

2-(4-Chlorobenzamido)acetic acid

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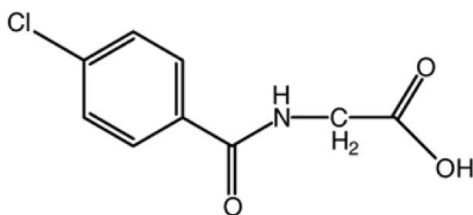
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 17.8.

In the crystal structure of the title molecule, $\text{C}_9\text{H}_8\text{ClNO}_3$, adjacent molecules are arranged into centrosymmetric dimers through pairs of intermolecular $\text{O}-\text{H}\cdots\text{O}$ interactions. Intermolecular $\text{N}-\text{H}\cdots\text{O}$ 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 arrangement, 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

$\text{C}_9\text{H}_8\text{ClNO}_3$
 $M_r = 213.61$
 Monoclinic, $P2_1/c$

$a = 10.5035$ (2) Å
 $b = 13.2105$ (4) Å
 $c = 7.1226$ (2) Å

$\beta = 102.203$ (1)°
 $V = 965.98$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.37$ mm⁻¹
 $T = 296$ K
 $0.36 \times 0.21 \times 0.13$ mm

Data collection

Bruker APEXII CCD
 diffractometer
 9027 measured reflections

2365 independent reflections
 1627 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.098$
 $S = 1.02$
 2365 reflections
 133 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^{\text{i}}$	0.83 (2)	2.06 (2)	2.8491 (19)	160 (2)
$\text{O3}-\text{H1O}\cdots\text{O1}^{\text{ii}}$	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: *S SAINT* (Bruker, 2007); data reduction: *S 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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2688).

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supplementary materials

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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 their 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)].

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.

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 bonded 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.

Figures

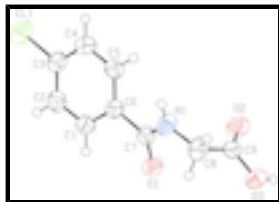


Fig. 1. The title molecule with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

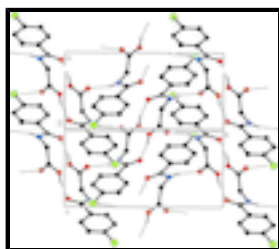


Fig. 2. View of the centrosymmetric dimers forming through a pair of O—H...O interactions which are connected to each other through intermolecular N—H...O interactions. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

2-(4-Chlorobenzamido)acetic acid

Crystal data

$C_9H_8ClNO_3$

$M_r = 213.61$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.5035 (2) \text{ \AA}$

$b = 13.2105 (4) \text{ \AA}$

$c = 7.1226 (2) \text{ \AA}$

$\beta = 102.203 (1)^\circ$

$V = 965.98 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 440$

$D_x = 1.469 \text{ Mg m}^{-3}$

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

Cell parameters from 3071 reflections

$\theta = 2.5\text{--}26.5^\circ$

$\mu = 0.37 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Needle, colourless

$0.36 \times 0.21 \times 0.13 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: sealed tube
graphite

φ and ω scans

9027 measured reflections

2365 independent reflections

1627 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 3.3^\circ$

$h = -13 \rightarrow 13$

$k = -17 \rightarrow 17$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.098$

$S = 1.02$

2365 reflections

133 parameters

2 restraints

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 0.1658P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

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

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	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 (\AA , $^\circ$)

C11—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
C11—C3—C2	119.31 (13)	C3—C2—H2	121.00
C11—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—C11	177.54 (11)	C5—C6—C7—N1	23.8 (2)
C1—C2—C3—C4	-0.9 (2)	N1—C8—C9—O2	16.7 (2)
C11—C3—C4—C5	-178.18 (11)	N1—C8—C9—O3	-163.65 (14)
C2—C3—C4—C5	0.3 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O2 ⁱ	0.83 (2)	2.06 (2)	2.8491 (19)	160 (2)
O3—H1O \cdots O1 ⁱⁱ	0.83 (1)	1.85 (2)	2.6613 (16)	165 (2)

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

Fig. 1

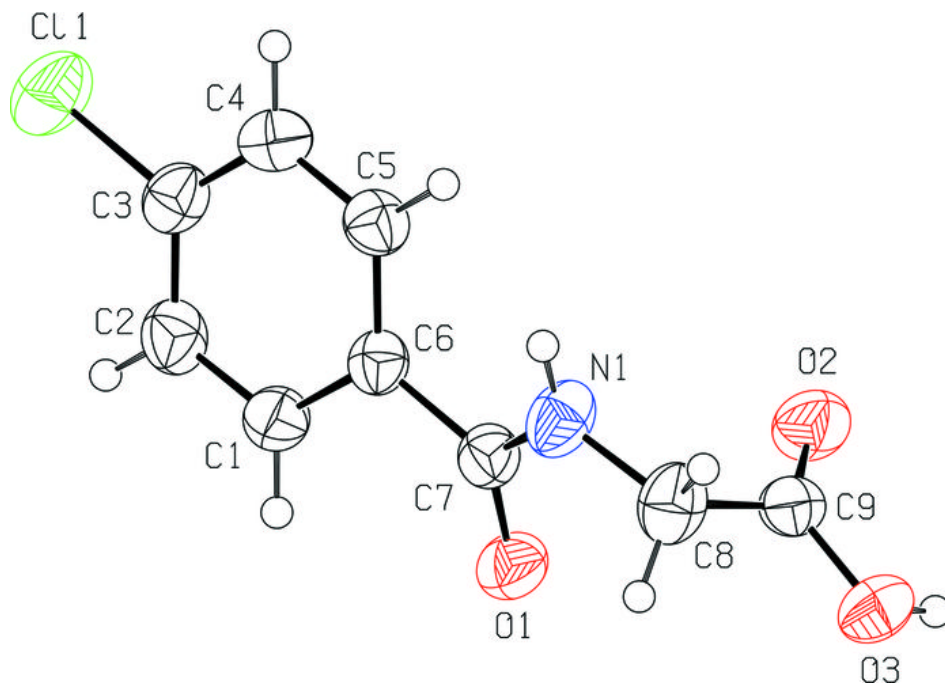


Fig. 2

