

Methyl 3-[(3,5-dichloroanilino)carbonyl]-propionate

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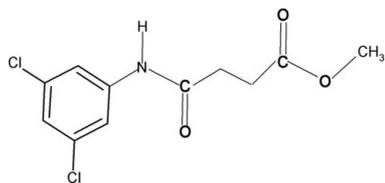
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.061; wR factor = 0.166; data-to-parameter ratio = 14.4.

In the title compound, $\text{C}_{11}\text{H}_{11}\text{Cl}_2\text{NO}_3$, the amide O atom and the carbonyl O atom of the ester segment are *anti* to each other and *anti* to the H atoms of the adjacent $-\text{CH}_2$ groups. In the crystal structure, molecules are packed into centrosymmetric dimers through intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The dimers are linked into a layer structure extending parallel to $(\bar{1}02)$ by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related structures, see: Gowda *et al.* (2009a,b,c).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{11}\text{Cl}_2\text{NO}_3$
 $M_r = 276.11$
Monoclinic, $C2/c$
 $a = 12.865$ (2) Å
 $b = 14.753$ (3) Å
 $c = 14.114$ (2) Å
 $\beta = 109.59$ (2)°

$V = 2523.7$ (7) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.51$ mm⁻¹
 $T = 299$ K
 $0.50 \times 0.16 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford

Diffraction, 2009)
 $T_{\min} = 0.785$, $T_{\max} = 0.941$
4503 measured reflections
2259 independent reflections
1453 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.166$
 $S = 1.09$
2259 reflections
157 parameters
1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.37$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{N1}-\text{H1N}\cdots\text{O2}^i$ | 0.85 (2) | 2.17 (2) | 3.017 (4) | 172 (4) |
| $\text{C4}-\text{H4}\cdots\text{O1}^{ii}$ | 0.93 | 2.45 | 3.379 (5) | 174 |

Symmetry codes: (i) $-x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5017).

References

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supplementary materials

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Methyl 3-[(3,5-dichloroanilino)carbonyl]propionate

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Comment

As a part of studying the effect of ring and side chain substitutions on the structures of biologically significant compounds (Gowda *et al.*, 2009*a,b,c*), the crystal structure of *N*-(3,5-dichlorophenyl)methylsuccinamate [systematic name: 3-[(3,5-dichloro)-aminocarbonyl]propionate] has been determined.

The conformation of the amide O atom and the carbonyl O atom of the ester segment are *anti* to each other and both are *anti* to the H atoms of the adjacent -CH₂ groups (Fig. 1), similar to that observed in *N*-(3,5-dichlorophenyl)methylsuccinamic acid (Gowda *et al.*, 2009*c*) and *N*-(3,5-dimethylphenyl)ethylsuccinamate (Gowda *et al.*, 2009*a*).

In the crystal, molecules are packed into centrosymmetric dimers through intermolecular N—H...O hydrogen bonds (Table 1 and Fig.2).

Experimental

A solution of succinic anhydride (0.02 mol) in toluene (25 ml) was treated dropwise with a solution of 3,5-dichloroaniline (0.02 mol) in toluene (20 ml) with constant stirring. The resulting mixture was stirred for 1 h and set aside for an additional 1 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 methanol. Pure *N*-(3,5-dichlorophenyl)succinamic acid in methanol was refluxed with 2 ml of conc. sulfuric acid for 2 h and was subjected to slow evaporation. The resulting *N*-(3,5-dichlorophenyl)methylsuccinamate was recrystallized from methanol. The purity of the compound was checked and characterized by its IR and NMR spectra. Single crystals were grown in a methanol solution by slow evaporation at room temperature.

Refinement

The H atom of the NH group was located in a difference map and refined with a N—H distance restraint of 0.86 (2) Å. The remaining H atoms were positioned geometrically [C—H = 0.93–0.97 Å] and refined using a riding model. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

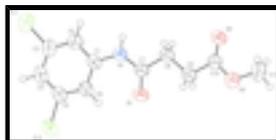


Fig. 1. Molecular structure of the title compound, showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

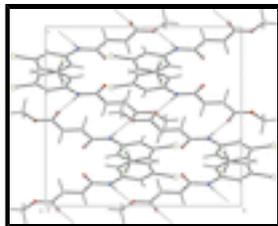


Fig. 2. Part of the crystal structure of the title compound, showing hydrogen-bonded (dashed lines) dimers.

Methyl 3-[(3,5-dichloroanilino)carbonyl]propionate

Crystal data

$C_{11}H_{11}Cl_2NO_3$

$M_r = 276.11$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 12.865\ (2)\ \text{\AA}$

$b = 14.753\ (3)\ \text{\AA}$

$c = 14.114\ (2)\ \text{\AA}$

$\beta = 109.59\ (2)^\circ$

$V = 2523.7\ (7)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1136$

$D_x = 1.453\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1000 reflections

$\theta = 3.0\text{--}28.1^\circ$

$\mu = 0.51\ \text{mm}^{-1}$

$T = 299\ \text{K}$

Needle, colourless

$0.50 \times 0.16 \times 0.12\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube graphite

Rotation method data acquisition using ω and φ scans.

Absorption correction: multi-scan (*Crys.Alis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.785$, $T_{\max} = 0.941$

4503 measured reflections

2259 independent reflections

1453 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 3.0^\circ$

$h = -11 \rightarrow 15$

$k = -17 \rightarrow 10$

$l = -17 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.166$

$S = 1.09$

2259 reflections

157 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 4.6291P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.37\ \text{e \AA}^{-3}$

1 restraint

$$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| C1 | 0.0757 (3) | 0.6059 (3) | 0.6618 (3) | 0.0532 (9) |
| C2 | 0.0482 (3) | 0.5146 (3) | 0.6460 (3) | 0.0591 (10) |
| H2 | -0.0243 | 0.4975 | 0.6116 | 0.071* |
| C3 | 0.1280 (3) | 0.4501 (3) | 0.6814 (3) | 0.0616 (11) |
| C4 | 0.2370 (3) | 0.4721 (3) | 0.7337 (3) | 0.0627 (11) |
| H4 | 0.2906 | 0.4276 | 0.7580 | 0.075* |
| C5 | 0.2620 (3) | 0.5621 (3) | 0.7481 (3) | 0.0583 (10) |
| C6 | 0.1848 (3) | 0.6306 (3) | 0.7149 (3) | 0.0565 (10) |
| H6 | 0.2048 | 0.6911 | 0.7275 | 0.068* |
| C7 | -0.0024 (3) | 0.7610 (3) | 0.6273 (3) | 0.0560 (10) |
| C8 | -0.1087 (3) | 0.8104 (3) | 0.5736 (3) | 0.0603 (11) |
| H8A | -0.1369 | 0.7910 | 0.5040 | 0.072* |
| H8B | -0.1632 | 0.7953 | 0.6047 | 0.072* |
| C9 | -0.0911 (3) | 0.9108 (3) | 0.5778 (3) | 0.0624 (11) |
| H9A | -0.0324 | 0.9245 | 0.5512 | 0.075* |
| H9B | -0.0665 | 0.9298 | 0.6477 | 0.075* |
| C10 | -0.1908 (3) | 0.9652 (3) | 0.5209 (3) | 0.0551 (10) |
| C11 | -0.2543 (4) | 1.1146 (3) | 0.4756 (4) | 0.0849 (15) |
| H11A | -0.3094 | 1.1103 | 0.5074 | 0.102* |
| H11B | -0.2866 | 1.0998 | 0.4056 | 0.102* |
| H11C | -0.2258 | 1.1753 | 0.4824 | 0.102* |
| N1 | -0.0090 (2) | 0.6699 (2) | 0.6230 (3) | 0.0569 (9) |
| H1N | -0.072 (2) | 0.645 (3) | 0.595 (3) | 0.068* |
| O1 | 0.0828 (2) | 0.8016 (2) | 0.6710 (3) | 0.0875 (11) |
| O2 | -0.2818 (2) | 0.93505 (18) | 0.4784 (2) | 0.0668 (8) |
| O3 | -0.1659 (2) | 1.05231 (19) | 0.5228 (2) | 0.0754 (9) |
| Cl1 | 0.09280 (11) | 0.33629 (8) | 0.66222 (11) | 0.0926 (5) |
| Cl2 | 0.39797 (8) | 0.59326 (9) | 0.81343 (10) | 0.0845 (5) |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0445 (19) | 0.062 (2) | 0.051 (2) | 0.0079 (18) | 0.0133 (17) | 0.0045 (19) |
| C2 | 0.057 (2) | 0.059 (3) | 0.058 (2) | -0.0010 (19) | 0.0150 (18) | -0.001 (2) |
| C3 | 0.067 (2) | 0.056 (3) | 0.064 (3) | 0.010 (2) | 0.024 (2) | 0.004 (2) |
| C4 | 0.061 (2) | 0.067 (3) | 0.061 (3) | 0.019 (2) | 0.021 (2) | 0.008 (2) |
| C5 | 0.048 (2) | 0.070 (3) | 0.056 (2) | 0.0104 (19) | 0.0149 (18) | 0.002 (2) |
| C6 | 0.048 (2) | 0.061 (2) | 0.056 (2) | 0.0050 (19) | 0.0113 (18) | 0.0050 (19) |
| C7 | 0.044 (2) | 0.053 (2) | 0.061 (3) | -0.0037 (18) | 0.0048 (17) | 0.0035 (19) |
| C8 | 0.045 (2) | 0.057 (2) | 0.066 (3) | -0.0019 (18) | 0.0017 (18) | 0.004 (2) |
| C9 | 0.045 (2) | 0.058 (3) | 0.072 (3) | -0.0013 (18) | 0.0032 (19) | 0.005 (2) |
| C10 | 0.052 (2) | 0.054 (2) | 0.055 (2) | -0.0041 (18) | 0.0130 (18) | -0.0002 (19) |
| C11 | 0.096 (4) | 0.058 (3) | 0.101 (4) | 0.018 (3) | 0.033 (3) | 0.010 (3) |
| N1 | 0.0402 (16) | 0.055 (2) | 0.065 (2) | -0.0013 (15) | 0.0040 (15) | 0.0031 (16) |
| O1 | 0.0455 (16) | 0.0632 (19) | 0.125 (3) | -0.0082 (14) | -0.0102 (16) | 0.0064 (18) |
| O2 | 0.0458 (15) | 0.0670 (18) | 0.075 (2) | -0.0014 (13) | 0.0035 (13) | 0.0041 (15) |
| O3 | 0.0653 (18) | 0.0543 (18) | 0.095 (2) | 0.0024 (14) | 0.0120 (16) | 0.0036 (16) |
| Cl1 | 0.0935 (9) | 0.0592 (7) | 0.1195 (12) | 0.0048 (6) | 0.0283 (8) | -0.0022 (7) |
| Cl2 | 0.0464 (6) | 0.0885 (9) | 0.1033 (10) | 0.0137 (5) | 0.0047 (6) | -0.0017 (7) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------|-----------|------------|------------|
| C1—C2 | 1.391 (5) | C7—C8 | 1.510 (5) |
| C1—C6 | 1.401 (5) | C8—C9 | 1.496 (5) |
| C1—N1 | 1.407 (5) | C8—H8A | 0.97 |
| C2—C3 | 1.366 (5) | C8—H8B | 0.97 |
| C2—H2 | 0.93 | C9—C10 | 1.499 (5) |
| C3—C4 | 1.387 (6) | C9—H9A | 0.97 |
| C3—C11 | 1.736 (4) | C9—H9B | 0.97 |
| C4—C5 | 1.365 (6) | C10—O2 | 1.207 (4) |
| C4—H4 | 0.93 | C10—O3 | 1.322 (4) |
| C5—C6 | 1.384 (5) | C11—O3 | 1.440 (5) |
| C5—Cl2 | 1.743 (4) | C11—H11A | 0.96 |
| C6—H6 | 0.93 | C11—H11B | 0.96 |
| C7—O1 | 1.219 (4) | C11—H11C | 0.96 |
| C7—N1 | 1.347 (5) | N1—H1N | 0.850 (19) |
| C2—C1—C6 | 119.5 (4) | C7—C8—H8A | 109.4 |
| C2—C1—N1 | 117.8 (3) | C9—C8—H8B | 109.4 |
| C6—C1—N1 | 122.7 (4) | C7—C8—H8B | 109.4 |
| C3—C2—C1 | 119.8 (4) | H8A—C8—H8B | 108.0 |
| C3—C2—H2 | 120.1 | C8—C9—C10 | 114.7 (3) |
| C1—C2—H2 | 120.1 | C8—C9—H9A | 108.6 |
| C2—C3—C4 | 122.2 (4) | C10—C9—H9A | 108.6 |
| C2—C3—C11 | 119.4 (3) | C8—C9—H9B | 108.6 |
| C4—C3—C11 | 118.3 (3) | C10—C9—H9B | 108.6 |
| C5—C4—C3 | 116.9 (4) | H9A—C9—H9B | 107.6 |

| | | | |
|--------------|------------|---------------|------------|
| C5—C4—H4 | 121.5 | O2—C10—O3 | 123.8 (4) |
| C3—C4—H4 | 121.5 | O2—C10—C9 | 125.6 (4) |
| C4—C5—C6 | 123.6 (4) | O3—C10—C9 | 110.6 (3) |
| C4—C5—C12 | 118.7 (3) | O3—C11—H11A | 109.5 |
| C6—C5—C12 | 117.7 (3) | O3—C11—H11B | 109.5 |
| C5—C6—C1 | 117.9 (4) | H11A—C11—H11B | 109.5 |
| C5—C6—H6 | 121.1 | O3—C11—H11C | 109.5 |
| C1—C6—H6 | 121.1 | H11A—C11—H11C | 109.5 |
| O1—C7—N1 | 123.0 (4) | H11B—C11—H11C | 109.5 |
| O1—C7—C8 | 121.8 (4) | C7—N1—C1 | 128.6 (3) |
| N1—C7—C8 | 115.2 (3) | C7—N1—H1N | 119 (3) |
| C9—C8—C7 | 111.0 (3) | C1—N1—H1N | 113 (3) |
| C9—C8—H8A | 109.4 | C10—O3—C11 | 117.5 (3) |
| C6—C1—C2—C3 | -0.8 (6) | O1—C7—C8—C9 | 3.2 (6) |
| N1—C1—C2—C3 | 179.3 (4) | N1—C7—C8—C9 | -176.8 (4) |
| C1—C2—C3—C4 | 0.4 (6) | C7—C8—C9—C10 | 176.4 (4) |
| C1—C2—C3—C11 | 179.5 (3) | C8—C9—C10—O2 | 4.0 (6) |
| C2—C3—C4—C5 | -0.6 (6) | C8—C9—C10—O3 | -175.6 (4) |
| C11—C3—C4—C5 | -179.6 (3) | O1—C7—N1—C1 | -2.7 (7) |
| C3—C4—C5—C6 | 1.1 (6) | C8—C7—N1—C1 | 177.3 (4) |
| C3—C4—C5—C12 | -180.0 (3) | C2—C1—N1—C7 | -178.9 (4) |
| C4—C5—C6—C1 | -1.5 (6) | C6—C1—N1—C7 | 1.2 (6) |
| C12—C5—C6—C1 | 179.6 (3) | O2—C10—O3—C11 | 3.3 (6) |
| C2—C1—C6—C5 | 1.3 (6) | C9—C10—O3—C11 | -177.0 (4) |
| N1—C1—C6—C5 | -178.8 (4) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|----------|-------------|-------------|---------------|
| N1—H1N \cdots O2 ⁱ | 0.85 (2) | 2.17 (2) | 3.017 (4) | 172 (4) |
| C4—H4 \cdots O1 ⁱⁱ | 0.93 | 2.45 | 3.379 (5) | 174 |

Symmetry codes: (i) $-x-1/2, -y+3/2, -z+1$; (ii) $-x+1/2, y-1/2, -z+3/2$.

Fig. 1

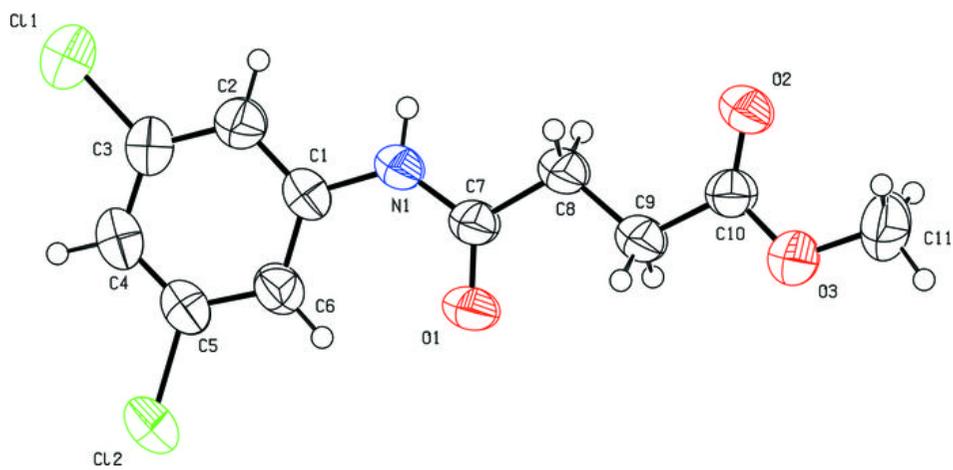


Fig. 2

