organic compounds

2-(4-Chlorobenzamido)acetic acid

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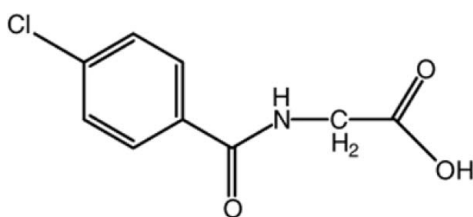Correspondence e-mail: iukhan@gcu.edu.pk, akkurt@erciyes.edu.tr

In the crystal structure of the title molecule, $C_9H_8ClNO_3$, adjacent molecules are arranged into centrosymmetric dimers through pairs of intermolecular O—H⋯O interactions. Intermolecular N—H⋯O hydrogen bonds link the dimers into a layer parallel to the bc plane. In the layer, molecules are packed in a face-to-face π-stacked arrangment, showing π–π stacking interactions between the benzene rings with a centroid–centroid distance of 3.6884 (8) Å.

Related literature

For crystallographic studies of benzamide derivatives, see: Donnelly *et al.* (2008); Mugnoli *et al.* (1991); Stensland *et al.* (1995). For standard bond lengths, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_9H_8ClNO_3$	$a = 10.5035$ (2) Å
$M_r = 213.61$	$b = 13.2105$ (4) Å
Monoclinic, $P2_1/c$	$c = 7.1226$ (2) Å

$\beta = 102.203$ (1)°	$\mu = 0.37$ mm^{-1}
$V = 965.98$ (4) Å^3	$T = 296$ K
$Z = 4$	$0.36 \times 0.21 \times 0.13$ mm
Mo $K\alpha$ radiation	

Data collection

Bruker APEXII CCD diffractometer	2365 independent reflections
9027 measured reflections	1627 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.098$	
$S = 1.02$	
2365 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
133 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
2 restraints	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯O2^i	0.83 (2)	2.06 (2)	2.8491 (19)	160 (2)
O3—H1O⋯O1^{ii}	0.83 (1)	1.85 (2)	2.6613 (16)	165 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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supporting information

2-(4-Chlorobenzamido)acetic acid

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S1. Comment

Benzamide is originally a derivative of benzoic acid. Some benzamide derivatives are in use as Analgesics (Ethenzamide, Salicylamide), Antiemetics/Prokinetics (Alizapride, Bromopride, Cinitapride, Cisapride, Clebopride) and Antipsychotics (Amisulpride, Nemonapride, Remoxipride, Sulpiride, Sultopride). Other benzamides are being prepared and there crystallographic studies are done (Donnelly *et al.*, 2008; Stensland *et al.*, 1995; Mugnoli *et al.*, 1991). The given benzamide derivative was prepared using the simple route using water as solvent.

In the title compound (I), (Fig. 1), the bond lengths and bond angles are in agreement with those reported in the literature (Allen *et al.*, 1987). The C1—C6—C7—O1, C1—C6—C7—N1, O1—C7—N1—C8, N1—C8—C9—O2 and N1—C8—C9—O3 torsion angles are 20.2 (2), -159.08 (14), -3.2 (2), 17.7 (2) and -163.65 (14)°, respectively.

In the crystal structure, the molecules adopt a face-to-face π-stacked packing arrangement showing π–π stacking interactions involving the benzene rings [$Cg1\cdots Cg1^i$ = 3.6884 (8) Å; symmetry code: (i) x, 3/2 - y, -1/2 + z; $Cg1$ is a centroid of the benzene ring (C1–C6)].

S2. Experimental

The calculated amount of glycine (0.5 g, 6.494 mmol) was carefully weighed and transferred to R.B.F (50 ml) containing 10 ml of distilled water. The pH of the water was maintained at 8 with 10% Sod. Carbonate solution which results in the complete dissolution of glycine. Then 4-chlorobenzoyl chloride (0.83 ml, 6.494 mmol) was added and pH was maintained at 8. After 3.5 h the TLC showed the completion of reaction giving a single spot of the product. The reaction mixture was then acidified with 3 N HCl up to pH 3 which resulted in the insoluble precipitate formation. Precipitates were filtered, washed, dried and then crystallized in methanol.

S3. Refinement

In the last cycles of the refinement, 2 reflections (1 0 0) and (0 2 0) were eliminated due to being poorly measured in the vicinity of the beam stop. H atoms bounded to C atoms were positioned geometrically with C—H = 0.93 and 0.97 Å, and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C). The hydroxyl and amine H atoms were located in a difference Fourier map, and refined with the distance restraints N—H = 0.86 (2) Å and O—H = 0.82 (2) Å. Their isotropic displacement parameters were set to be 1.2U_{eq}(N) for amine and 1.5U_{eq}(O) for hydroxyl.
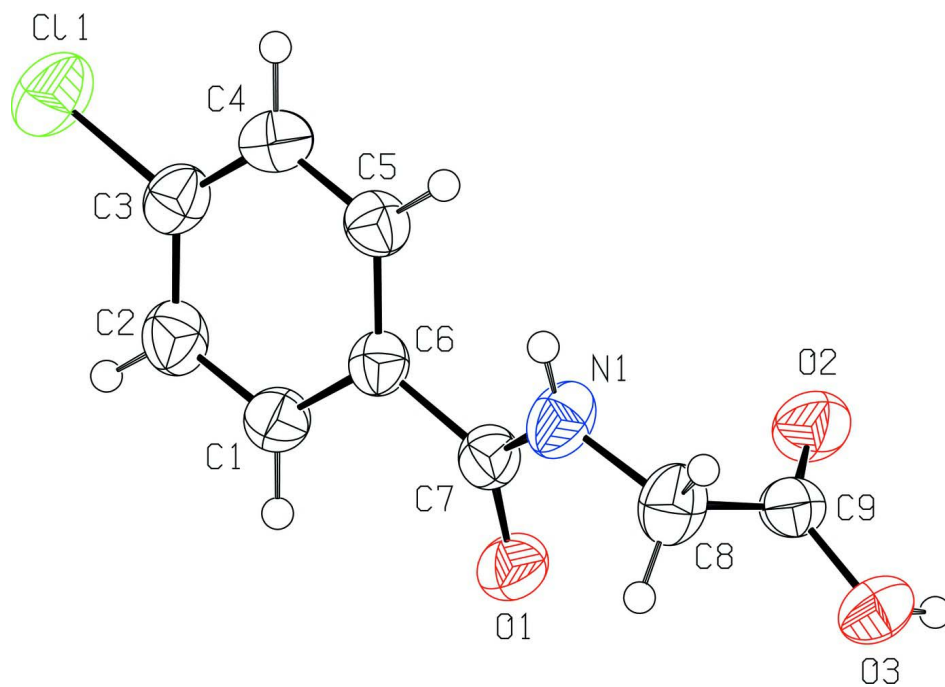
Figure 1

The title molecule with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.
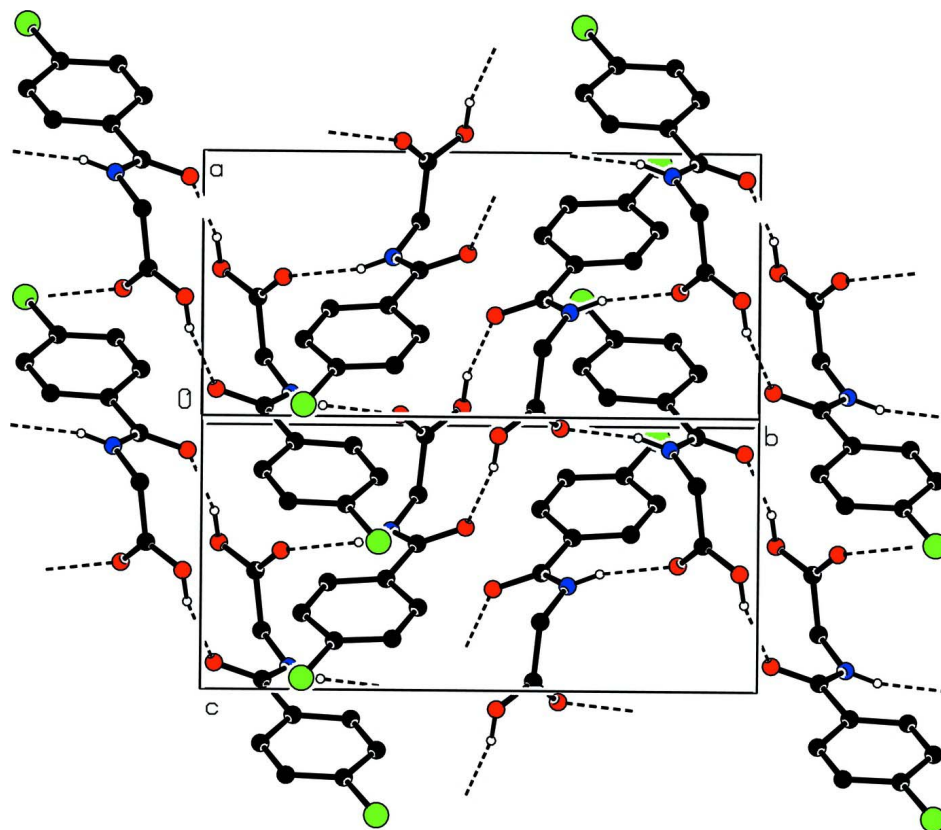
Figure 2

View of the centrosymmetric dimers forming through a pair of O—H···O interactions which are connected to each other through intermolecular N—H···O interactions. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

2-(4-Chlorobenzamido)acetic acid

Crystal data

$C_9H_8ClNO_3$	$F(000) = 440$
$M_r = 213.61$	$D_x = 1.469$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3071 reflections
$a = 10.5035$ (2) Å	$\theta = 2.5–26.5°$
$b = 13.2105$ (4) Å	$\mu = 0.37$ mm^{-1}
$c = 7.1226$ (2) Å	$T = 296$ K
$\beta = 102.203$ (1)°	Needle, colourless
$V = 965.98$ (4) Å^3	$0.36 \times 0.21 \times 0.13$ mm
$Z = 4$	

Data collection

Bruker APEXII CCD diffractometer	1627 reflections with $I > 2\sigma(I)$
Radiation source: sealed tube	$R_{int} = 0.028$
Graphite monochromator	$\theta_{max} = 28.3°$, $\theta_{min} = 3.3°$
φ and ω scans	$h = -13 \rightarrow 13$
9027 measured reflections	$k = -17 \rightarrow 17$
2365 independent reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.098$	
$S = 1.02$	H atoms treated by a mixture of independent and constrained refinement
2365 reflections	
133 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.1658P]$
2 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.21$ e Å^{-3}
	$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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}
Cl1	1.15861 (4)	0.81838 (4)	0.19567 (7)	0.0678 (2)
O1	0.65691 (11)	0.52525 (8)	0.26411 (16)	0.0518 (4)
O2	0.46720 (11)	0.64368 (9)	0.49155 (17)	0.0576 (4)
O3	0.31367 (11)	0.53263 (9)	0.37136 (17)	0.0519 (4)
N1	0.53959 (13)	0.65903 (11)	0.1375 (2)	0.0515 (5)
C1	0.88594 (15)	0.60968 (12)	0.1921 (2)	0.0457 (5)
C2	1.00353 (16)	0.65563 (13)	0.1908 (2)	0.0499 (6)
C3	1.00957 (15)	0.75964 (13)	0.1880 (2)	0.0453 (5)
C4	0.90087 (16)	0.81827 (12)	0.1838 (2)	0.0465 (5)
C5	0.78344 (15)	0.77170 (11)	0.1832 (2)	0.0423 (5)
C6	0.77497 (14)	0.66710 (11)	0.1887 (2)	0.0379 (4)
C7	0.65311 (14)	0.61215 (11)	0.2002 (2)	0.0407 (5)
C8	0.41924 (16)	0.61044 (15)	0.1527 (2)	0.0539 (6)
C9	0.40478 (14)	0.59869 (11)	0.3568 (2)	0.0411 (5)
H1	0.88100	0.53940	0.19530	0.0550*
H1N	0.538 (2)	0.7167 (13)	0.092 (3)	0.0810*
H1O	0.315 (2)	0.5241 (19)	0.487 (2)	0.1020*
H2	1.07760	0.61690	0.19170	0.0600*
H4	0.90650	0.88850	0.18130	0.0560*
H5	0.70930	0.81080	0.17910	0.0510*
H8A	0.41540	0.54410	0.09330	0.0650*
H8B	0.34680	0.65000	0.08250	0.0650*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0478 (3)	0.0785 (4)	0.0752 (3)	−0.0209 (2)	0.0086 (2)	−0.0017 (2)
O1	0.0541 (7)	0.0422 (7)	0.0616 (7)	0.0003 (5)	0.0180 (5)	0.0100 (5)
O2	0.0571 (8)	0.0578 (7)	0.0566 (7)	−0.0178 (6)	0.0089 (6)	−0.0106 (6)
O3	0.0439 (6)	0.0499 (7)	0.0600 (7)	−0.0122 (5)	0.0067 (5)	0.0017 (6)
N1	0.0389 (8)	0.0556 (9)	0.0604 (9)	0.0027 (6)	0.0113 (6)	0.0169 (7)
C1	0.0449 (9)	0.0391 (8)	0.0547 (9)	0.0039 (7)	0.0141 (7)	0.0045 (7)
C2	0.0392 (9)	0.0546 (10)	0.0566 (10)	0.0065 (7)	0.0119 (7)	0.0016 (7)
C3	0.0414 (9)	0.0532 (10)	0.0401 (8)	−0.0082 (7)	0.0060 (6)	−0.0003 (7)
C4	0.0524 (10)	0.0391 (8)	0.0461 (9)	−0.0040 (7)	0.0061 (7)	0.0011 (7)
C5	0.0431 (9)	0.0409 (8)	0.0425 (8)	0.0065 (6)	0.0081 (6)	0.0036 (6)
C6	0.0395 (8)	0.0407 (8)	0.0338 (7)	0.0019 (6)	0.0084 (6)	0.0048 (6)
C7	0.0435 (9)	0.0406 (9)	0.0394 (8)	0.0018 (6)	0.0117 (6)	0.0035 (6)
C8	0.0374 (9)	0.0698 (12)	0.0530 (10)	−0.0044 (8)	0.0065 (7)	−0.0001 (8)
C9	0.0300 (8)	0.0351 (8)	0.0568 (9)	0.0026 (6)	0.0062 (7)	−0.0004 (7)

Geometric parameters (Å, º)

Cl1—C3	1.7377 (17)	C3—C4	1.375 (2)
O1—C7	1.2325 (18)	C4—C5	1.378 (2)
O2—C9	1.1993 (19)	C5—C6	1.386 (2)
O3—C9	1.3153 (19)	C6—C7	1.489 (2)
O3—H1O	0.829 (14)	C8—C9	1.501 (2)
N1—C8	1.442 (2)	C1—H1	0.9300
N1—C7	1.334 (2)	C2—H2	0.9300
N1—H1N	0.827 (18)	C4—H4	0.9300
C1—C2	1.378 (2)	C5—H5	0.9300
C1—C6	1.387 (2)	C8—H8A	0.9700
C2—C3	1.376 (2)	C8—H8B	0.9700
C9—O3—H1O	108.0 (16)	N1—C8—C9	112.86 (13)
C7—N1—C8	120.26 (14)	O2—C9—O3	123.33 (14)
C7—N1—H1N	120.1 (15)	O2—C9—C8	125.00 (14)
C8—N1—H1N	119.6 (15)	O3—C9—C8	111.66 (13)
C2—C1—C6	120.68 (15)	C2—C1—H1	120.00
C1—C2—C3	118.96 (15)	C6—C1—H1	120.00
C2—C3—C4	121.49 (15)	C1—C2—H2	120.00
Cl1—C3—C2	119.31 (13)	C3—C2—H2	121.00
Cl1—C3—C4	119.18 (13)	C3—C4—H4	120.00
C3—C4—C5	119.17 (15)	C5—C4—H4	120.00
C4—C5—C6	120.53 (14)	C4—C5—H5	120.00
C1—C6—C5	119.16 (14)	C6—C5—H5	120.00
C1—C6—C7	117.49 (13)	N1—C8—H8A	109.00
C5—C6—C7	123.29 (14)	N1—C8—H8B	109.00
N1—C7—C6	118.25 (13)	C9—C8—H8A	109.00
O1—C7—N1	120.86 (14)	C9—C8—H8B	109.00

O1—C7—C6	120.89 (13)	H8A—C8—H8B	108.00
C8—N1—C7—C6	−177.54 (13)	C3—C4—C5—C6	0.6 (2)
C7—N1—C8—C9	67.7 (2)	C4—C5—C6—C7	176.22 (13)
C8—N1—C7—O1	3.2 (2)	C4—C5—C6—C1	−0.9 (2)
C2—C1—C6—C7	−177.03 (13)	C1—C6—C7—O1	20.2 (2)
C2—C1—C6—C5	0.3 (2)	C1—C6—C7—N1	−159.08 (14)
C6—C1—C2—C3	0.6 (2)	C5—C6—C7—O1	−156.98 (14)
C1—C2—C3—Cl1	177.54 (11)	C5—C6—C7—N1	23.8 (2)
C1—C2—C3—C4	−0.9 (2)	N1—C8—C9—O2	16.7 (2)
Cl1—C3—C4—C5	−178.18 (11)	N1—C8—C9—O3	−163.65 (14)
C2—C3—C4—C5	0.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2[i]	0.83 (2)	2.06 (2)	2.8491 (19)	160 (2)
O3—H1*O*···O1[ii]	0.83 (1)	1.85 (2)	2.6613 (16)	165 (2)

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