Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Chloro-4-methylphenyl)succinamic acid

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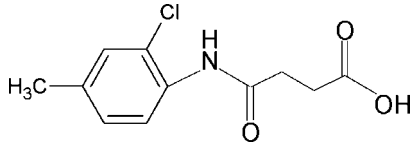
Received 3 February 2012; accepted 8 February 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.053; wR factor = 0.120; data-to-parameter ratio = 15.0.

In the title compound, $C_{11}H_{12}ClNO_3$, the N—C=O fragment is twisted from the plane of the attached benzene ring by 48.39 (12)°. The carboxylic acid group is involved in O—H···O hydrogen bonding, which links pairs of molecules into centrosymmetric dimers. N—H···O hydrogen bonds link these dimers, related by translation along the *a* axis, into ribbons.

Related literature

For the crystal structures of related compounds studied by our group, see: Gowda *et al.* (2012) and references therein.



Experimental

Crystal data

$C_{11}H_{12}ClNO_3$
M_r = 241.67
Triclinic, $P\overline{1}$
a = 4.8097 (8) Å
b = 7.3909 (9) Å
c = 16.147 (2) Å
α = 85.15 (1)°
β = 85.86 (1)°
γ = 89.57 (1)°
V = 570.45 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 293 K
0.40 × 0.18 × 0.09 mm

Data collection

Oxford Xcalibur diffractometer with Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.881, T_{max} = 0.971
3625 measured reflections
2284 independent reflections
1883 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.120
S = 1.11
2284 reflections
152 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.84 (2)	2.16 (2)	2.973 (3)	163 (3)
O3—H3*O*···O2^{ii}	0.83 (2)	1.85 (2)	2.674 (3)	172 (4)

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

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*.

BTG thanks the University Grants Commission, Government of India, New Delhi, for a special grant under a UGC-BSR one-time Grant to Faculty.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5244).

References

Gowda, B. T., Foro, S. & Chaithanya, U. (2012). *Acta Cryst.* E**68**, o221.
Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, Oxfordshire, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2012). E**68**, o785 [doi:10.1107/S1600536812005648]

N-(2-Chloro-4-methylphenyl)succinamic acid

U. Chaithanya, Sabine Foro and B. Thimme Gowda

Comment

Recently, we reported the crystal structure of *N*-(2-chloro-5-methylphenyl)succinamic acid (Gowda *et al.*, 2012). We report here the crystal structure of very closely related *N*-(2-chloro-4-methylphenyl)succinamic acid, (I).

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those observed in related compounds (Gowda *et al.*, 2012, and references therein). The conformations of the amide oxygen and the carboxyl oxygen of the acid segment are *anti* to each other and both are *anti* 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. The dihedral angle between the benzene ring and the amide group is 48.39 (12)°.

The packing of molecules in the crystal through intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) is shown in Fig. 2.

Experimental

The solution of succinic anhydride (0.01 mole) in toluene (25 ml) was treated dropwise with the solution of 2-chloro-4-methylaniline (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-chloro-4-methylaniline. 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. Rod like colourless single crystals used in X-ray diffraction studies were grown in ethanol solution by slow evaporation at room temperature.

Refinement

The H atoms of the NH and OH groups were located in a difference map and restrained to the distances N—H = 0.86 (2) Å and O—H = 0.82 (2) Å, respectively. The other 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. All H atoms were refined with isotropic displacement parameters set to 1.2–1.5U_{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).

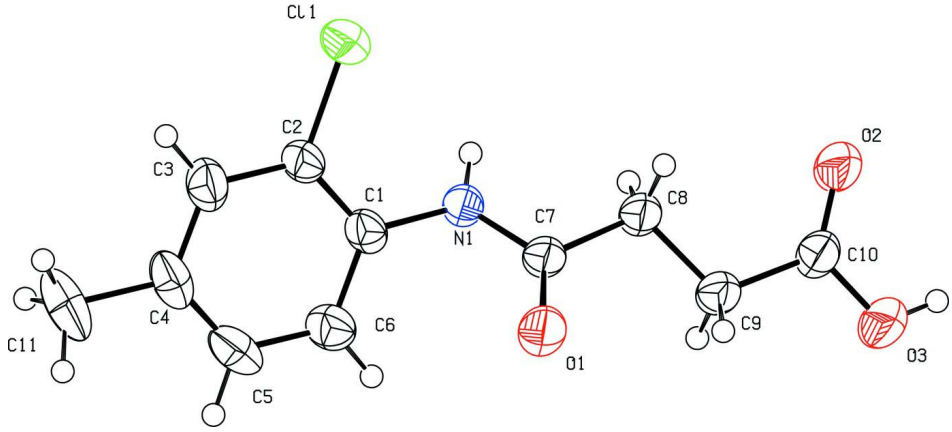


Figure 1

The molecular structure of (I) showing the atom labelling scheme and 50% probability displacement ellipsoids.

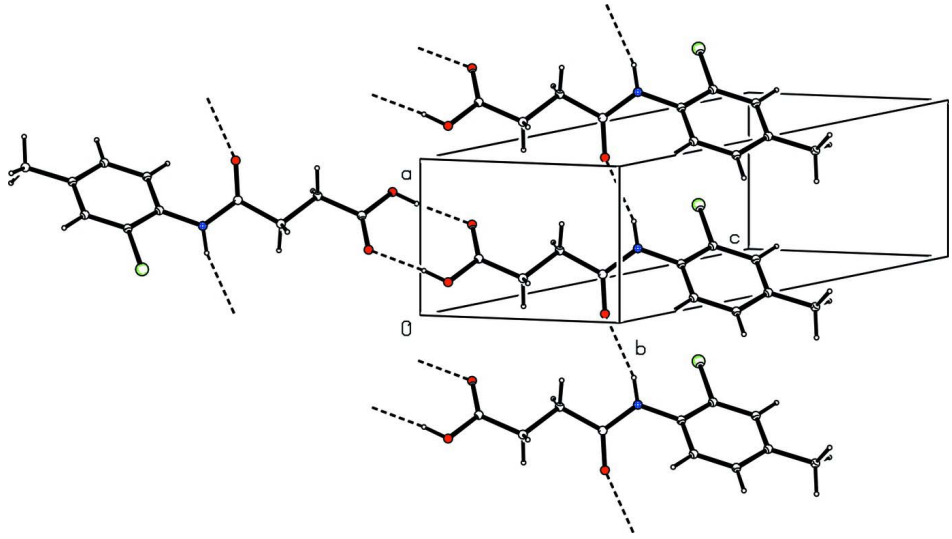


Figure 2

A portion of the crystal packing with hydrogen bonds shown as dashed lines.

N-(2-Chloro-4-methylphenyl)succinamic acid

Crystal data

$C_{11}H_{12}ClNO_3$
$M_r = 241.67$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.8097\ (8)$ Å
$b = 7.3909\ (9)$ Å
$c = 16.147\ (2)$ Å
$\alpha = 85.15\ (1)°$
$\beta = 85.86\ (1)°$
$\gamma = 89.57\ (1)°$
$V = 570.45\ (14)$ Å^3
$Z = 2$
$F(000) = 252$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2016 reflections
$\theta = 2.5–27.9°$
$\mu = 0.33$ mm^{-1}
$T = 293$ K
Rod, colourless
0.40 × 0.18 × 0.09 mm

Data collection

Oxford Xcalibur
diffractometer with Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω and φ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.881, T_{max} = 0.971

3625 measured reflections
2284 independent reflections
1883 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 26.4°, θ_{min} = 2.5°
h = −5→5
k = −9→9
l = −17→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.120
S = 1.11
2284 reflections
152 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
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 0.5262P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.7464 (5)	0.1423 (3)	0.72556 (16)	0.0372 (5)
C2	0.6617 (5)	0.1119 (3)	0.64792 (16)	0.0397 (6)
C3	0.7600 (6)	−0.0346 (3)	0.60606 (18)	0.0479 (6)
H3	0.6972	−0.0528	0.5543	0.057*
C4	0.9510 (6)	−0.1542 (4)	0.64083 (19)	0.0516 (7)
C5	1.0408 (6)	−0.1206 (4)	0.7175 (2)	0.0544 (7)
H5	1.1728	−0.1976	0.7412	0.065*
C6	0.9412 (5)	0.0232 (4)	0.76017 (18)	0.0465 (6)
H6	1.0040	0.0408	0.8120	0.056*
C7	0.7910 (5)	0.4059 (3)	0.80500 (15)	0.0386 (6)
C8	0.6293 (5)	0.5544 (4)	0.84608 (17)	0.0431 (6)
H8A	0.4480	0.5081	0.8679	0.052*
H8B	0.5999	0.6544	0.8047	0.052*

C9	0.7800 (5)	0.6234 (4)	0.91616 (17)	0.0449 (6)
H9A	0.7896	0.5262	0.9602	0.054*
H9B	0.9696	0.6544	0.8955	0.054*
C10	0.6475 (5)	0.7848 (4)	0.95222 (16)	0.0441 (6)
C11	1.0540 (8)	−0.3163 (4)	0.5961 (2)	0.0790 (11)
H11A	1.0422	−0.2903	0.5371	0.095*
H11B	0.9409	−0.4201	0.6151	0.095*
H11C	1.2442	−0.3414	0.6075	0.095*
N1	0.6355 (4)	0.2886 (3)	0.76914 (14)	0.0404 (5)
H1N	0.463 (4)	0.306 (4)	0.7706 (17)	0.049*
O1	1.0450 (3)	0.3984 (3)	0.80286 (14)	0.0594 (6)
O2	0.4442 (4)	0.8624 (3)	0.92593 (15)	0.0741 (7)
O3	0.7748 (6)	0.8378 (4)	1.01368 (17)	0.0888 (9)
H3O	0.696 (8)	0.924 (4)	1.035 (2)	0.107*
Cl1	0.42759 (15)	0.26087 (10)	0.60111 (5)	0.0573 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0262 (11)	0.0349 (12)	0.0506 (14)	0.0017 (9)	0.0016 (10)	−0.0079 (10)
C2	0.0289 (12)	0.0395 (13)	0.0520 (15)	0.0062 (10)	−0.0033 (10)	−0.0110 (11)
C3	0.0484 (15)	0.0411 (14)	0.0554 (16)	0.0046 (12)	0.0006 (12)	−0.0164 (12)
C4	0.0498 (16)	0.0356 (13)	0.0685 (19)	0.0076 (11)	0.0104 (14)	−0.0118 (12)
C5	0.0447 (15)	0.0441 (15)	0.073 (2)	0.0164 (12)	−0.0009 (14)	0.0012 (14)
C6	0.0374 (14)	0.0482 (15)	0.0540 (16)	0.0057 (11)	−0.0033 (11)	−0.0056 (12)
C7	0.0255 (12)	0.0447 (13)	0.0474 (14)	0.0032 (10)	−0.0044 (10)	−0.0137 (11)
C8	0.0266 (12)	0.0492 (14)	0.0570 (16)	0.0044 (10)	−0.0076 (11)	−0.0217 (12)
C9	0.0332 (13)	0.0532 (15)	0.0516 (15)	0.0059 (11)	−0.0090 (11)	−0.0192 (12)
C10	0.0353 (13)	0.0533 (15)	0.0463 (14)	−0.0003 (11)	−0.0036 (11)	−0.0192 (12)
C11	0.094 (3)	0.0468 (17)	0.095 (3)	0.0276 (17)	0.015 (2)	−0.0168 (17)
N1	0.0214 (9)	0.0463 (12)	0.0568 (13)	0.0056 (8)	−0.0042 (9)	−0.0223 (10)
O1	0.0219 (9)	0.0689 (13)	0.0934 (16)	0.0049 (8)	−0.0080 (9)	−0.0395 (12)
O2	0.0578 (13)	0.0868 (16)	0.0892 (16)	0.0282 (12)	−0.0307 (12)	−0.0551 (13)
O3	0.0870 (18)	0.0996 (19)	0.0954 (19)	0.0402 (14)	−0.0513 (15)	−0.0655 (16)
Cl1	0.0547 (4)	0.0582 (4)	0.0627 (5)	0.0234 (3)	−0.0177 (3)	−0.0183 (3)

Geometric parameters (Å, º)

C1—C2	1.382 (3)	C7—C8	1.512 (3)
C1—C6	1.395 (3)	C8—C9	1.512 (3)
C1—N1	1.420 (3)	C8—H8A	0.9700
C2—C3	1.387 (3)	C8—H8B	0.9700
C2—Cl1	1.736 (2)	C9—C10	1.490 (3)
C3—C4	1.385 (4)	C9—H9A	0.9700
C3—H3	0.9300	C9—H9B	0.9700
C4—C5	1.383 (4)	C10—O2	1.215 (3)
C4—C11	1.513 (4)	C10—O3	1.292 (3)
C5—C6	1.379 (4)	C11—H11A	0.9600
C5—H5	0.9300	C11—H11B	0.9600
C6—H6	0.9300	C11—H11C	0.9600

C7—O1	1.221 (3)	N1—H1N	0.839 (17)
C7—N1	1.343 (3)	O3—H3O	0.827 (19)
C2—C1—C6	117.9 (2)	C9—C8—H8A	109.2
C2—C1—N1	121.0 (2)	C7—C8—H8B	109.2
C6—C1—N1	121.1 (2)	C9—C8—H8B	109.2
C1—C2—C3	121.6 (2)	H8A—C8—H8B	107.9
C1—C2—Cl1	119.42 (18)	C10—C9—C8	114.3 (2)
C3—C2—Cl1	118.9 (2)	C10—C9—H9A	108.7
C4—C3—C2	120.5 (3)	C8—C9—H9A	108.7
C4—C3—H3	119.8	C10—C9—H9B	108.7
C2—C3—H3	119.8	C8—C9—H9B	108.7
C5—C4—C3	117.7 (2)	H9A—C9—H9B	107.6
C5—C4—C11	121.7 (3)	O2—C10—O3	122.8 (2)
C3—C4—C11	120.6 (3)	O2—C10—C9	124.0 (2)
C6—C5—C4	122.2 (3)	O3—C10—C9	113.2 (2)
C6—C5—H5	118.9	C4—C11—H11A	109.5
C4—C5—H5	118.9	C4—C11—H11B	109.5
C5—C6—C1	120.0 (3)	H11A—C11—H11B	109.5
C5—C6—H6	120.0	C4—C11—H11C	109.5
C1—C6—H6	120.0	H11A—C11—H11C	109.5
O1—C7—N1	123.2 (2)	H11B—C11—H11C	109.5
O1—C7—C8	121.7 (2)	C7—N1—C1	124.10 (19)
N1—C7—C8	115.08 (19)	C7—N1—H1N	117.9 (19)
C7—C8—C9	111.9 (2)	C1—N1—H1N	117.9 (19)
C7—C8—H8A	109.2	C10—O3—H3O	113 (3)
C6—C1—C2—C3	1.7 (4)	C2—C1—C6—C5	−0.8 (4)
N1—C1—C2—C3	−177.4 (2)	N1—C1—C6—C5	178.4 (2)
C6—C1—C2—Cl1	−178.02 (19)	O1—C7—C8—C9	−28.9 (4)
N1—C1—C2—Cl1	2.8 (3)	N1—C7—C8—C9	153.3 (2)
C1—C2—C3—C4	−1.0 (4)	C7—C8—C9—C10	173.0 (2)
Cl1—C2—C3—C4	178.8 (2)	C8—C9—C10—O2	−3.6 (4)
C2—C3—C4—C5	−0.7 (4)	C8—C9—C10—O3	177.9 (3)
C2—C3—C4—C11	178.7 (3)	O1—C7—N1—C1	0.6 (4)
C3—C4—C5—C6	1.7 (4)	C8—C7—N1—C1	178.4 (2)
C11—C4—C5—C6	−177.8 (3)	C2—C1—N1—C7	−132.0 (3)
C4—C5—C6—C1	−1.0 (4)	C6—C1—N1—C7	48.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.84 (2)	2.16 (2)	2.973 (3)	163 (3)
O3—H3*O*···O2^{ii}	0.83 (2)	1.85 (2)	2.674 (3)	172 (4)

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