176.2 (7)
C6—C1—C2—Cl1	−179.4 (4)	O1—C7—C8—C9B	24.5 (13)
N1—C1—C2—Cl1	−1.2 (7)	N1—C7—C8—C9B	−154.4 (10)
C1—C2—C3—C4	−0.6 (8)	C9B—C8—C9A—C10A	79 (2)

Cl1—C2—C3—C4	−179.7 (4)	C7—C8—C9A—C10A	160.8 (11)
C2—C3—C4—C5	−0.6 (8)	C9A—C8—C9B—C10B	−70 (2)
C3—C4—C5—C6	0.8 (8)	C7—C8—C9B—C10B	179.8 (15)
C3—C4—C5—Cl2	−178.7 (4)	C8—C9A—C10A—O3A	152.1 (15)
C4—C5—C6—C1	0.1 (8)	C8—C9A—C10A—O2A	−48 (2)
Cl2—C5—C6—C1	179.7 (4)	C8—C9B—C10B—O2B	−33 (3)
C2—C1—C6—C5	−1.3 (7)	C8—C9B—C10B—O3B	136 (3)
N1—C1—C6—C5	−179.4 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*A*—H2*A*···O3*A*^{i}	0.82	1.90	2.687 (15)	162
O2*B*—H2*B*···O3*B*^{i}	0.82	1.90	2.64 (2)	150
N1—H1*N*···O1^{ii}	0.85 (2)	2.07 (2)	2.901 (6)	167 (5)

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