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2-Carboxy-1-phenylethanaminium perchlorate

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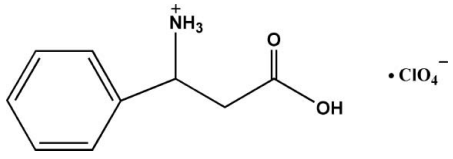
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.053; wR factor = 0.110; data-to-parameter ratio = 16.4.

In the title compound, $C_9H_{12}NO_2^+\cdot ClO_4^-$, an intramolecular N—H···O interaction results in the formation of a six-membered ring having a twisted chair conformation. In the crystal structure, intermolecular O—H···O, N—H···O and C—H···O interactions link the molecules into a network. A weak C—H···π 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

$C_9H_{12}NO_2^+\cdot ClO_4^-$
M_r = 265.65
Orthorhombic, *Pbca*
a = 6.6583 (13) Å
b = 13.826 (3) Å
c = 24.300 (5) Å
V = 2237.0 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 294 K
0.45 × 0.35 × 0.12 mm

Data collection

Rigaku SCXmini diffractometer
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_{int} = 0.058

Refinement

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

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

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

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S1. 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···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···O, N-H···O and C-H···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···*π* interaction (Table 1).

S2. 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.

S3. Refinement

H atoms were positioned geometrically with O-H = 0.82 Å (for OH), N-H = 0.89 Å (for NH_3), 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 NH_3 H and x = 1.2 for all other H atoms.

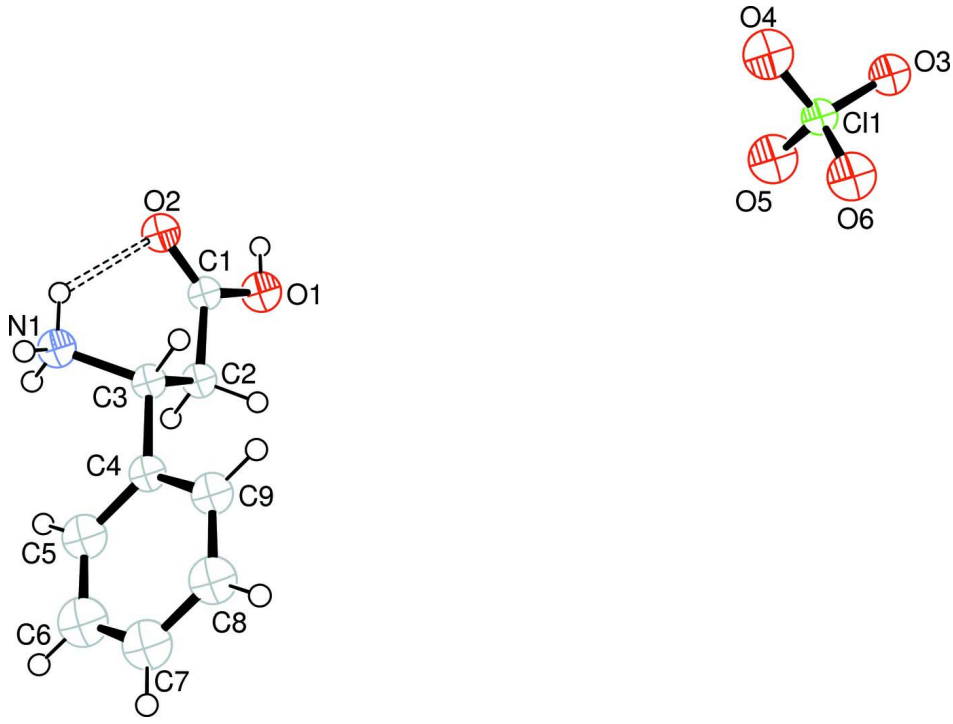

Figure 1

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

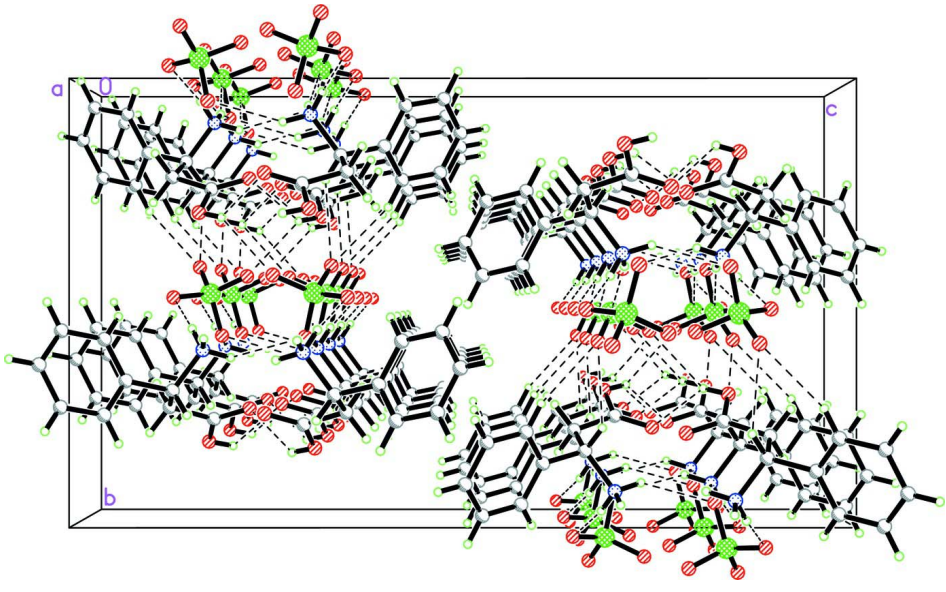

Figure 2

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) Å^3
Z = 8
F(000) = 1104
D_x = 1.578 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1979 reflections
θ = 3.1–27.5°
μ = 0.36 mm^{-1}
T = 294 K
Prism, colorless
0.45 × 0.35 × 0.12 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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_{int} = 0.058
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −8→8
k = −17→17
l = −31→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
0 restraints
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	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*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	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 (Å, º)

Cl1—O5	1.4067 (19)	C3—N1	1.490 (3)
Cl1—O4	1.421 (2)	C3—C4	1.510 (3)
Cl1—O6	1.4269 (19)	C3—H3	0.9800
Cl1—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—Cl1—O4	110.73 (15)	N1—C3—C2	109.89 (18)
O5—Cl1—O6	110.28 (13)	C4—C3—C2	113.42 (18)
O4—Cl1—O6	109.36 (13)	N1—C3—H3	107.5
O5—Cl1—O3	108.97 (12)	C4—C3—H3	107.5
O4—Cl1—O3	108.21 (12)	C2—C3—H3	107.5
O6—Cl1—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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···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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