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3-[(3,5-Dichloroanilino)carbonyl]-propionic acid

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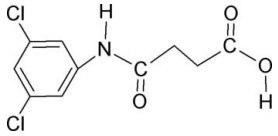
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.170; data-to-parameter ratio = 13.3.

In the crystal structure of the title compound, $C_{10}H_9Cl_2NO_3$, the conformations of the amide O atom and the carbonyl O atom of the acid segment are *anti* to the H atoms of the adjacent –CH_2 groups. The C═O and O—H bonds of the acid group are in relatively rare *anti* positions with respect to each other. This is an obvious consequence of the concerted effects of both the all-*anti* molecular conformation and the intermolecular hydrogen bond donated to the amide carbonyl group. In the crystal, molecules are packed into infinite chains through intermolecular N—H···O and O—H···O hydrogen bonds.

Related literature

For the effect of ring and side-chain substitutions on the structures of amide compounds, see: Gowda *et al.* (2009). For the packing of molecules involving dimeric hydrogen-bonded association of each carboxyl group with a centrosymmetrically related neighbor, see: Jagannathan *et al.* (1994). For the various modes of interlinking carboxylic acids by hydrogen bonds, see: Leiserowitz (1976).



Experimental

Crystal data

$C_{10}H_9Cl_2NO_3$ M_r = 262.08
Monoclinic, $P2_1/n$
a = 7.350 (1) Å
b = 10.318 (2) Å
c = 15.031 (3) Å
β = 99.44 (2)°
V = 1124.5 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 5.15 mm^{-1}
T = 299 K
0.48 × 0.30 × 0.28 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.142, T_{max} = 0.242
4204 measured reflections
2005 independent reflections
1794 reflections with $I > 2\sigma(I)$
R_{int} = 0.111
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.170
S = 1.13
2005 reflections
151 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O3^{i}	0.95 (4)	1.93 (4)	2.857 (3)	167 (3)
O2—H2*O*···O1^{ii}	0.82 (2)	1.85 (2)	2.656 (3)	170 (4)

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); 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: CS2111).

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supporting information

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3-[(3,5-Dichloroanilino)carbonyl]propionic acid

B. Thimme Gowda, Sabine Foro, B. S. Saraswathi, Hiromitsu Terao and Hartmut Fuess

S1. Comment

The amide moiety is an important constituent of many biologically significant compounds. As a part of studying the effect of ring and side chain substitutions on the structures of this class of compounds (Gowda *et al.*, 2009), we have determined the crystal structure of *N*-(3,5-dichlorophenyl)-succinamic acid (N35DCPSA, systematic name: 3-[(3,5-dichloro)-aminocarbonyl]propionic acid). The conformations of N—H and C=O bonds in the amide segment of the structure are *anti* to each other and those of the amide O atom and the carbonyl O atom of the acid segment are also *anti* to the H atoms attached to the adjacent C atoms (Fig.1). Further, C=O and O—H bonds of the acid group are *anti* to each other, contrary to the more general *syn* conformation observed for C=O and O—H bonds of the acid group e.g. *N*-(2,6-dimethylphenyl)- succinamic acid (N26DMPSA, Gowda *et al.*, 2009). The various 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). In the present study, the rare *anti* conformation of the C=O and O—H bonds of the acid group has been observed. The torsional angles of the groups, C2—C1—N1—C7, C6—C1—N1—C7, C1—N1—C7—C8, C1—N1—C7—O1, N1—C7—C8—C9, C7—C8—C9—C10, O1—C7—C8—C9, C8—C9—C10—O2 and C8—C9—C10—O3 in the side chain of N35DCPSA are -180.0 (3)°, -1.3 (4)°, -174.4 (3)°, 4.3 (5)°, 178.9 (2)°, -175.5 (2)°, 0.4 (2)°, 175.0 (3)° and -5.3 (5)°, respectively, compared to the corresponding values of 114.1 (2)°, -66.5 (2)°, -176.2 (1)°, 2.0 (3)°, -145.4 (2)°, -175.5 (1)°, 36.3 (2)°, -161.1 (2)° and 19.1 (3)°, respectively, for N26DMPSA. The N—H···O and O—H···O intermolecular hydrogen bonds pack the molecules into infinite chains in the structure (Table 1, Fig.2).

S2. Experimental

The solution of succinic anhydride (0.025 mol) in toluene (25 ml) was treated dropwise with the solution of 3,5-dichloroaniline (0.025 mol) 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 the completion of reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 3,5-dichloroaniline. The resultant solid *N*-(3,5-dichlorophenyl)-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 single crystals used in X-ray diffraction studies were grown in an ethanol solution by slow evaporation at room temperature.

S3. Refinement

The N-bound and O-bound H atoms were located in a difference map. The position of the N-bound H atom was refined with N—H = 0.95 (4) Å and that of the O—H was refined with a distance restrained to 0.82 (2) Å. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93–0.97Å]. All H atoms were treated with

isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

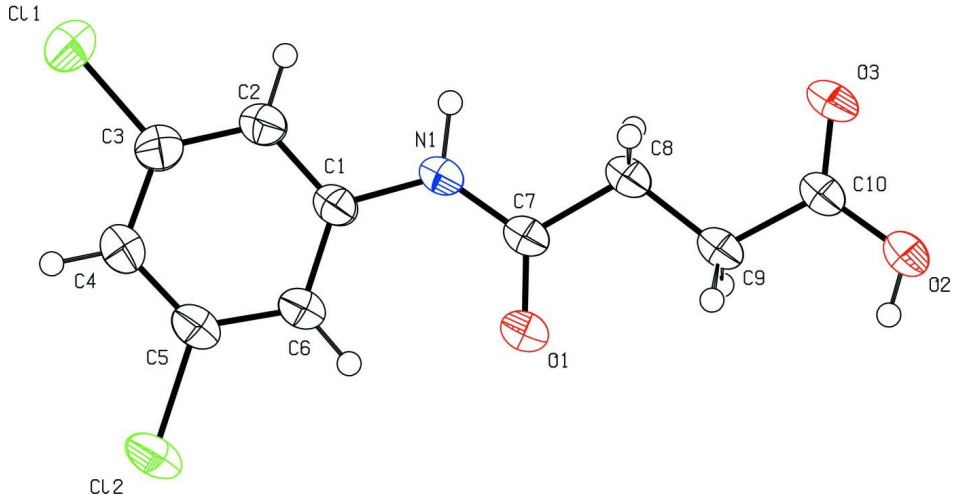

Figure 1

Molecular structure of the title molecule with atom labeling. Displacement ellipsoids are at the 50% probability level, H atoms represented as small spheres of arbitrary radii.

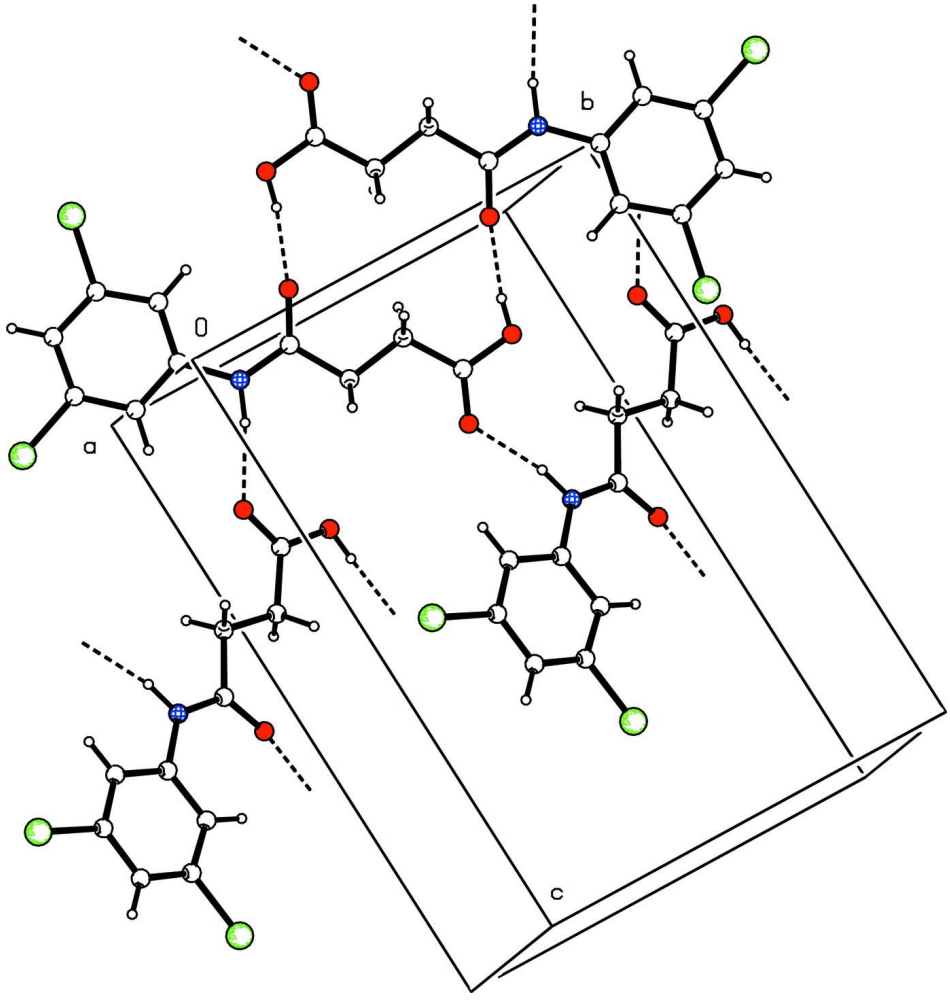

Figure 2

Molecular packing detail in the title crystal with hydrogen bonds shown as dashed lines.

3-[(3,5-Dichloroanilino)carbonyl]propionic acid

Crystal data

$C_{10}H_9Cl_2NO_3$
$M_r = 262.08$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.350$ (1) Å
$b = 10.318$ (2) Å
$c = 15.031$ (3) Å
$\beta = 99.44$ (2)°
$V = 1124.5$ (3) Å^3
$Z = 4$
$F(000) = 536$
$D_x = 1.548$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 7.4$–20.6°
$\mu = 5.15$ mm^{-1}
$T = 299$ K
Prism, colourless
0.48 × 0.30 × 0.28 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.142$, $T_{max} = 0.242$

4204 measured reflections
2005 independent reflections
1794 reflections with $I > 2\sigma(I)$
$R_{int} = 0.111$
$\theta_{max} = 67.1°$, $\theta_{min} = 5.2°$

$h = 0 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$
3 standard reflections every 120 min
intensity decay: 1.0%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.170$
$S = 1.13$
2005 reflections
151 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0804P)^2 + 0.4071P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.009$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2687 (3)	0.5234 (3)	0.51327 (16)	0.0360 (6)
C2	0.1971 (3)	0.4036 (3)	0.48363 (18)	0.0390 (6)
H2	0.1668	0.3863	0.4223	0.047*
C3	0.1715 (4)	0.3108 (3)	0.54585 (19)	0.0415 (6)
C4	0.2153 (4)	0.3313 (3)	0.63743 (19)	0.0453 (7)
H4	0.1971	0.2676	0.6789	0.054*
C5	0.2872 (4)	0.4502 (4)	0.66443 (18)	0.0453 (7)
C6	0.3155 (4)	0.5480 (3)	0.60551 (17)	0.0435 (7)
H6	0.3642	0.6276	0.6266	0.052*
C7	0.3499 (3)	0.7371 (3)	0.45487 (16)	0.0374 (6)
C8	0.3313 (4)	0.8125 (3)	0.36730 (17)	0.0421 (7)
H8A	0.3996	0.7685	0.3263	0.051*
H8B	0.2025	0.8148	0.3395	0.051*
C9	0.4014 (4)	0.9480 (3)	0.38180 (17)	0.0447 (7)
H9A	0.5322	0.9451	0.4055	0.054*
H9B	0.3400	0.9891	0.4268	0.054*
C10	0.3724 (4)	1.0293 (3)	0.29825 (18)	0.0436 (7)
N1	0.2863 (3)	0.6158 (3)	0.44600 (15)	0.0403 (6)
H1N	0.242 (4)	0.584 (4)	0.387 (3)	0.048*
O1	0.4152 (3)	0.7867 (3)	0.52683 (13)	0.0543 (6)
O2	0.4186 (4)	1.1531 (3)	0.30787 (14)	0.0622 (7)
H2O	0.459 (5)	1.167 (5)	0.3609 (15)	0.075*
O3	0.3114 (4)	0.9893 (3)	0.22424 (13)	0.0620 (7)
Cl1	0.07961 (12)	0.16217 (8)	0.50728 (6)	0.0580 (3)
Cl2	0.34909 (13)	0.47994 (11)	0.77952 (5)	0.0696 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0448 (12)	0.0368 (17)	0.0252 (12)	0.0015 (11)	0.0026 (9)	0.0014 (11)
C2	0.0487 (13)	0.0390 (17)	0.0287 (12)	0.0004 (12)	0.0050 (10)	−0.0022 (11)
C3	0.0490 (13)	0.0379 (17)	0.0382 (14)	−0.0009 (12)	0.0089 (10)	−0.0034 (12)
C4	0.0589 (16)	0.044 (2)	0.0339 (14)	−0.0048 (13)	0.0098 (11)	0.0050 (12)
C5	0.0585 (15)	0.051 (2)	0.0265 (13)	−0.0048 (13)	0.0071 (10)	0.0019 (12)
C6	0.0590 (15)	0.0454 (19)	0.0254 (13)	−0.0089 (13)	0.0044 (10)	−0.0008 (12)
C7	0.0463 (12)	0.0401 (17)	0.0242 (11)	−0.0013 (12)	0.0007 (9)	0.0008 (11)
C8	0.0598 (15)	0.0408 (17)	0.0237 (12)	−0.0014 (13)	0.0007 (10)	0.0025 (11)
C9	0.0643 (15)	0.0440 (19)	0.0230 (12)	−0.0036 (14)	−0.0009 (10)	0.0038 (12)
C10	0.0611 (15)	0.0426 (18)	0.0252 (12)	−0.0021 (13)	0.0012 (10)	0.0043 (12)
N1	0.0602 (12)	0.0370 (15)	0.0220 (10)	−0.0033 (11)	0.0019 (8)	−0.0001 (9)
O1	0.0824 (13)	0.0483 (15)	0.0272 (10)	−0.0156 (11)	−0.0056 (9)	0.0019 (9)
O2	0.1047 (17)	0.0458 (15)	0.0289 (11)	−0.0123 (13)	−0.0107 (10)	0.0092 (10)
O3	0.1032 (17)	0.0545 (16)	0.0224 (10)	−0.0094 (13)	−0.0071 (10)	0.0031 (9)
Cl1	0.0808 (6)	0.0403 (6)	0.0533 (5)	−0.0134 (4)	0.0127 (4)	−0.0065 (3)
Cl2	0.1032 (7)	0.0795 (8)	0.0243 (4)	−0.0260 (5)	0.0056 (4)	0.0014 (3)

Geometric parameters (Å, º)

C1—C2	1.388 (4)	C7—N1	1.335 (4)
C1—C6	1.396 (4)	C7—C8	1.516 (4)
C1—N1	1.411 (4)	C8—C9	1.494 (5)
C2—C3	1.372 (4)	C8—H8A	0.97
C2—H2	0.93	C8—H8B	0.97
C3—C4	1.378 (4)	C9—C10	1.496 (4)
C3—Cl1	1.737 (3)	C9—H9A	0.97
C4—C5	1.371 (5)	C9—H9B	0.97
C4—H4	0.93	C10—O3	1.202 (4)
C5—C6	1.381 (4)	C10—O2	1.323 (4)
C5—Cl2	1.742 (3)	N1—H1N	0.95 (4)
C6—H6	0.93	O2—H2O	0.82 (2)
C7—O1	1.221 (3)		
C2—C1—C6	120.0 (3)	N1—C7—C8	114.5 (2)
C2—C1—N1	116.5 (2)	C9—C8—C7	112.0 (2)
C6—C1—N1	123.5 (3)	C9—C8—H8A	109
C3—C2—C1	119.3 (2)	C7—C8—H8A	109
C3—C2—H2	120.3	C9—C8—H8B	109
C1—C2—H2	120.3	C7—C8—H8B	109
C2—C3—C4	122.6 (3)	H8A—C8—H8B	108
C2—C3—Cl1	118.5 (2)	C8—C9—C10	113.8 (2)
C4—C3—Cl1	118.9 (2)	C8—C9—H9A	109
C5—C4—C3	116.7 (3)	C10—C9—H9A	109
C5—C4—H4	121.7	C8—C9—H9B	109
C3—C4—H4	121.7	C10—C9—H9B	109

C4—C5—C6	123.7 (3)	H9A—C9—H9B	108
C4—C5—Cl2	118.5 (2)	O3—C10—O2	118.8 (3)
C6—C5—Cl2	117.8 (3)	O3—C10—C9	124.4 (3)
C5—C6—C1	117.7 (3)	O2—C10—C9	116.8 (3)
C5—C6—H6	121.1	C7—N1—C1	129.3 (2)
C1—C6—H6	121.1	C7—N1—H1N	118 (2)
O1—C7—N1	124.2 (3)	C1—N1—H1N	112 (2)
O1—C7—C8	121.3 (3)	C10—O2—H2O	109 (3)
O1—C7—C8—C9	0.2 (4)	O1—C7—N1—C1	4.3 (5)
N1—C7—C8—C9	178.9 (2)	C8—C7—N1—C1	−174.4 (3)
C7—C8—C9—C10	−175.5 (2)	C2—C1—N1—C7	180.0 (3)
C8—C9—C10—O3	−5.3 (5)	C6—C1—N1—C7	1.3 (4)
C8—C9—C10—O2	175.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O3^{i}	0.95 (4)	1.93 (4)	2.857 (3)	167 (3)
O2—H2*O*···O1^{ii}	0.82 (2)	1.85 (2)	2.656 (3)	170 (4)

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