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(*S*)-2-Ammonio-3-(4-nitrophenyl)-propanoate monohydrate

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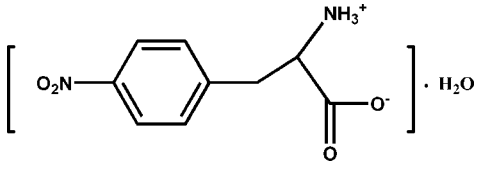
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.114; data-to-parameter ratio = 10.0.

The title compound, $C_9H_{10}N_2O_4\cdot H_2O$, exists as a zwitterion with a deprotonated carboxyl group and a protonated amino group. The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds, building sheets parallel to the (001) plane. The absolute configuration was deduced from the synthetic pathway.

Related literature

For details of α-amino acids as precursors for the synthesis of novel biologically active compounds, see: Lucchese *et al.* (2007); Arki *et al.* (2004); Hauck *et al.* (2006); Azim *et al.* (2006).



Experimental

Crystal data

$C_9H_{10}N_2O_4\cdot H_2O$
M_r = 228.21
Orthorhombic, $P2_12_12_1$
a = 5.3141 (8) Å
b = 6.2823 (7) Å
c = 30.752 (4) Å
V = 1026.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
0.25 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.970, T_{max} = 0.974
10728 measured reflections
1466 independent reflections
1261 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.113
S = 1.09
1466 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O1W	0.89	1.88	2.760 (3)	168
N2—H2A···O1W^{i}	0.89	2.43	3.029 (3)	125
N2—H2A···O4^{ii}	0.89	2.29	2.913 (3)	127
N2—H2C···O3^{iii}	0.89	1.92	2.797 (3)	166
O1W—H1WB···O3^{iv}	0.85	2.23	2.764 (3)	121
O1W—H1WC···O4^{v}	0.85	2.00	2.740 (3)	146

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

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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2340).

References

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supporting information

Acta Cryst. (2008). E**64**, o974 [doi:10.1107/S1600536808011203]

(*S*)-2-Ammonio-3-(4-nitrophenyl)propanoate monohydrate

Wei Dai and Da-Wei Fu

S1. Comment

α-Amino acids are important molecules due to their pharmacological properties. Recently, there has been an increased interest in the enantiomeric preparation of *α*-amino acids as precursors for the synthesis of novel biologically active compounds (Lucchese *et al.*, (2007); Arki *et al.*, (2004); Hauck *et al.*, (2006); Azim *et al.*, (2006)).Here we report the synthesis and crystal structure of the title compound.

The title compound exists as a zwitter ion with a deprotonated carboxyl group and a protonated amino group (Fig. 1). It crystallizes with one water molecule in the asymmetric unit. The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds building sheets parallel to the (0 0 1) plane (Table 1, Figs. 2).

The *S* absolute configuration at C8 is deduced from the synthetic pathway.

S2. Experimental

Under nitrogen protection, 2-amino-3-phenylpropanoic acid (30 mmol), nitric acid (50 mmol) and sulfuric acid (20 mmol) were added in a flask. The mixture was stirred at 110 °C for 3 h. The resulting solution was poured into ice water (100 mL), then filtered and washed with distilled water. The crude product was recrystallized with distilled water to yield colorless block-like crystals, suitable for X-ray analysis.

S3. Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.98 Å (methine), 0.97 Å (methylene), 0.93 Å (aromatic) and N—H = 0.89 Å with $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(N)$. H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O-H= 0.85 (1)Å and H···H= 1.39 (2)Å) with $U_{iso}(H) = 1.5U_{eq}(O)$. In the last stage of refinement they were treated as riding on their parent O atom.

In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined by the X-ray analyses and then the Friedel pairs were merged and any references to the Flack parameter were removed.

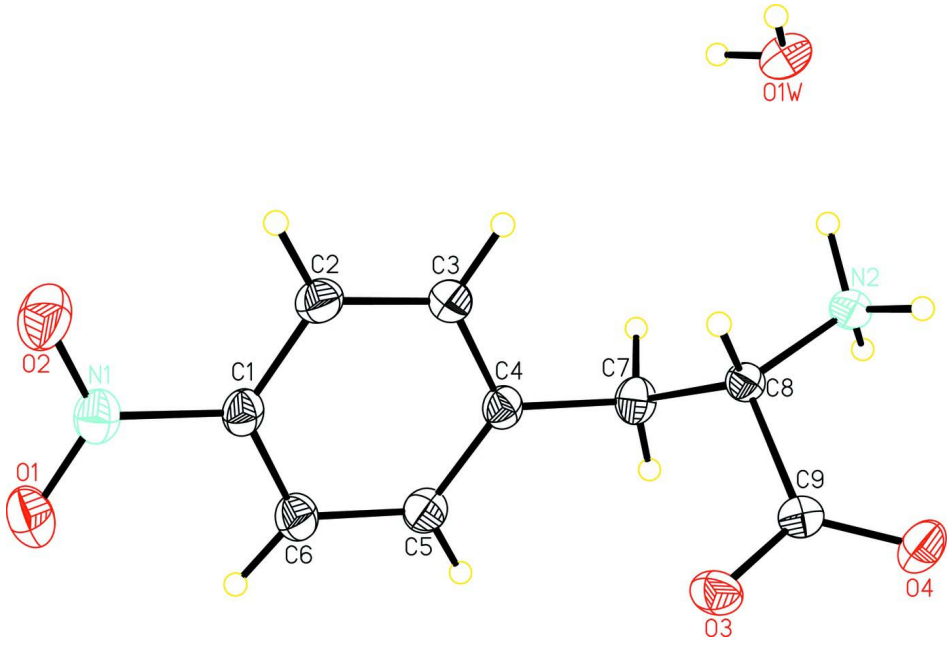

Figure 1

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

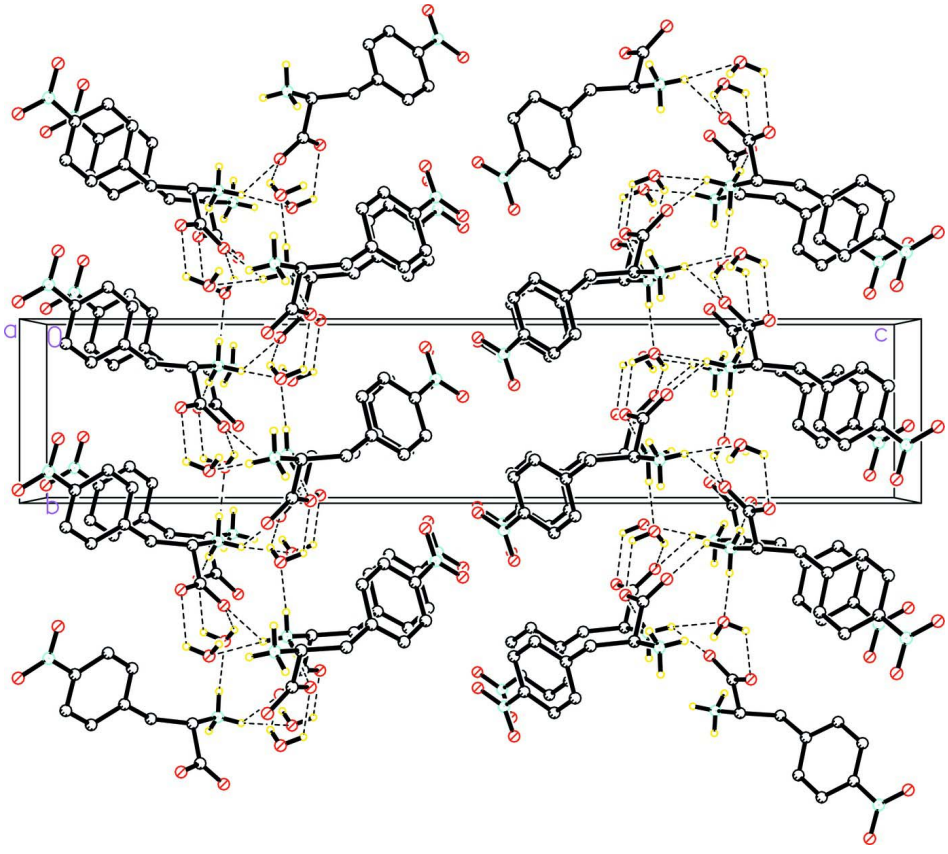

Figure 2

The crystal packing of the title compound viewed along the *a* axis and all hydrogen atoms not involved in hydrogen bonding (dashed lines) were omitted for clarity.

(S)-2-Ammonio-3-(4-nitrophenyl)propanoate monohydrate

Crystal data

$C_9H_{10}N_2O_4·H_2O$
M_r = 228.21
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 5.3141 (8) Å
b = 6.2823 (7) Å
c = 30.752 (4) Å
V = 1026.7 (2) Å^3
Z = 4
$F(000)$ = 480
D_x = 1.476 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1470 reflections
θ = 3.0–27.5°
μ = 0.12 mm^{-1}
T = 293 K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.970, T_{max} = 0.974
10728 measured reflections
1466 independent reflections
1261 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.8°, θ_{min} = 3.3°
h = −6→6
k = −8→8
l = −40→40

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.113$
$S = 1.09$
1466 reflections
146 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
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.0774P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.8183 (3)	0.4808 (3)	0.17549 (6)	0.0391 (4)
N2	0.2254 (4)	0.2416 (3)	0.20604 (6)	0.0346 (5)
H2A	0.2535	0.2749	0.2337	0.052*
H2B	0.1637	0.1101	0.2044	0.052*
H2C	0.1152	0.3328	0.1947	0.052*
O4	0.5036 (4)	0.5887 (3)	0.21776 (6)	0.0452 (5)
O1	1.3141 (4)	−0.0801 (4)	0.00871 (7)	0.0588 (6)
C1	1.0030 (5)	−0.0711 (4)	0.06148 (7)	0.0342 (6)
N1	1.2123 (5)	−0.1761 (4)	0.03883 (7)	0.0410 (5)
C7	0.4109 (5)	0.2328 (4)	0.13231 (8)	0.0369 (6)
H7A	0.2588	0.1496	0.1284	0.044*
H7B	0.3801	0.3735	0.1205	0.044*
C9	0.6059 (5)	0.4594 (4)	0.19280 (7)	0.0301 (5)
C8	0.4651 (4)	0.2532 (4)	0.18137 (7)	0.0283 (5)
H8	0.5704	0.1325	0.1901	0.034*
C3	0.6976 (6)	−0.0771 (4)	0.11762 (8)	0.0391 (6)
H3	0.6180	−0.1481	0.1403	0.047*
C5	0.7376 (5)	0.2308 (4)	0.07239 (8)	0.0376 (6)
H5	0.6850	0.3663	0.0643	0.045*
C4	0.6219 (5)	0.1297 (4)	0.10701 (7)	0.0327 (5)
C2	0.8872 (5)	−0.1774 (4)	0.09522 (8)	0.0405 (6)
H2	0.9366	−0.3146	0.1027	0.049*
C6	0.9322 (5)	0.1322 (4)	0.04943 (8)	0.0397 (6)
H6	1.0122	0.2016	0.0266	0.048*
O2	1.2768 (5)	−0.3521 (4)	0.05090 (7)	0.0589 (6)

O1W	0.0704 (4)	−0.1769 (3)	0.21144 (6)	0.0468 (5)
H1WB	0.0482	−0.2262	0.1860	0.070*
H1WC	0.1824	−0.2497	0.2244	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0300 (9)	0.0402 (10)	0.0470 (10)	−0.0064 (8)	0.0032 (7)	0.0024 (8)
N2	0.0329 (10)	0.0331 (10)	0.0377 (11)	−0.0040 (10)	0.0050 (8)	−0.0008 (9)
O4	0.0382 (10)	0.0407 (10)	0.0568 (11)	−0.0015 (9)	−0.0003 (9)	−0.0179 (9)
O1	0.0581 (13)	0.0682 (14)	0.0501 (12)	0.0028 (12)	0.0185 (10)	−0.0021 (11)
C1	0.0371 (14)	0.0365 (12)	0.0291 (11)	−0.0012 (11)	−0.0015 (10)	−0.0080 (10)
N1	0.0388 (12)	0.0467 (13)	0.0374 (11)	0.0022 (11)	−0.0027 (9)	−0.0075 (10)
C7	0.0366 (13)	0.0428 (13)	0.0311 (12)	0.0021 (12)	−0.0043 (10)	−0.0044 (11)
C9	0.0295 (12)	0.0295 (11)	0.0312 (11)	0.0004 (10)	−0.0057 (9)	0.0031 (10)
C8	0.0274 (11)	0.0284 (11)	0.0293 (11)	−0.0003 (10)	0.0012 (9)	0.0001 (9)
C3	0.0486 (15)	0.0338 (12)	0.0349 (12)	−0.0017 (12)	0.0074 (12)	0.0008 (11)
C5	0.0471 (15)	0.0344 (12)	0.0313 (12)	0.0031 (12)	−0.0031 (11)	0.0021 (10)
C4	0.0348 (13)	0.0351 (12)	0.0283 (11)	−0.0023 (11)	−0.0019 (9)	−0.0050 (10)
C2	0.0537 (16)	0.0315 (12)	0.0364 (12)	0.0009 (12)	0.0017 (12)	0.0013 (11)
C6	0.0473 (15)	0.0415 (14)	0.0304 (11)	0.0002 (12)	0.0038 (12)	0.0001 (11)
O2	0.0594 (14)	0.0560 (12)	0.0612 (13)	0.0206 (12)	0.0009 (12)	−0.0009 (11)
O1W	0.0493 (11)	0.0347 (9)	0.0563 (11)	−0.0032 (9)	−0.0051 (9)	−0.0047 (9)

Geometric parameters (Å, º)

O3—C9	1.255 (3)	C7—H7B	0.9700
N2—C8	1.484 (3)	C9—C8	1.537 (3)
N2—H2A	0.8900	C8—H8	0.9800
N2—H2B	0.8900	C3—C2	1.374 (4)
N2—H2C	0.8900	C3—C4	1.398 (4)
O4—C9	1.243 (3)	C3—H3	0.9300
O1—N1	1.231 (3)	C5—C4	1.384 (3)
C1—C2	1.379 (4)	C5—C6	1.397 (4)
C1—C6	1.382 (4)	C5—H5	0.9300
C1—N1	1.469 (3)	C2—H2	0.9300
N1—O2	1.216 (3)	C6—H6	0.9300
C7—C4	1.511 (3)	O1W—H1WB	0.8502
C7—C8	1.541 (3)	O1W—H1WC	0.8496
C7—H7A	0.9700		
C8—N2—H2A	109.5	N2—C8—C7	109.62 (18)
C8—N2—H2B	109.5	C9—C8—C7	112.6 (2)
H2A—N2—H2B	109.5	N2—C8—H8	108.1
C8—N2—H2C	109.5	C9—C8—H8	108.1
H2A—N2—H2C	109.5	C7—C8—H8	108.1
H2B—N2—H2C	109.5	C2—C3—C4	121.3 (2)
C2—C1—C6	121.9 (2)	C2—C3—H3	119.3

C2—C1—N1	118.5 (2)	C4—C3—H3	119.3
C6—C1—N1	119.6 (2)	C4—C5—C6	120.9 (2)
O2—N1—O1	123.5 (3)	C4—C5—H5	119.5
O2—N1—C1	118.5 (2)	C6—C5—H5	119.5
O1—N1—C1	118.0 (2)	C5—C4—C3	118.6 (2)
C4—C7—C8	113.6 (2)	C5—C4—C7	121.9 (2)
C4—C7—H7A	108.8	C3—C4—C7	119.5 (2)
C8—C7—H7A	108.8	C3—C2—C1	118.8 (2)
C4—C7—H7B	108.8	C3—C2—H2	120.6
C8—C7—H7B	108.8	C1—C2—H2	120.6
H7A—C7—H7B	107.7	C1—C6—C5	118.4 (2)
O4—C9—O3	125.8 (2)	C1—C6—H6	120.8
O4—C9—C8	118.6 (2)	C5—C6—H6	120.8
O3—C9—C8	115.5 (2)	H1WB—O1W—H1WC	109.4
N2—C8—C9	110.03 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O1*W*	0.89	1.88	2.760 (3)	168
N2—H2*A*···O1*W*^{i}	0.89	2.43	3.029 (3)	125
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O1*W*—H1*WC*···O4^{v}	0.85	2.00	2.740 (3)	146

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) *x*−1, *y*, *z*; (iv) *x*−1, *y*−1, *z*; (v) *x*, *y*−1, *z*.