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3-Ammonio-3-(4-pyridyl)propanoate dihydrate

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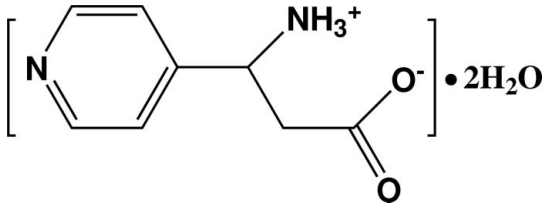
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.099; data-to-parameter ratio = 13.1.

The title compound, $C_8H_{10}N_2O_2\cdot 2H_2O$, exists as a zwitterion with a deprotonated carboxyl group and a protonated amino group. The crystal packing is stabilized by N—H···O, O—H···O and O—H···N hydrogen bonds.

Related literature

For details of 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).



Experimental

Crystal data

$C_8H_{10}N_2O_2\cdot 2H_2O$
M_r = 202.21
Monoclinic, $P2_1/n$
a = 12.6440 (12) Å
b = 5.7698 (5) Å
c = 13.9238 (13) Å
β = 102.601 (2)°
V = 991.32 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 291 (2) K
0.32 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.97, T_{max} = 0.97
5638 measured reflections
1943 independent reflections
1587 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.099
S = 1.09
1943 reflections
148 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.90 (2)	1.81 (2)	2.705 (2)	173.7 (18)
N2—H2B···O1^{ii}	0.90 (2)	1.87 (2)	2.765 (2)	173.5 (18)
N2—H2C···O2^{iii}	0.87 (2)	1.99 (2)	2.8165 (18)	156.7 (18)
O3—H3B···O4	0.87 (2)	1.83 (2)	2.706 (2)	178 (2)
O3—H3A···N1^{iv}	0.86 (2)	2.03 (2)	2.810 (2)	150 (2)
O4—H4A···O1^{v}	0.81 (3)	2.42 (2)	2.8346 (19)	113 (2)
O4—H4B···O2^{vi}	0.88 (2)	2.01 (3)	2.8321 (19)	154 (2)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+2$; (iii) $-x+1, -y, -z+2$; (iv) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (vi) $x, y+1, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2415).

References

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Zeller, E. A., Ramachander, G., Fleisher, G. A., Ishimaru, T. & Zeller, V. (1965). *Biochem. J.* **95**, 262–269.

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supplementary materials

Acta Cryst. (2007). E**63**, o3400 [**doi:10.1107/S1600536807031583**]

3-Ammonio-3-(4-pyridyl)propanoate dihydrate

H. Zhao

Comment

β-Amino acids are important molecules due to their pharmacological properties. Recently, there has been an increased interest in the enantiomeric preparation 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).

The title compound exists as a zwitter ion with a deprotonated carboxyl group and a protonated amino group (Fig. 1). It crystallizes with two water molecules in the asymmetric unit. The crystal packing is stabilized by N—H···O O—H···O O—H···N hydrogen bonds (Figs. 2,3).

Experimental

Under nitrogen protection, isonicotinaldehyde (3.21 g, 30 mmol), malonic acid (5.0 g, 48 mmol) and ammonium acetate (6.0 g, 78 mmol) were added in a flask and refluxed for 10 minutes yielding a white precipitate. After being cooled to room temperature, the solution was filtered and the title compound was obtained as colorless block shaped crystals.

Refinement

All H atoms were located in a difference map. The coordinates of those bonded to N and O were and refined with U_{iso}(H) = 1.2U_{eq}(N, O). Other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and with U_{iso}(H) = 1.2 times U_{eq}(C).

Figures

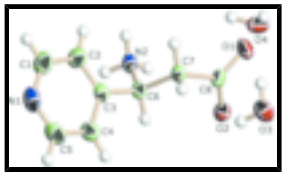

Fig. 1. The molecular structure of the title compound with atom labels and 30% probability displacement ellipsoids for non-H atoms.

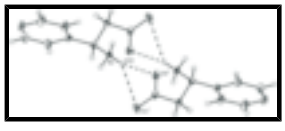

Fig. 2. Dimer formation by hydrogen bonds. Displacement ellipsoids are drawn at the 30% probability level.(i: −*x*, −*y* + 1, −*z* + 1),

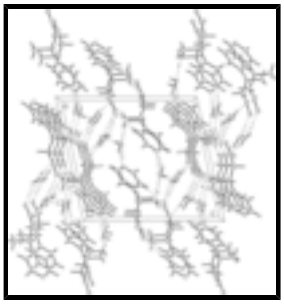

Fig. 3. View of the packing along the *b* axis; hydrogen bonds are shown as dashed lines.

3-Ammonio-3-(4-pyridyl)propanoate dihydrate

Crystal data

$C_8H_{10}N_2O_2·2H_2O$	F_{000} = 432
M_r = 202.21	D_x = 1.355 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3174 reflections
a = 12.6440 (12) Å	θ = 2.6–28.0º
b = 5.7698 (5) Å	μ = 0.11 mm^{-1}
c = 13.9238 (13) Å	T = 291 (2) K
β = 102.601 (2)º	Block, colorless
V = 991.32 (16) Å^3	0.32 × 0.26 × 0.24 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1943 independent reflections
Radiation source: sealed tube	1587 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 291(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −15→15
T_{min} = 0.97, T_{max} = 0.97	k = −7→6
5638 measured reflections	l = −17→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.099	$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.1335P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.09	$(\Delta/\sigma)_{max} < 0.001$
1943 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
148 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.88367 (14)	0.7166 (4)	0.93120 (15)	0.0435 (5)
H1	0.9070	0.8553	0.9629	0.052*
C2	0.79614 (14)	0.6065 (3)	0.95572 (14)	0.0382 (4)
H2	0.7624	0.6698	1.0027	0.046*
C3	0.75982 (11)	0.4019 (3)	0.90947 (11)	0.0273 (3)
C4	0.81478 (13)	0.3175 (3)	0.84032 (13)	0.0368 (4)
H4	0.7931	0.1798	0.8072	0.044*
C5	0.90135 (15)	0.4394 (4)	0.82150 (15)	0.0454 (5)
H5	0.9374	0.3802	0.7754	0.055*
C6	0.66485 (11)	0.2688 (3)	0.93146 (11)	0.0264 (3)
H6	0.6692	0.1091	0.9087	0.032*
C7	0.55681 (11)	0.3694 (3)	0.87822 (12)	0.0263 (3)
H7A	0.5580	0.3924	0.8095	0.032*
H7B	0.5467	0.5195	0.9063	0.032*
C8	0.46128 (11)	0.2123 (3)	0.88535 (11)	0.0265 (3)
N1	0.93620 (11)	0.6381 (3)	0.86598 (12)	0.0430 (4)
N2	0.67000 (11)	0.2622 (3)	1.03866 (10)	0.0286 (3)
H2A	0.7367 (16)	0.213 (3)	1.0692 (14)	0.034*
H2B	0.6601 (15)	0.403 (4)	1.0621 (14)	0.034*
H2C	0.6293 (15)	0.152 (4)	1.0534 (14)	0.034*
O1	0.37631 (9)	0.3022 (2)	0.89856 (10)	0.0425 (3)
O2	0.47508 (9)	−0.0015 (2)	0.87733 (9)	0.0362 (3)
O3	0.37183 (11)	0.3952 (3)	0.61713 (10)	0.0411 (3)
H3B	0.3594 (17)	0.511 (4)	0.6537 (16)	0.049*
H3A	0.435 (2)	0.341 (4)	0.6431 (17)	0.049*
O4	0.32889 (12)	0.7481 (3)	0.73083 (11)	0.0456 (3)
H4A	0.3076 (19)	0.856 (4)	0.6942 (18)	0.055*

H4B	0.3876 (19)	0.788 (4)	0.7747 (18)	0.055*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0413 (9)	0.0422 (11)	0.0490 (11)	−0.0178 (8)	0.0143 (8)	0.0002 (8)
C2	0.0387 (9)	0.0349 (9)	0.0444 (10)	−0.0132 (7)	0.0164 (7)	−0.0035 (8)
C3	0.0209 (7)	0.0302 (8)	0.0310 (8)	−0.0028 (6)	0.0059 (6)	0.0077 (6)
C4	0.0331 (8)	0.0384 (10)	0.0423 (10)	−0.0026 (7)	0.0153 (7)	−0.0007 (7)
C5	0.0390 (9)	0.0551 (13)	0.0504 (11)	−0.0005 (8)	0.0276 (8)	0.0047 (9)
C6	0.0232 (7)	0.0225 (8)	0.0340 (8)	−0.0049 (6)	0.0077 (6)	0.0007 (6)
C7	0.0212 (7)	0.0273 (8)	0.0306 (8)	−0.0052 (6)	0.0057 (5)	0.0003 (6)
C8	0.0243 (7)	0.0334 (9)	0.0217 (7)	−0.0079 (6)	0.0046 (5)	−0.0018 (6)
N1	0.0287 (7)	0.0517 (10)	0.0517 (9)	−0.0107 (6)	0.0158 (6)	0.0120 (8)
N2	0.0220 (6)	0.0300 (7)	0.0344 (7)	−0.0070 (6)	0.0076 (5)	0.0060 (6)
O1	0.0235 (5)	0.0423 (8)	0.0638 (9)	−0.0030 (5)	0.0145 (5)	−0.0052 (6)
O2	0.0302 (6)	0.0303 (7)	0.0502 (7)	−0.0102 (5)	0.0135 (5)	−0.0032 (5)
O3	0.0367 (6)	0.0511 (8)	0.0425 (7)	−0.0025 (6)	0.0237 (5)	0.0089 (6)
O4	0.0482 (8)	0.0426 (8)	0.0459 (8)	0.0093 (6)	0.0102 (6)	−0.0128 (6)

Geometric parameters (Å, °)

C1—N1	1.317 (3)	C6—H6	0.9800
C1—C2	1.382 (2)	C7—C8	1.5304 (19)
C1—H1	0.9300	C7—H7A	0.9700
C2—C3	1.375 (2)	C7—H7B	0.9700
C2—H2	0.9300	C8—O1	1.2424 (19)
C3—C4	1.392 (2)	C8—O2	1.254 (2)
C3—C6	1.5117 (19)	N2—H2A	0.90 (2)
C4—C5	1.374 (2)	N2—H2B	0.90 (2)
C4—H4	0.9300	N2—H2C	0.87 (2)
C5—N1	1.332 (3)	O3—H3B	0.87 (2)
C5—H5	0.9300	O3—H3A	0.86 (2)
C6—N2	1.480 (2)	O4—H4A	0.81 (3)
C6—C7	1.520 (2)	O4—H4B	0.88 (2)
N1—C1—C2	124.33 (19)	C7—C6—H6	108.0
N1—C1—H1	117.8	C6—C7—C8	112.36 (12)
C2—C1—H1	117.8	C6—C7—H7A	109.1
C3—C2—C1	118.86 (17)	C8—C7—H7A	109.1
C3—C2—H2	120.6	C6—C7—H7B	109.1
C1—C2—H2	120.6	C8—C7—H7B	109.1
C2—C3—C4	117.25 (14)	H7A—C7—H7B	107.9
C2—C3—C6	122.69 (14)	O1—C8—O2	124.43 (14)
C4—C3—C6	120.06 (15)	O1—C8—C7	118.86 (15)
C5—C4—C3	119.45 (18)	O2—C8—C7	116.71 (13)
C5—C4—H4	120.3	C1—N1—C5	116.83 (15)
C3—C4—H4	120.3	C6—N2—H2A	108.3 (12)
N1—C5—C4	123.28 (17)	C6—N2—H2B	111.4 (12)

N1—C5—H5	118.4	H2A—N2—H2B	107.6 (17)
C4—C5—H5	118.4	C6—N2—H2C	111.0 (12)
N2—C6—C3	110.80 (12)	H2A—N2—H2C	101.5 (17)
N2—C6—C7	109.79 (12)	H2B—N2—H2C	116.3 (17)
C3—C6—C7	112.11 (12)	H3B—O3—H3A	108 (2)
N2—C6—H6	108.0	H4A—O4—H4B	110 (2)
C3—C6—H6	108.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O3^{i}	0.90 (2)	1.81 (2)	2.705 (2)	173.7 (18)
N2—H2B···O1^{ii}	0.90 (2)	1.87 (2)	2.765 (2)	173.5 (18)
N2—H2C···O2^{iii}	0.87 (2)	1.99 (2)	2.8165 (18)	156.7 (18)
O3—H3B···O4	0.87 (2)	1.83 (2)	2.706 (2)	178 (2)
O3—H3A···N1^{iv}	0.86 (2)	2.03 (2)	2.810 (2)	150 (2)
O4—H4A···O1^{v}	0.81 (3)	2.42 (2)	2.8346 (19)	113 (2)
O4—H4B···O2^{vi}	0.88 (2)	2.01 (3)	2.8321 (19)	154 (2)

Symmetry codes: (i) *x*+1/2, −*y*+1/2, *z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+2; (iii) −*x*+1, −*y*, −*z*+2; (iv) −*x*+3/2, *y*−1/2, −*z*+3/2; (v) −*x*+1/2, *y*+1/2, −*z*+3/2; (vi) *x*, *y*+1, *z*.

Fig. 1

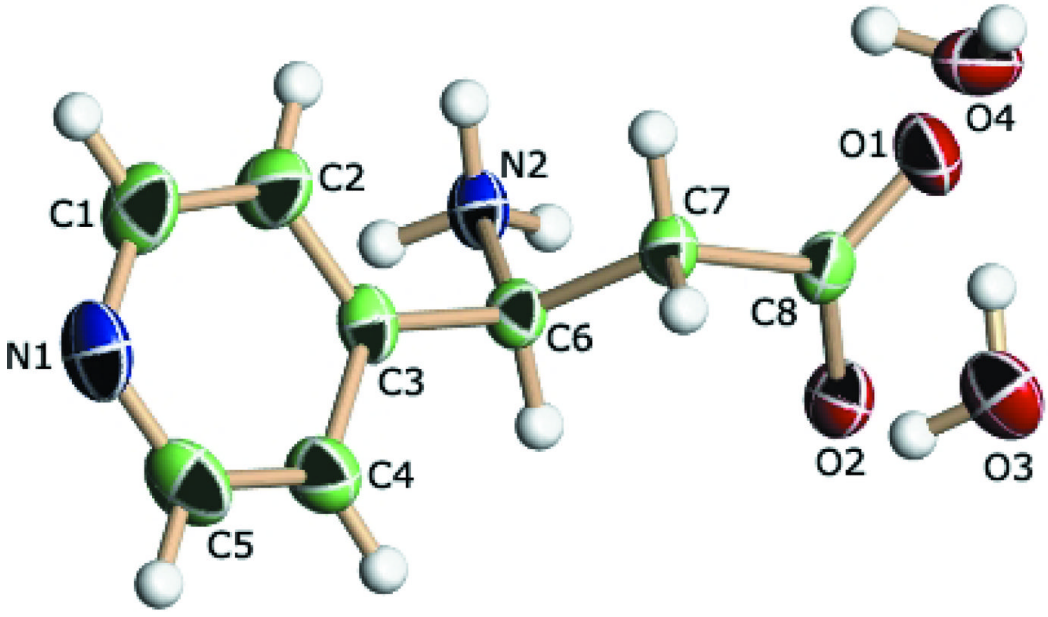

Fig. 2

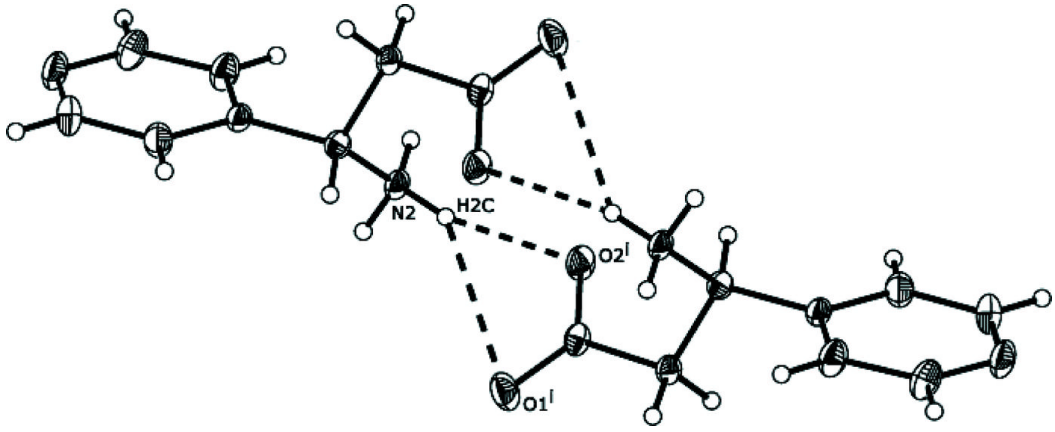

Fig. 3