

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-(3,5-Dichloroanilino)carbonylpropionic acid

Farooq Ali Shah,^a M. Nawaz Tahir,^{b*} Saqib Ali^a and Muhammad Akram Kashmiri^c^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan,^bUniversity of Sargodha, Department of Physics, Sargodha, Pakistan, and^cGovernment College University, Department of Chemistry, Lahore, Pakistan

Correspondence e-mail: dmntahir_uos@yahoo.com

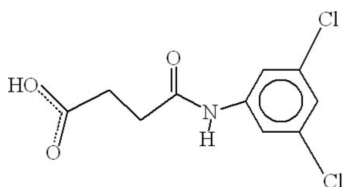
Received 30 March 2008; accepted 31 March 2008

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.125; data-to-parameter ratio = 17.3.

The crystal structure of the title compound, $\text{C}_{10}\text{H}_9\text{Cl}_2\text{NO}_3$, consists of dimers due to intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding forming an $R_2^2(8)$ ring through the carboxyl groups. These dimers are linked to each other by intermolecular hydrogen bonds between the amine group and the adjacent carbonyl O atom. A single $\text{C}-\text{Cl}\cdots\pi$ interaction is also observed between the chloro-substituted aromatic rings.

Related literature

For related literature, see: Nath *et al.* (2001); Wardell *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_9\text{Cl}_2\text{NO}_3$ $M_r = 262.08$ Triclinic, $P\bar{1}$ $a = 4.8568$ (2) Å $b = 8.6677$ (4) Å $c = 13.9038$ (8) Å $\alpha = 74.467$ (3)° $\beta = 80.495$ (2)° $\gamma = 82.712$ (3)° $V = 554.09$ (5) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.57$ mm⁻¹ $T = 296$ (2) K

0.25 × 0.12 × 0.10 mm

Data collection

Bruker Kappa APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2005)

 $T_{\min} = 0.870$, $T_{\max} = 0.945$

12157 measured reflections

2971 independent reflections

2065 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.040$ $wR(F^2) = 0.125$ $S = 1.07$

2971 reflections

172 parameters

Only H-atom coordinates refined

 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O3}^{\text{i}}$	0.84 (3)	2.07 (3)	2.904 (2)	175 (2)
$\text{O1}-\text{H1}\cdots\text{O2}^{\text{ii}}$	0.92 (4)	1.74 (4)	2.658 (3)	175 (4)
$\text{C7}-\text{Cl1}\cdots\text{Cg}^{\text{iii}}$	1.74 (1)	3.54 (1)	4.033 (2)	93 (1)

Symmetry codes: (i) $x + 1, y, z$; (ii) $-x + 1, -y, -z$; (iii) $x - 1, y, z$. Cg is the centroid of atoms C5–C10.

Data collection: APEX2 (Bruker, 2007); cell refinement: APEX2; data reduction: SAINT (Bruker, 2007); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

The authors acknowledge the Higher Education Commission, Islamabad, Pakistan, for funding the purchase of the diffractometer. Dr Saqib Ali is also grateful to the PSF for financial support under project No. PSF/R&D/C-QU/Chem(270).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2109).

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supplementary materials

Acta Cryst. (2008). E64, o787 [doi:10.1107/S1600536808008556]

3-(3,5-Dichloroanilincarbonyl)propionic acid

F. A. Shah, M. N. Tahir, S. Ali and M. A. Kashmiri

Comment

Carboxylic acids catch the interest of people due to wide use of their metal complexes in biological and industrial field. On the other hand amino acids are one of the best sources to formulate the structure-activity correlation of metal derivatives as a biologically active agent (Nath *et al.*, 2001) and widen the scope of investigation on the coordination behavior of the ligand in biological system. The title compound (I) has been prepared for complexation with different metals.

The structure of 3-(3-Nitrophenylaminocarbonyl)-propionic acid (Wardell *et al.*, 2006) has been published. The title compound have replacement of 3-nitro with Cl and also an additional Cl-atom at 5-position of benzene ring. Therefore, the bond distances and packing of (I) is being compared with the mentioned reported structure. In (I) the C=O bond distances for carboxylate and carbonyl group have values of (C1=O2: 1.219 (3) Å) and (C4=O3: 1.225 (2) Å) in comparison to 1.223 (2) and 1.2214 (17) Å, respectively. The C—N bond distances are comparable within experimental errors. In both compounds similar intermolecular H-bonding (Table 1, Fig. 2) has been observed. The dihedral angle between the aromatic ring A(C5—C10) and (C1,C2,C3,O1,O2) have a value of 82.24 (8)°, whereas with (N1,C3,C4,O3) its value is 44.42 (12)°. The value of dihedral angle between (C1,C2,C3,O1,O2) and (N1,C3,C4,O3) is 38.36 (13)°. There exist a single C—Cl \cdots π interaction at a distance of 3.5398 (11) Å [C7—Cl1 \cdots CgAⁱⁱⁱ: symmetry code iii = -1 + x, y, z].

Experimental

3,5-Dichloroaniline (16.2 g, 0.1 mole) and succinic anhydride (10 g, 0.1 mole) were dissolved in glacial acetic acid separately and mixed. The mixed solution was stirred at room temperature for 24 h. The precipitated material was filtered, washed with distilled water and dried at 413–423 K. The title compound (I) was obtained by recrystallizing the dried product using acetone. (Yield: 90%, m.p. 437 K).

Figures

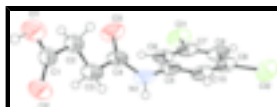


Fig. 1. ORTEP-3 for Windows (Farrugia, 1997) drawing of the title compound, C₁₀H₉Cl₂NO₃ with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii.

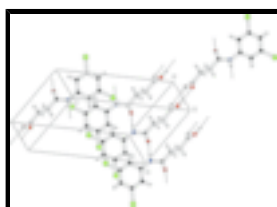


Fig. 2. The unit cell packing of (I) (Spek, 2003), showing the dimeric nature and the linkage of dimers.

3-(3,5-Dichloroanilincarbonyl)propionic acid

Crystal data

$C_{10}H_9Cl_2NO_3$	$Z = 2$
$M_r = 262.08$	$F_{000} = 268$
Triclinic, $P\bar{1}$	$D_x = 1.571 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 4.8568 (2) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 8.6677 (4) \text{ \AA}$	Cell parameters from 2971 reflections
$c = 13.9038 (8) \text{ \AA}$	$\theta = 1.5\text{--}29.2^\circ$
$\alpha = 74.467 (3)^\circ$	$\mu = 0.58 \text{ mm}^{-1}$
$\beta = 80.495 (2)^\circ$	$T = 296 (2) \text{ K}$
$\gamma = 82.712 (3)^\circ$	Needle, colourless
$V = 554.09 (5) \text{ \AA}^3$	$0.25 \times 0.12 \times 0.10 \text{ mm}$

Data collection

Bruker KappaAPEXII CCD diffractometer	2971 independent reflections
Radiation source: fine-focus sealed tube	2065 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.027$
Detector resolution: $7.4 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 29.2^\circ$
$T = 296(2) \text{ K}$	$\theta_{\text{min}} = 1.5^\circ$
ω scans	$h = -6 \rightarrow 6$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -11 \rightarrow 11$
$T_{\text{min}} = 0.870$, $T_{\text{max}} = 0.945$	$l = -19 \rightarrow 18$
12157 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	Only H-atom coordinates refined
$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.2158P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
2971 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
172 parameters	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$
	Extinction correction: none

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	-0.50817 (12)	1.03658 (7)	0.31192 (6)	0.0754 (2)
C12	0.18783 (13)	0.70434 (7)	0.57110 (5)	0.0704 (2)
O1	0.1715 (4)	0.0213 (3)	0.07439 (17)	0.0910 (7)
H1	0.281 (8)	-0.046 (4)	0.039 (3)	0.109*
O2	0.5114 (3)	0.1840 (3)	0.01798 (15)	0.0829 (6)
O3	-0.1931 (3)	0.5285 (3)	0.18654 (16)	0.0814 (6)
N1	0.2023 (3)	0.5827 (3)	0.23147 (16)	0.0607 (5)
H1A	0.377 (6)	0.563 (3)	0.222 (2)	0.073*
C1	0.2819 (4)	0.1536 (4)	0.06543 (17)	0.0629 (7)
C2	0.1014 (4)	0.2656 (4)	0.1188 (2)	0.0636 (7)
H2A	-0.054 (6)	0.295 (3)	0.089 (2)	0.076*
H2B	0.031 (6)	0.202 (3)	0.186 (2)	0.076*
C3	0.2413 (4)	0.4095 (4)	0.1194 (2)	0.0653 (7)
H3A	0.413 (6)	0.381 (3)	0.139 (2)	0.078*
H3B	0.275 (6)	0.480 (3)	0.051 (2)	0.078*
C4	0.0626 (4)	0.5118 (3)	0.18182 (18)	0.0588 (6)
C5	0.0789 (4)	0.6763 (3)	0.29919 (18)	0.0525 (5)
C6	-0.1369 (4)	0.7959 (3)	0.2744 (2)	0.0558 (5)
H6	-0.196 (5)	0.813 (3)	0.208 (2)	0.067*
C7	-0.2454 (4)	0.8838 (2)	0.3433 (2)	0.0556 (6)
C8	-0.1529 (4)	0.8582 (2)	0.4348 (2)	0.0565 (6)
H8	-0.234 (6)	0.918 (3)	0.4805 (19)	0.068*
C9	0.0627 (4)	0.7390 (2)	0.45673 (19)	0.0529 (5)
C10	0.1799 (4)	0.6482 (2)	0.39000 (19)	0.0534 (5)
H10	0.323 (5)	0.569 (3)	0.4041 (18)	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0467 (3)	0.0511 (3)	0.1229 (6)	0.0104 (2)	-0.0224 (3)	-0.0129 (3)
C12	0.0688 (4)	0.0548 (3)	0.0942 (5)	-0.0021 (3)	-0.0275 (3)	-0.0220 (3)
O1	0.0561 (10)	0.1287 (19)	0.1042 (16)	-0.0241 (11)	0.0243 (10)	-0.0704 (14)

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O2	0.0471 (9)	0.1186 (16)	0.0902 (13)	-0.0130 (9)	0.0204 (9)	-0.0543 (12)
O3	0.0227 (6)	0.1166 (16)	0.1185 (15)	0.0053 (8)	-0.0064 (8)	-0.0601 (13)
N1	0.0212 (7)	0.0772 (13)	0.0841 (14)	0.0006 (7)	-0.0002 (7)	-0.0273 (11)
C1	0.0344 (9)	0.110 (2)	0.0533 (13)	-0.0055 (11)	-0.0032 (9)	-0.0381 (13)
C2	0.0293 (9)	0.104 (2)	0.0631 (15)	-0.0026 (10)	-0.0004 (9)	-0.0360 (14)
C3	0.0273 (9)	0.0925 (19)	0.0749 (16)	0.0001 (10)	0.0053 (9)	-0.0290 (14)
C4	0.0240 (8)	0.0786 (15)	0.0716 (15)	-0.0001 (8)	-0.0007 (8)	-0.0208 (12)
C5	0.0242 (7)	0.0512 (11)	0.0789 (15)	-0.0053 (7)	0.0001 (8)	-0.0146 (10)
C6	0.0306 (8)	0.0564 (13)	0.0747 (15)	-0.0046 (8)	-0.0058 (9)	-0.0074 (11)
C7	0.0301 (8)	0.0381 (10)	0.0934 (17)	-0.0015 (7)	-0.0078 (9)	-0.0086 (10)
C8	0.0404 (10)	0.0379 (11)	0.0926 (18)	-0.0046 (8)	-0.0083 (10)	-0.0187 (11)
C9	0.0406 (9)	0.0367 (10)	0.0822 (15)	-0.0072 (8)	-0.0133 (9)	-0.0118 (10)
C10	0.0337 (9)	0.0388 (10)	0.0864 (17)	-0.0017 (7)	-0.0114 (9)	-0.0125 (10)

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

Cl1—C7	1.737 (2)	C3—C4	1.503 (3)
Cl2—C9	1.734 (2)	C3—H3A	0.91 (3)
O1—C1	1.295 (3)	C3—H3B	0.99 (3)
O1—H1	0.92 (4)	C5—C10	1.380 (3)
O2—C1	1.219 (3)	C5—C6	1.394 (3)
O3—C4	1.225 (2)	C6—C7	1.378 (3)
N1—C4	1.343 (3)	C6—H6	0.98 (3)
N1—C5	1.415 (3)	C7—C8	1.372 (3)
N1—H1A	0.84 (3)	C8—C9	1.386 (3)
C1—C2	1.488 (3)	C8—H8	0.93 (3)
C2—C3	1.497 (4)	C9—C10	1.381 (3)
C2—H2A	0.90 (3)	C10—H10	0.92 (3)
C2—H2B	0.97 (3)		
C1—O1—H1	113 (2)	O3—C4—C3	121.8 (2)
C4—N1—C5	125.67 (16)	N1—C4—C3	115.51 (17)
C4—N1—H1A	114.4 (19)	C10—C5—C6	120.6 (2)
C5—N1—H1A	119.8 (19)	C10—C5—N1	118.47 (19)
O2—C1—O1	123.4 (2)	C6—C5—N1	121.0 (2)
O2—C1—C2	123.1 (3)	C7—C6—C5	118.0 (2)
O1—C1—C2	113.5 (2)	C7—C6—H6	124.9 (15)
C1—C2—C3	113.79 (18)	C5—C6—H6	117.1 (15)
C1—C2—H2A	107.1 (18)	C8—C7—C6	123.18 (19)
C3—C2—H2A	111.1 (18)	C8—C7—Cl1	118.42 (18)
C1—C2—H2B	107.3 (16)	C6—C7—Cl1	118.39 (19)
C3—C2—H2B	114.0 (16)	C7—C8—C9	117.3 (2)
H2A—C2—H2B	103 (2)	C7—C8—H8	121.1 (17)
C2—C3—C4	112.22 (18)	C9—C8—H8	121.6 (17)
C2—C3—H3A	111.7 (18)	C10—C9—C8	121.8 (2)
C4—C3—H3A	111.0 (17)	C10—C9—Cl2	119.54 (16)
C2—C3—H3B	110.6 (17)	C8—C9—Cl2	118.68 (19)
C4—C3—H3B	105.7 (17)	C5—C10—C9	119.2 (2)
H3A—C3—H3B	105 (2)	C5—C10—H10	118.7 (16)
O3—C4—N1	122.7 (2)	C9—C10—H10	122.1 (16)

O2—C1—C2—C3	-7.0 (4)	C5—C6—C7—C8	-0.6 (3)
O1—C1—C2—C3	172.9 (2)	C5—C6—C7—C11	178.35 (15)
C1—C2—C3—C4	-174.8 (2)	C6—C7—C8—C9	0.9 (3)
C5—N1—C4—O3	4.0 (4)	C11—C7—C8—C9	-178.06 (14)
C5—N1—C4—C3	-176.1 (2)	C7—C8—C9—C10	-0.4 (3)
C2—C3—C4—O3	-34.9 (4)	C7—C8—C9—C12	179.37 (15)
C2—C3—C4—N1	145.1 (2)	C6—C5—C10—C9	0.6 (3)
C4—N1—C5—C10	133.9 (2)	N1—C5—C10—C9	179.70 (17)
C4—N1—C5—C6	-47.0 (3)	C8—C9—C10—C5	-0.3 (3)
C10—C5—C6—C7	-0.2 (3)	C12—C9—C10—C5	179.91 (15)
N1—C5—C6—C7	-179.26 (18)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A \cdots O3 ⁱ	0.84 (3)	2.07 (3)	2.904 (2)	175 (2)
O1—H1 \cdots O2 ⁱⁱ	0.92 (4)	1.74 (4)	2.658 (3)	175 (4)
C7—C11 \cdots Cg ⁱⁱⁱ	1.737 (2)	3.5398 (11)	4.033 (2)	93.34 (7)

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

Fig. 1

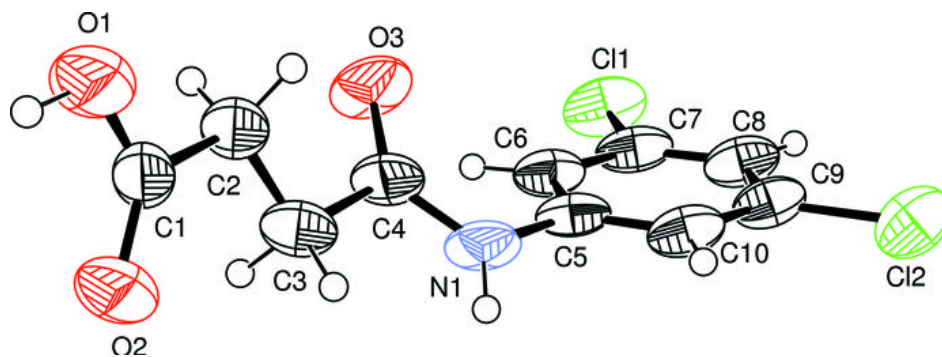


Fig. 2

