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D-Phenylglycinium nitrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.056
wR factor = 0.138
Data-to-parameter ratio = 11.4

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

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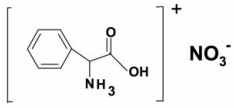
In the title compound, $C_8H_{10}NO_2^+\cdot NO_3^-$, there are two crystallographically independent cations and two anions, the pairs being related by a pseudo-inversion centre at approximately (0, $\frac{1}{4}$, $\frac{1}{4}$). The phenylglycinium cations and nitrate anions are linked to each other through strong N—H···O and O—H···O hydrogen bonds, forming a three-dimensional complex network.

Comment

Studies of organic–inorganic hybrid materials, including amino acids and various inorganic acids (Bouchouit *et al.*, 2002; Benali-Cherif, Cherouana *et al.*, 2002; Cherouana *et al.*, 2002; Benali-Cherif, Abouimrane *et al.*, 2002; Bendheif *et al.*, 2003) have received a great deal of attention in recent years because of their electrical, magnetic and optical properties (Kagan *et al.*, 1999; Hill, 1998).

Phenylglycines play an important role as starting materials in the production of semisynthetic penicillins and cephalosporins. In recent years, some phenylglycine derivatives have attracted considerable attention in the synthesis of antitumour 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 D-phenylglycinium sulfate monohydrate (Srinivasan *et al.*, 2001) have been reported, and here we describe the structure of D-phenylglycinium nitrate, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The asymmetric unit contains two crystallographically independent monoprotonated phenylglycinium cations and two nitrate anions. Both the anions and cations form layers parallel to the *ac* plane (Fig. 2). In the nitrate anions, two of the N—O distances, involving atoms O3 and O4, are slightly longer than the third, involving atom O5, while the O—N—O angles range from 117.6 (3) to 121.7 (3)°. The bond distances and angles of the phenylglycinium cation are normal.

In α-glycine (Marsh, 1958), the O1—C1—C2—N1 torsion angles are 19.1 and 0.3°, and in diglycine hydrochloride (Natarajan *et al.*, 1992), the same angle is 16.5°. In bis(D-phenylglycinium) sulfate monohydrate (Srinivasan *et al.*, 2001), these angles are 25 and 30.7 (5)°. In (I), the O1*A*—C1*A*—C2*A*—N1*A* and O1*B*—C1*B*—C2*B*—N1*B* torsion angles are 24.1 (5) and −155.8 (3)°, respectively. This clearly

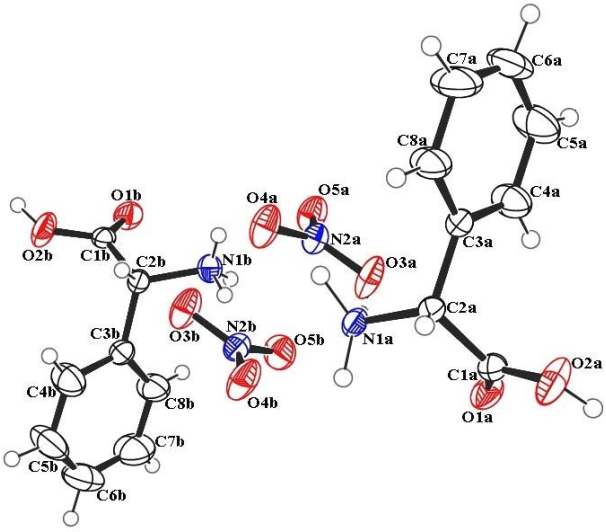


Figure 1
A view of (I), showing the atomic labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

shows that the orientation of the carboxyl group is influenced by the phenyl substitution at the C^{α} atom in glycine.

The nitrate anion in (I) plays an important role in hydrogen bonding, with all three O atoms (O3, O4 and O5) being involved, and details are given in Table 1. The phenylglycinium residue forms three strong O—H···O hydrogen bonds with the nitrate anion. The amino N atom of the phenylglycinium residue forms eight N—H···O hydrogen bonds *via* atoms O3, O4 and O5 of the nitrate anion, and two *via* the carboxyl atom O1.

Experimental

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

Crystal data

$C_8H_{10}NO_2^+\cdot NO_3^-$
M_r = 214.18
Monoclinic, $P2_1$
a = 10.4320 (3) Å
b = 5.6450 (2) Å
c = 16.7830 (2) Å
β = 94.943 (3)°
V = 984.65 (5) Å^3
Z = 4
D_x = 1.445 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3980 reflections
θ = 2.0–26.3°
μ = 0.12 mm^{-1}
T = 293 (2) K
Prism, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans
Absorption correction: none
7185 measured reflections
3752 independent reflections
2831 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 26.3°
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 6$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.138
S = 1.16
3752 reflections
328 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

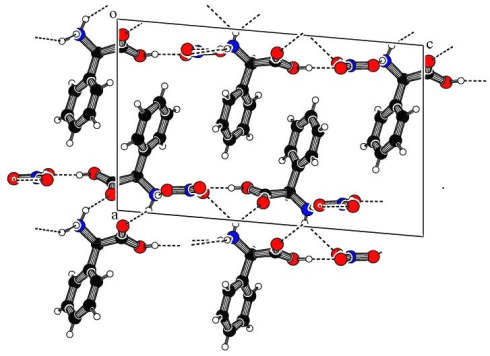


Figure 2
A diagram of the layered crystal packing in (I), viewed down the *b* axis.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2A—H···O3B	0.93	2.51	2.966 (5)	110
O2A—H···O5B	0.93	1.74	2.653 (5)	166
O2B—H′···O4A^{i}	0.87	1.77	2.593 (5)	158
N1A—H10···O1A^{ii}	1.01	2.18	2.958 (5)	133
N1A—H10···O3B^{iii}	1.01	2.48	2.982 (5)	110
N1A—H11···O4A^{iv}	0.94	2.54	3.142 (5)	122
N1A—H11···O5A^{iv}	0.94	1.91	2.842 (5)	174
N1A—H12···O3A^{v}	0.85	2.18	3.015 (5)	170
N1A—H12···O5A^{v}	0.85	2.43	2.976 (5)	123
N1B—H100···O3B^{vi}	1.00	2.59	3.158 (5)	116
N1B—H100···O4B^{vi}	1.00	1.84	2.829 (5)	168
N1B—H110···O1B^{vii}	0.94	2.11	2.906 (4)	142
N1B—H120···O4B	1.01	2.51	2.981 (5)	108
N1B—H120···O5B	1.01	1.98	2.958 (5)	163

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

Friedel pairs were merged and the $\Delta f''$ term was set to zero; the absolute configuration was known from the starting materials. All H atoms were located in difference Fourier maps and refined isotropically, but with the distances held fixed in the final cycle of refinement.

Data collection: *KappaCCD Reference Manual* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*2002 (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 (Farrugia, 1997) and *PLUTON* (Spek, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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