

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2-Carboxy-1-phenylethanaminium nitrate

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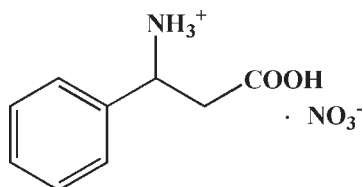
Received 17 September 2009; accepted 18 October 2009

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  
R factor = 0.080; wR factor = 0.231; data-to-parameter ratio = 17.1.

In the title salt,  $\text{C}_9\text{H}_{12}\text{NO}_2^+\cdot\text{NO}_3^-$ , the cation and anion are linked by a bifurcated  $\text{N}-\text{H}\cdots(\text{O},\text{O})$  hydrogen bond. The crystal packing is stabilized by intermolecular  $\text{N}-\text{H}\cdots\text{O}$ ,  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, which connect neighbouring cations and anions, resulting in a two-dimensional network.

## Related literature

For details of the preparation of  $\beta$ -amino acids, see: Cohen *et al.* (2002); Qu *et al.* (2004).



## Experimental

## Crystal data

$\text{C}_9\text{H}_{12}\text{NO}_2^+\cdot\text{NO}_3^-$   
 $M_r = 228.21$   
Monoclinic,  $P2_1/c$   
 $a = 6.2017$  (12) Å  
 $b = 10.313$  (2) Å  
 $c = 18.077$  (4) Å  
 $\beta = 105.36$  (3)°

$V = 1114.9$  (4) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.50 \times 0.30 \times 0.15$  mm

## Data collection

Rigaku SCXmini diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.960$ ,  $T_{\max} = 0.982$

11050 measured reflections  
2549 independent reflections  
1652 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.061$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.080$   
 $wR(F^2) = 0.231$   
 $S = 1.11$   
2549 reflections  
149 parameters

1 restraint  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.59$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C8}-\text{H8}\cdots\text{O2}^{\text{i}}$	0.93	2.56	3.414 (5)	152
$\text{C2}-\text{H2A}\cdots\text{O4}^{\text{ii}}$	0.97	2.46	3.256 (5)	140
$\text{O2}-\text{H2}\cdots\text{O5}^{\text{iii}}$	0.82	2.01	2.743 (4)	148
$\text{N1}-\text{H1C}\cdots\text{O5}^{\text{ii}}$	0.88	2.13	2.979 (4)	160
$\text{N1}-\text{H1C}\cdots\text{O4}^{\text{ii}}$	0.88	2.41	3.129 (4)	139
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{iv}}$	0.88	1.97	2.830 (4)	166
$\text{N1}-\text{H1A}\cdots\text{O4}$	0.88	2.39	3.101 (4)	138
$\text{N1}-\text{H1A}\cdots\text{O3}$	0.88	2.07	2.933 (4)	165

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

Data collection: *CrystalClear* (Rigaku 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

This work was supported by the Technical Fund Financing Projects (No. 9207042464 and 9207041482) from Southeast University to ZRQ.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2656).

## References

- Cohen, J. H., Abdel-Magid, A. F., Almond, H. R. Jr & Maryanoff, C. A. (2002). *Tetrahedron Lett.* **43**, 1977–1981.  
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Qu, Z.-R., Zhao, H., Wang, Y.-P., Wang, X.-S., Ye, Q., Li, Y.-H., Xiong, R.-G., Abrahams, B. F., Liu, Z.-G. & Xue, Z.-L. (2004). *Chem. Eur. J.* **10**, 54–60.  
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.  
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**supplementary materials**

*Acta Cryst.* (2009). E65, o2895 [ doi:10.1107/S1600536809042792 ]

## 2-Carboxy-1-phenylethanaminium nitrate

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### Comment

$\beta$ -Amino acids are important molecules due to their pharmacological properties. Recently, there has been an increased interest in the enantiomeric preparation of  $\beta$ -amino acids as precursors for the synthesis of novel biologically active compounds (Cohen *et al.*, 2002; Qu *et al.* 2004).

The title compound  $C_9H_{12}NO_2^+ \cdot NO_3^-$  exists as two independent ions linked by bifurcated  $N-H \cdots O$  hydrogen bonds (Fig. 1). The crystal structure is stabilized by intermolecular  $N-H \cdots O$ ,  $O-H \cdots O$  and  $C-H \cdots O$  hydrogen bonds (Table 1) which connect neighbouring cations and anions, resulting in a two-dimensional network (Fig. 2).

### Experimental

Benzaldehyde (1.59 g, 15 mmol), malonic acid (2.5 g, 24 mmol) and ammonium acetate (3.0 g, 39 mmol) were added in a flask under nitrogen and refluxed for 24 h yielding a white precipitate. After cooling to room temperature, the solution was filtered to yield 3-amino-3-phenylpropionic acid. This was dissolved in ethanol and nitric acid. After slowly evaporating over a period of 5 d, colorless prism-like crystals of the title compound, suitable for X-ray diffraction experiments were isolated.

### Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C, N atoms to which they are bonded, with  $C-H = 0.93$  to  $1.00$  Å,  $U_{iso}(H) = 1.2 U_{eq}(C)$ ,  $N-H = 0.88$  Å,  $U_{iso}(H) = 1.5 U_{eq}(N)$ .

### Figures

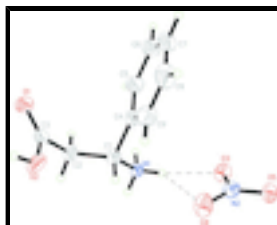


Fig. 1. The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level. Intramolecular hydrogen bonds are drawn as a dashed lines.

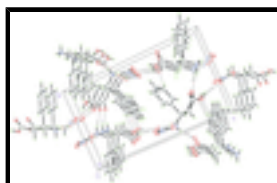


Fig. 2. Packing diagram of the title compound, showing the structure along the *b* axis. Hydrogen bonds are drawn as dashed lines.

## 2-Carboxy-1-phenylethanaminium nitrate

### Crystal data

$C_9H_{12}NO_2^+ \cdot NO_3^-$

$M_r = 228.21$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.2017$  (12) Å

$b = 10.313$  (2) Å

$c = 18.077$  (4) Å

$\beta = 105.36$  (3)°

$V = 1114.9$  (4) Å<sup>3</sup>

$Z = 4$

$F_{000} = 480$

$D_x = 1.360$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1797 reflections

$\theta = 3.1$ – $27.5$ °

$\mu = 0.11$  mm<sup>-1</sup>

$T = 293$  K

Prism, colorless

$0.50 \times 0.30 \times 0.15$  mm

### Data collection

Rigaku SCXmini  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 13.6612 pixels mm<sup>-1</sup>

$T = 293$  K

CCD\_Profile\_fitting scans

Absorption correction: multi-scan  
(CrystalClear; Rigaku, 2005)

$T_{\min} = 0.960$ ,  $T_{\max} = 0.982$

11050 measured reflections

2549 independent reflections

1652 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$

$\theta_{\max} = 27.5$ °

$\theta_{\min} = 3.1$ °

$h = -8 \rightarrow 8$

$k = -13 \rightarrow 13$

$l = -23 \rightarrow 23$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.080$

$wR(F^2) = 0.231$

$S = 1.11$

2549 reflections

149 parameters

1 restraint

Primary atom site location: structure-invariant direct  
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring  
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0829P)^2 + 1.0943P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.59$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.23$  e Å<sup>-3</sup>

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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.1549 (5)	0.1241 (2)	0.30410 (14)	0.0613 (8)
O2	0.8967 (5)	0.2333 (3)	0.34151 (16)	0.0631 (8)
H2	0.9252	0.1813	0.3771	0.095*
N1	0.7398 (5)	0.3900 (2)	0.11238 (14)	0.0382 (6)
H1A	0.6020	0.3868	0.0823	0.066 (12)*
H1B	0.7688	0.4697	0.1305	0.050 (10)*
H1C	0.8352	0.3690	0.0856	0.050 (11)*
C3	1.0254 (6)	0.2102 (3)	0.29593 (19)	0.0466 (8)
C4	0.6953 (6)	0.1620 (3)	0.14746 (19)	0.0442 (8)
C1	0.7597 (6)	0.2980 (3)	0.17750 (18)	0.0434 (8)
H1	0.6540	0.3255	0.2063	0.052*
C2	0.9930 (6)	0.3062 (3)	0.23070 (19)	0.0505 (9)
H2A	1.1015	0.2890	0.2019	0.061*
H2B	1.0190	0.3933	0.2514	0.061*
C7	0.5759 (11)	-0.0891 (4)	0.0971 (3)	0.0857 (17)
H7	0.5361	-0.1733	0.0805	0.103*
C8	0.4647 (9)	-0.0261 (5)	0.1410 (4)	0.0903 (18)
H8	0.3468	-0.0675	0.1541	0.108*
C5	0.8088 (8)	0.0973 (4)	0.1026 (2)	0.0620 (11)
H5	0.9274	0.1374	0.0892	0.074*
C9	0.5224 (7)	0.1002 (4)	0.1675 (3)	0.0649 (11)
H9	0.4448	0.1417	0.1982	0.078*
C6	0.7458 (10)	-0.0285 (5)	0.0774 (3)	0.0817 (16)
H6	0.8219	-0.0711	0.0466	0.098*
O3	0.3218 (4)	0.3769 (3)	-0.01018 (15)	0.0562 (7)
O4	0.2293 (5)	0.3826 (3)	0.09586 (15)	0.0706 (9)
O5	-0.0245 (4)	0.3499 (3)	-0.00958 (16)	0.0597 (7)
N2	0.1772 (5)	0.3697 (3)	0.02545 (17)	0.0457 (7)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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## supplementary materials

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O1	0.094 (2)	0.0400 (14)	0.0476 (15)	0.0168 (14)	0.0138 (14)	0.0082 (11)
O2	0.0649 (18)	0.0546 (16)	0.0658 (18)	0.0237 (13)	0.0100 (14)	0.0187 (13)
N1	0.0436 (15)	0.0318 (14)	0.0352 (14)	-0.0020 (11)	0.0035 (12)	0.0044 (11)
C3	0.060 (2)	0.0397 (18)	0.0345 (17)	0.0015 (16)	0.0034 (15)	-0.0028 (14)
C4	0.053 (2)	0.0337 (16)	0.0381 (17)	-0.0049 (14)	-0.0023 (15)	0.0075 (14)
C1	0.0523 (19)	0.0366 (17)	0.0377 (17)	-0.0064 (14)	0.0055 (14)	0.0062 (13)
C2	0.059 (2)	0.0430 (19)	0.043 (2)	-0.0033 (16)	0.0017 (17)	0.0061 (15)
C7	0.110 (4)	0.037 (2)	0.079 (3)	-0.010 (3)	-0.028 (3)	0.002 (2)
C8	0.081 (3)	0.058 (3)	0.110 (4)	-0.032 (3)	-0.012 (3)	0.022 (3)
C5	0.078 (3)	0.046 (2)	0.056 (2)	0.0012 (19)	0.009 (2)	0.0072 (18)
C9	0.058 (2)	0.051 (2)	0.082 (3)	-0.0113 (18)	0.012 (2)	0.011 (2)
C6	0.125 (5)	0.052 (3)	0.054 (3)	0.017 (3)	-0.001 (3)	-0.002 (2)
O3	0.0519 (15)	0.0685 (17)	0.0521 (15)	-0.0016 (12)	0.0203 (12)	0.0067 (13)
O4	0.0683 (18)	0.105 (2)	0.0385 (15)	0.0053 (16)	0.0137 (13)	0.0178 (15)
O5	0.0421 (14)	0.0672 (17)	0.0668 (18)	-0.0069 (12)	0.0093 (12)	-0.0081 (14)
N2	0.0524 (17)	0.0380 (15)	0.0472 (17)	0.0036 (12)	0.0142 (14)	0.0113 (13)

### *Geometric parameters (Å, °)*

O1—C3	1.180 (4)	C2—H2B	0.9700
O2—C3	1.312 (4)	C7—C8	1.349 (8)
O2—H2	0.8200	C7—C6	1.352 (8)
N1—C1	1.492 (4)	C7—H7	0.9300
N1—H1A	0.8837	C8—C9	1.401 (7)
N1—H1B	0.8848	C8—H8	0.9300
N1—H1C	0.8849	C5—C6	1.396 (6)
C3—C2	1.512 (5)	C5—H5	0.9300
C4—C9	1.375 (5)	C9—H9	0.9300
C4—C5	1.378 (5)	C6—H6	0.9300
C4—C1	1.519 (5)	O3—N2	1.237 (4)
C1—C2	1.513 (5)	O4—N2	1.235 (4)
C1—H1	0.9800	O5—N2	1.260 (4)
C2—H2A	0.9700		
C3—O2—H2	109.5	C3—C2—H2B	109.3
C1—N1—H1A	109.0	C1—C2—H2B	109.3
C1—N1—H1B	109.4	H2A—C2—H2B	108.0
H1A—N1—H1B	109.4	C8—C7—C6	119.2 (4)
C1—N1—H1C	110.4	C8—C7—H7	120.4
H1A—N1—H1C	109.4	C6—C7—H7	120.4
H1B—N1—H1C	109.3	C7—C8—C9	121.7 (5)
O1—C3—O2	124.4 (3)	C7—C8—H8	119.2
O1—C3—C2	122.4 (3)	C9—C8—H8	119.2
O2—C3—C2	113.2 (3)	C4—C5—C6	120.0 (5)
C9—C4—C5	119.1 (4)	C4—C5—H5	120.0
C9—C4—C1	118.9 (4)	C6—C5—H5	120.0
C5—C4—C1	122.0 (3)	C4—C9—C8	119.2 (5)
N1—C1—C2	109.3 (3)	C4—C9—H9	120.4
N1—C1—C4	110.3 (3)	C8—C9—H9	120.4
C2—C1—C4	113.3 (3)	C7—C6—C5	121.0 (5)

N1—C1—H1	107.9	C7—C6—H6	119.5
C2—C1—H1	107.9	C5—C6—H6	119.5
C4—C1—H1	107.9	O4—N2—O3	120.2 (3)
C3—C2—C1	111.5 (3)	O4—N2—O5	119.3 (3)
C3—C2—H2A	109.3	O3—N2—O5	120.5 (3)
C1—C2—H2A	109.3		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C8—H8...O2 <sup>i</sup>	0.93	2.56	3.414 (5)	152
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Fig. 1

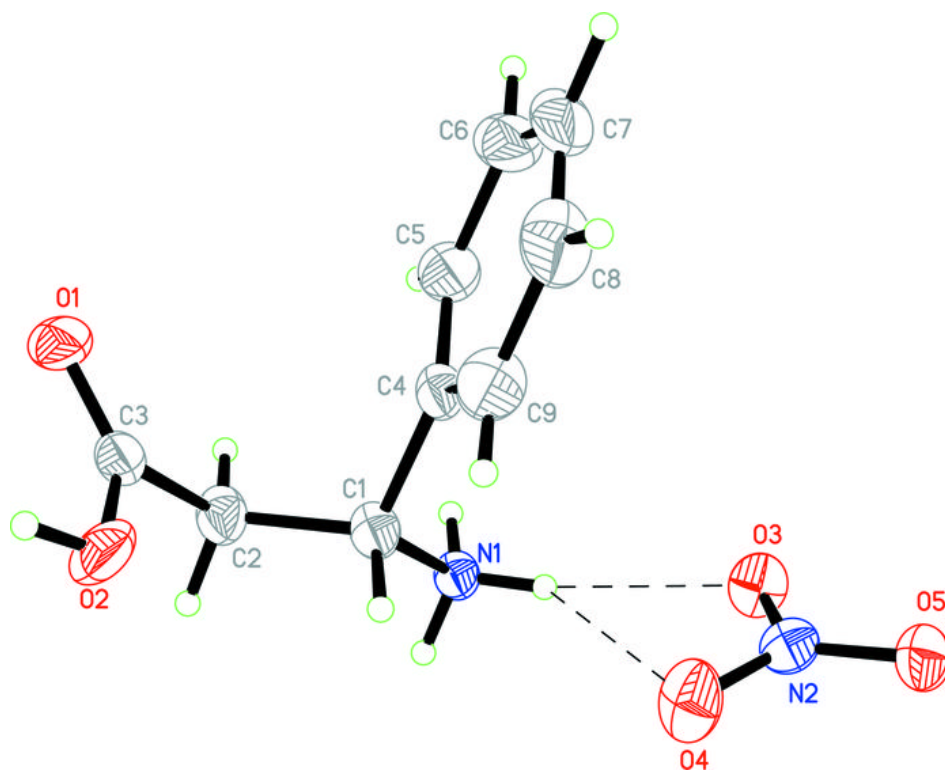




Fig. 2

