

S. Ramaswamy,^a B. Sridhar,^b V. Ramakrishnan^a and R. K. Rajaram^{b*}

^aLaser Laboratory, School of Physics, Madurai Kamaraj University, Madurai 625 021, India., and ^bDepartment of Physics, Madurai Kamaraj University, Madurai 625 021, India.

Correspondence e-mail: sshiya@yahoo.com

Key indicators

Single-crystal X-ray study
 T = 293 K
 Mean $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$
 Disorder in solvent or counterion
 R factor = 0.056
 wR factor = 0.161
 Data-to-parameter ratio = 7.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

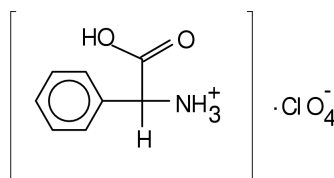
D-Phenylglycinium perchlorate

In the title compound, $\text{C}_8\text{H}_{10}\text{NO}_2^+\cdot\text{ClO}_4^-$, the phenylglycinium cation is linked to the perchlorate anion through a normal $\text{O}-\text{H}\cdots\text{O}$ [2.78 (2) Å] hydrogen bond. The phenylglycinium cation and perchlorate anion are also held together by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. Chelated three-centered hydrogen bonds are observed involving the amino-N atom and the perchlorate-O atoms. The molecular aggregation of the hydrophilic zone at $z = \frac{1}{4}$ is sandwiched between two hydrophobic zones.

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Comment

D-Phenylglycine is an important constituent in the production of semisynthetic penicillins and cephalosporins. Its derivatives are used in the synthesis of antitumor drugs and other pharmacological applications (Satyam *et al.*, 1996; Jayasinghe *et al.*, 1994). The crystal structures of D-phenylglycine hydrochloride (Ravichandran *et al.*, 1998) and bis(D-phenylglycinium) sulfate monohydrate (Srinivasan *et al.*, 2001a) have been reported. In the present study, the structure of D-phenylglycine recrystallized from perchloric acid has been investigated.



(I)

The asymmetric unit of the title compound, (I), consists of a protonated phenylglycinium cation and a perchlorate anion. In the phenylglycinium residue, the torsion angle $\text{O1A}-\text{C11}-\text{C12}-\text{N11}$, which is a measure of the relative orientation of the carboxyl group and the amino-N atom, is $16.1 (6)^\circ$, thereby indicating that the orientation of the carboxyl group is influenced by the phenyl substitution at the C^α atom. The corresponding angles in α -glycine (Marsh, 1958) and diglycine hydrochloride (Natarajan *et al.*, 1992) are found to be $19.1, 0.3$ and 16.5° , respectively. The branched-chain conformation angles χ^{11} and χ^{12} are $94.3 (6)$ and $-84.4 (5)^\circ$, respectively, indicating a folded conformation. The perchlorate anion has orientational disorder leading to considerable variations in the $\text{Cl}-\text{O}$ bond distances and the tetrahedral symmetry of the molecule.

The H atom of the perchloric acid links the D-phenylglycinium residues by a normal $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond [$\text{O1B}-\text{H12}\cdots\text{O3}(1+x, 1+y, z)$ 2.78 (2) Å]. All the O atoms of the perchlorate anion are involved in hydrogen bonding. A

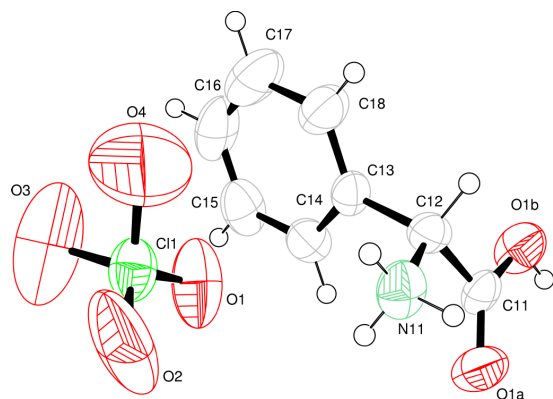


Figure 1
The molecular structure of the title compound, (I), showing the atomic numbering scheme and 50% probability displacement ellipsoids. (Johnson, 1976). O atoms for only one disordered site are shown.

normal hydrogen bond is observed between the amino-N atom and a perchlorate-O atom [N11—H11B···O2($-x, \frac{1}{2}+y, \frac{1}{2}-z$)]. Two chelated three-centered hydrogen bonds are observed, involving the amino-N atom and perchlorate-O atoms [N11—H11A···O2($1-x, \frac{1}{2}+y, \frac{1}{2}-z$) and N11—H11A···O4'($1-x, \frac{1}{2}+y, \frac{1}{2}-z$); N11—H11C···O1 and N11—H11C···O4'] (Jeffrey & Saenger, 1991). The packing arrangement shows a hydrophilic zone at $z = \frac{1}{4}$. The hydrophobic zone at $z = \frac{1}{2}$ is sandwiched between two hydrophilic zones at $z = \frac{1}{4}$ and $z = \frac{3}{4}$ (Fig. 2); similar situations have been reported in bis(D-phenylglycinium) sulfate monohydrate (Srinivasan *et al.*, 2001a), L-phenylalanine-nitric acid (2/1) (Srinivasan *et al.*, 2001b) and DL-phenylalaninium dihydrogen phosphate (Ravikumar *et al.*, 2001).

Experimental

The title compound, (I), was crystallized by slow evaporation from an aqueous solution of D-phenylglycine and perchloric acid in a stoichiometric ratio of 1:1.

Crystal data

$C_8H_{10}NO_2^+ClO_4^-$
 $M_r = 251.62$
 Orthorhombic, $P2_12_12_1$
 $a = 5.451$ (1) Å
 $b = 8.807$ (2) Å
 $c = 22.649$ (5) Å
 $V = 1087.3$ (4) Å³
 $Z = 4$
 $D_x = 1.537$ Mg m⁻³
 $D_m = 1.530$ Mg m⁻³

D_m measured by flotation technique using a mixture of carbon tetrachloride and xylene
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 13.6$ – 23.0°
 $\mu = 0.37$ mm⁻¹
 $T = 293$ (2) K
 Needle, colorless
 $0.55 \times 0.50 \times 0.15$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.864$, $T_{\max} = 0.947$
 1485 measured reflections
 1261 independent reflections
 1193 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 25.3^\circ$
 $h = -6 \rightarrow 6$
 $k = 0 \rightarrow 10$
 $l = 0 \rightarrow 27$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

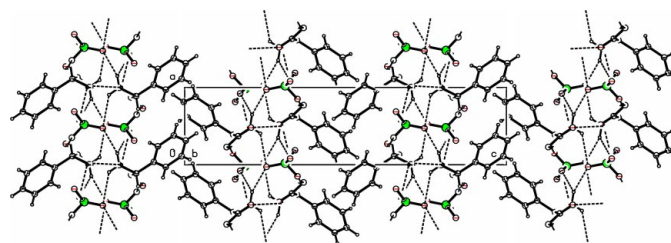


Figure 2
Packing diagram of the structure viewed down the b axis. O atoms for only one disordered site are shown.

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.161$
 $S = 1.07$
 1261 reflections
 181 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1125P)^2 + 0.6721P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³
 Absolute structure: Flack (1983)
 Flack parameter = -0.98 (16)

Table 1

Selected geometric parameters (Å, °).

O1A—C11	1.184 (5)	O1B—C11	1.287 (6)
O1A—C11—C12—N11	16.1 (6)	N11—C12—C13—C14	−84.4 (5)
N11—C12—C13—C18	94.3 (6)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1B—H12···O3 ⁱ	0.82	1.97	2.78 (2)	168
O1B—H12···O3 ⁱⁱ	0.82	1.94	2.76 (2)	178
N11—H11A···O2 ⁱⁱ	0.89	2.31	3.036 (17)	139
N11—H11A···O4 ⁱⁱⁱ	0.89	2.45	3.297 (16)	159
N11—H11A···O2 ⁱⁱ	0.89	2.60	3.29 (2)	136
N11—H11B···O2 ⁱⁱⁱ	0.89	2.22	3.076 (17)	161
N11—H11B···O2 ⁱⁱⁱ	0.89	2.37	3.23 (2)	162
N11—H11C···O1	0.89	2.47	2.906 (18)	111
N11—H11C···O4'	0.89	2.63	3.44 (2)	153

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

The perchlorate anion exhibits orientational disorder, leading to a distorted tetrahedral geometry. The site occupation factors for atoms O1/O2/O3/O4 and O1'/O2'/O3'/O4' were fixed at 0.5. All H atoms were fixed by geometric constraints using HFIX and allowed to ride on the attached atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL97*.

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