

2-Carboxy-1-phenylethanaminium perchlorate

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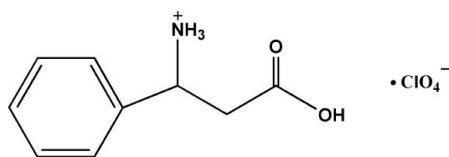
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.053; wR factor = 0.110; data-to-parameter ratio = 16.4.

In the title compound, $\text{C}_9\text{H}_{12}\text{NO}_2^+\cdot\text{ClO}_4^-$, an intramolecular $\text{N}-\text{H}\cdots\text{O}$ interaction results in the formation of a six-membered ring having a twisted chair conformation. In the crystal structure, intermolecular $\text{O}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions link the molecules into a network. A weak $\text{C}-\text{H}\cdots\pi$ interaction is also found.

Related literature

There has been an increased interest in the enantiomeric preparation of β -amino acids as precursors for the synthesis of novel biologically active compounds, see: Arki *et al.* (2004); Cohen *et al.* (2002); Zeller *et al.* (1965). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_9\text{H}_{12}\text{NO}_2^+\cdot\text{ClO}_4^-$ $V = 2237.0$ (8) Å³
 $M_r = 265.65$ $Z = 8$
 Orthorhombic, $Pbca$ $\text{Mo } K\alpha$ radiation
 $a = 6.6583$ (13) Å $\mu = 0.36$ mm⁻¹
 $b = 13.826$ (3) Å $T = 294$ K
 $c = 24.300$ (5) Å $0.45 \times 0.35 \times 0.12$ mm

Data collection

Rigaku SCXmini diffractometer 21012 measured reflections
 Absorption correction: multi-scan 2560 independent reflections
 (Blessing, 1995) 1966 reflections with $I > 2\sigma(I)$
 $T_{\min} = 0.863$, $T_{\max} = 0.957$ $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$ 156 parameters
 $wR(F^2) = 0.110$ H-atom parameters constrained
 $S = 1.10$ $\Delta\rho_{\max} = 0.27$ e Å⁻³
 2560 reflections $\Delta\rho_{\min} = -0.37$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O2}$	0.89	2.13	2.773 (3)	128
$\text{C9}-\text{H9}\cdots\text{O5}^i$	0.93	2.56	3.409 (3)	152
$\text{C3}-\text{H3}\cdots\text{O5}^i$	0.98	2.57	3.370 (3)	139
$\text{C3}-\text{H3}\cdots\text{O2}^{ii}$	0.98	2.58	3.286 (3)	129
$\text{N1}-\text{H1C}\cdots\text{O3}^{iii}$	0.89	2.05	2.892 (3)	158
$\text{N1}-\text{H1B}\cdots\text{O3}^{iv}$	0.89	2.28	3.046 (3)	144
$\text{N1}-\text{H1A}\cdots\text{O6}^v$	0.89	2.13	2.979 (3)	159
$\text{O1}-\text{H1}\cdots\text{O2}^{vi}$	0.82	2.41	3.046 (2)	135
$\text{O1}-\text{H1}\cdots\text{O4}^{vii}$	0.82	2.35	3.048 (3)	143
$\text{C8}-\text{H8}\cdots\text{Cg1}^{viii}$	0.93	2.79	3.688 (3)	162

Symmetry codes: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, z$; (ii) $x - \frac{1}{2}, y, -z + \frac{1}{2}$; (iii) $x - 1, y - 1, z$; (iv) $x - \frac{1}{2}, y - 1, -z + \frac{1}{2}$; (v) $x, y - 1, z$; (vi) $x + \frac{1}{2}, y, -z + \frac{1}{2}$; (vii) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$; (viii) $-x - 1, y + \frac{1}{2}, -z + \frac{1}{2}$. Cg1 is the centroid of the C4-C9 ring.

Data collection: *CrystalClear* (Rigaku/MSC, 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: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2675).

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supplementary materials

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2-Carboxy-1-phenylethanaminium perchlorate

X.-Z. Li, H. Li and Z.-R. Qu

Comment

β -Amino acids are important molecules, due to their pharmacological properties. Recently, there has been an increased interest in the enantiomeric preparations of β -amino acids, as precursors for the synthesis of novel biologically active compounds (Arki *et al.*, 2004; Cohen *et al.*, 2002; Zeller *et al.*, 1965). We report herein the crystal structure of the title compound.

The asymmetric unit of the title compound contains one cation and one anion (Fig.1), in which the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Ring A (C4-C9) is, of course, planar. Intramolecular N-H \cdots O interaction results in the formation of a six-membered ring B (O2/N1/C1-C3/H1B) having twisted conformation.

In the crystal structure, intermolecular O-H \cdots O, N-H \cdots O and C-H \cdots O interactions (Table 1) link the molecules into a network (Fig. 2), in which they may be effective in the stabilization of the structure. There also exists a weak C—H \cdots π interaction (Table 1).

Experimental

Under nitrogen protection, benzaldehyde (3.18 g, 30 mmol), malonic acid (5.00 g, 48 mmol) and ammonium acetate (6.00 g, 78 mmol) were added into a flask and refluxed for 10 h to yield a colorless precipitate. The crude product was obtained after filtration, then it was dissolved in ethanol/ perchloric acid (1:1), after slowly evaporating over a period of 4 d, colorless prism crystals of the title compound suitable for X-ray analysis were isolated.

Refinement

H atoms were positioned geometrically with O-H = 0.82 Å (for OH), N-H = 0.89 Å (for NH₃), C-H = 0.93, 0.98 and 0.97 Å, for aromatic, methine and methylene H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,O,N)$, where $x = 1.5$ for OH and NH₃ H and $x = 1.2$ for all other H atoms.

Figures

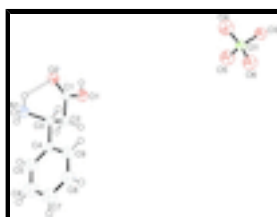


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bond is shown as dashed line.

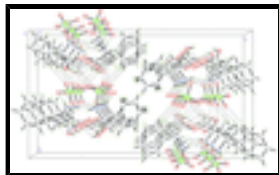


Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

2-Carboxy-1-phenylethanaminium perchlorate

Crystal data

$C_9H_{12}NO_2^+ \cdot ClO_4^-$

$M_r = 265.65$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 6.6583$ (13) Å

$b = 13.826$ (3) Å

$c = 24.300$ (5) Å

$V = 2237.0$ (8) Å³

$Z = 8$

$F_{000} = 1104$

$D_x = 1.578$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1979 reflections

$\theta = 3.1$ – 27.5°

$\mu = 0.36$ mm⁻¹

$T = 294$ K

Prism, colorless

$0.45 \times 0.35 \times 0.12$ mm

Data collection

Rigaku SCXmini
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294$ K

CCD_Profile_fitting scans

Absorption correction: multi-scan
(Blessing, 1995)

$T_{\min} = 0.863$, $T_{\max} = 0.957$

21012 measured reflections

2560 independent reflections

1966 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 3.1^\circ$

$h = -8 \rightarrow 8$

$k = -17 \rightarrow 17$

$l = -31 \rightarrow 31$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.110$

$S = 1.10$

2560 reflections

156 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 1.6729P]$

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

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

$\Delta\rho_{\text{max}} = 0.27$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.99041 (8)	0.97917 (4)	0.32015 (2)	0.03337 (17)
O1	0.9660 (3)	0.30715 (14)	0.32107 (7)	0.0428 (5)
H1	1.0262	0.3182	0.2923	0.064*
O2	0.7570 (2)	0.22973 (13)	0.26501 (6)	0.0365 (4)
O3	1.0688 (3)	1.07288 (12)	0.30762 (7)	0.0439 (5)
O4	0.9012 (3)	0.94103 (16)	0.27168 (8)	0.0630 (6)
O5	1.1483 (3)	0.91952 (15)	0.33824 (10)	0.0649 (6)
O6	0.8413 (3)	0.98786 (15)	0.36203 (8)	0.0584 (6)
N1	0.4988 (3)	0.09957 (16)	0.31532 (7)	0.0349 (5)
H1A	0.5772	0.0550	0.3307	0.052*
H1B	0.5491	0.1165	0.2828	0.052*
H1C	0.3759	0.0756	0.3107	0.052*
C1	0.8076 (3)	0.25365 (17)	0.31064 (9)	0.0289 (5)
C2	0.6999 (3)	0.22487 (17)	0.36217 (9)	0.0306 (5)
H2A	0.6911	0.2806	0.3863	0.037*
H2B	0.7778	0.1756	0.3810	0.037*
C3	0.4889 (3)	0.18613 (17)	0.35177 (9)	0.0289 (5)
H3	0.4139	0.2364	0.3321	0.035*
C4	0.3749 (3)	0.16333 (17)	0.40392 (9)	0.0310 (5)
C5	0.3897 (4)	0.0760 (2)	0.43014 (10)	0.0464 (7)
H5	0.4766	0.0289	0.4167	0.056*
C6	0.2752 (5)	0.0576 (2)	0.47670 (12)	0.0565 (8)
H6	0.2840	-0.0023	0.4940	0.068*
C7	0.1499 (4)	0.1267 (2)	0.49722 (11)	0.0537 (8)
H7	0.0739	0.1143	0.5286	0.064*
C8	0.1367 (4)	0.2138 (2)	0.47155 (11)	0.0493 (7)
H8	0.0524	0.2613	0.4857	0.059*
C9	0.2472 (4)	0.2322 (2)	0.42480 (9)	0.0390 (6)
H9	0.2352	0.2916	0.4072	0.047*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0277 (3)	0.0306 (3)	0.0418 (3)	-0.0002 (2)	0.0000 (2)	0.0054 (3)
O1	0.0336 (10)	0.0582 (12)	0.0365 (9)	-0.0188 (8)	0.0033 (8)	-0.0024 (9)
O2	0.0335 (9)	0.0489 (10)	0.0270 (8)	-0.0081 (8)	0.0018 (7)	-0.0040 (7)
O3	0.0422 (10)	0.0332 (10)	0.0564 (11)	-0.0080 (8)	-0.0014 (9)	0.0095 (8)
O4	0.0594 (13)	0.0684 (14)	0.0612 (13)	-0.0217 (11)	-0.0103 (11)	-0.0133 (11)
O5	0.0553 (13)	0.0554 (13)	0.0841 (15)	0.0219 (11)	-0.0044 (12)	0.0233 (11)
O6	0.0517 (12)	0.0644 (13)	0.0591 (12)	0.0037 (10)	0.0210 (10)	0.0119 (11)
N1	0.0307 (11)	0.0439 (12)	0.0300 (10)	-0.0085 (9)	0.0019 (9)	-0.0045 (9)
C1	0.0242 (11)	0.0291 (12)	0.0333 (12)	0.0002 (9)	0.0011 (9)	-0.0020 (10)
C2	0.0296 (12)	0.0359 (13)	0.0262 (11)	-0.0056 (10)	-0.0013 (9)	-0.0024 (10)
C3	0.0253 (11)	0.0355 (13)	0.0259 (11)	0.0009 (10)	0.0013 (9)	-0.0003 (9)
C4	0.0238 (11)	0.0435 (14)	0.0256 (11)	-0.0039 (10)	-0.0005 (9)	0.0008 (10)
C5	0.0485 (16)	0.0501 (16)	0.0406 (14)	0.0037 (13)	0.0071 (13)	0.0065 (13)
C6	0.066 (2)	0.0596 (19)	0.0435 (16)	-0.0121 (16)	0.0047 (15)	0.0180 (14)
C7	0.0425 (16)	0.085 (2)	0.0339 (14)	-0.0167 (16)	0.0088 (12)	0.0037 (15)
C8	0.0388 (15)	0.073 (2)	0.0363 (14)	0.0045 (14)	0.0094 (12)	-0.0057 (14)
C9	0.0323 (12)	0.0520 (16)	0.0326 (12)	0.0016 (11)	0.0003 (11)	0.0012 (12)

Geometric parameters (\AA , $^\circ$)

C11—O5	1.4067 (19)	C3—N1	1.490 (3)
C11—O4	1.421 (2)	C3—C4	1.510 (3)
C11—O6	1.4269 (19)	C3—H3	0.9800
C11—O3	1.4297 (18)	C4—C5	1.369 (3)
O1—H1	0.8200	C4—C9	1.373 (3)
N1—H1A	0.8900	C5—C6	1.388 (4)
N1—H1B	0.8900	C5—H5	0.9300
N1—H1C	0.8900	C6—C7	1.363 (4)
C1—O2	1.205 (3)	C6—H6	0.9300
C1—O1	1.313 (3)	C7—C8	1.359 (4)
C1—C2	1.497 (3)	C7—H7	0.9300
C2—C3	1.524 (3)	C8—C9	1.377 (3)
C2—H2A	0.9700	C8—H8	0.9300
C2—H2B	0.9700	C9—H9	0.9300
O5—C11—O4	110.73 (15)	N1—C3—C2	109.89 (18)
O5—C11—O6	110.28 (13)	C4—C3—C2	113.42 (18)
O4—C11—O6	109.36 (13)	N1—C3—H3	107.5
O5—C11—O3	108.97 (12)	C4—C3—H3	107.5
O4—C11—O3	108.21 (12)	C2—C3—H3	107.5
O6—C11—O3	109.25 (12)	C5—C4—C9	119.0 (2)
C1—O1—H1	109.5	C5—C4—C3	122.6 (2)
C3—N1—H1A	109.5	C9—C4—C3	118.5 (2)
C3—N1—H1B	109.5	C4—C5—C6	120.1 (3)
C3—N1—H1C	109.5	C4—C5—H5	120.0

H1A—N1—H1B	109.5	C6—C5—H5	120.0
H1A—N1—H1C	109.5	C7—C6—C5	120.4 (3)
H1B—N1—H1C	109.5	C7—C6—H6	119.8
O2—C1—O1	123.8 (2)	C5—C6—H6	119.8
O2—C1—C2	124.2 (2)	C8—C7—C6	119.5 (3)
O1—C1—C2	111.91 (19)	C8—C7—H7	120.2
C1—C2—C3	113.35 (18)	C6—C7—H7	120.2
C1—C2—H2A	108.9	C7—C8—C9	120.5 (3)
C3—C2—H2A	108.9	C7—C8—H8	119.7
C1—C2—H2B	108.9	C9—C8—H8	119.7
C3—C2—H2B	108.9	C4—C9—C8	120.5 (3)
H2A—C2—H2B	107.7	C4—C9—H9	119.7
N1—C3—C4	110.68 (19)	C8—C9—H9	119.7

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1B...O2	0.89	2.13	2.773 (3)	128
C9—H9...O5 ⁱ	0.93	2.56	3.409 (3)	152
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C3—H3...O2 ⁱⁱ	0.98	2.58	3.286 (3)	129
N1—H1C...O3 ⁱⁱⁱ	0.89	2.05	2.892 (3)	158
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Fig. 1

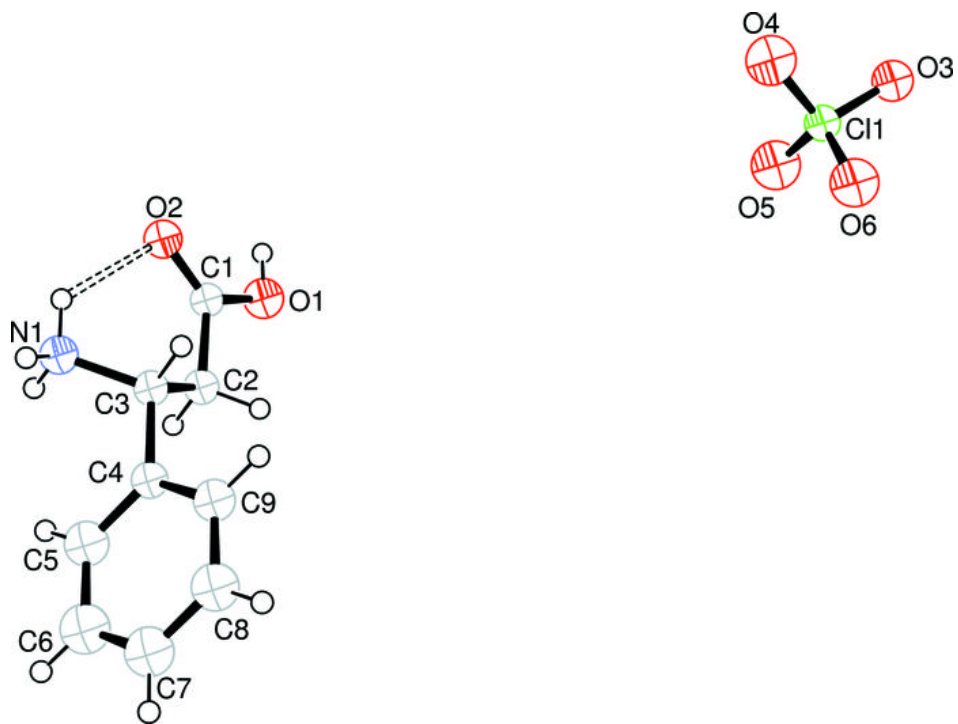


Fig. 2

