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Benzimidazolium L-aspartate

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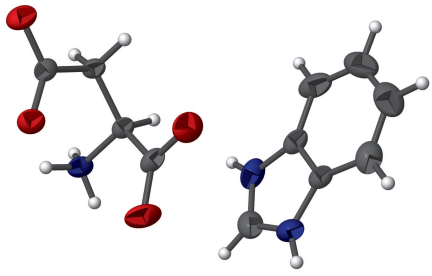
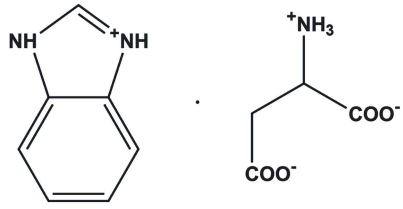
In the cation of the title molecular salt, $C_7H_7N_2^+\cdot C_4H_6NO_4^-$ (systematic name: 1*H*-benzo[*d*]imidazol-3-ium 2-azaniumylsuccinate), the benzimidazole ring system is almost planar (r.m.s. deviation = 0.012 Å). The cation is protonated at the N atom and the L-aspartate zwitterion is deprotonated at both carboxyl groups. In the anion, an N—H···O hydrogen bond and an N—H···O short contact generate *S*(6) graph-set motifs. In the crystal, the anions are linked *via* three N—H···O hydrogen bonds involving the NH_3^+ group, forming layers parallel to the *ab* plane. The benzimidazolium cations are linked to these layers by N—H···O hydrogen bonds. The layers are linked *via* C—H···O hydrogen bonds involving the benzimidazolium cation, forming a three-dimensional structure. There are also C—H···π interactions present involving inversion-related benzimidazolium cations.

3D view



Chemical scheme



Structure description

Benzimidazole derivatives possess antitumour activities (Lukevics *et al.*, 2001; Ignatovich *et al.*, 2010). Herein, we report on the synthesis and the crystal structure of the title molecular salt.

The title compound, Fig. 1, contains a benzimidazole cation, which is protonated at atom N1, and a deprotonated L-aspartate zwitterion. The geometric parameters are comparable with those reported for similar structures (Ennajih *et al.*, 2010; Haque *et al.*, 2012). The benzimidazole ring system is almost planar [maximum deviation = 0.016 (1) Å]. In the anion, N3—H3*A*···O2 and N3—H3*B*···O4 short contacts (Table 1) generate *S*(6) graph-set motifs.

In the crystal, the anions are linked *via* three N—H···O hydrogen bonds involving the NH_3^+ group, forming layers parallel to the *ab* plane (Table 1 and Fig. 2). The benzimidazolium cations are linked to these layers by N—H···O hydrogen bonds (Table 1),

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

*Cg*2 is the centroid of the C2–C7 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O2	0.89	2.38	2.9637 (14)	123
N3—H3*B*···O4	0.89	2.31	2.6544 (15)	103
N1—H1*A*···O1^{i}	0.86	1.77	2.6306 (14)	174
N2—H2···O3^{ii}	0.86	1.78	2.6261 (14)	166
N3—H3*A*···O1^{iii}	0.89	2.15	2.8286 (13)	133
N3—H3*B*···O4^{iv}	0.89	1.97	2.8320 (13)	163
N3—H3*C*···O2^{ii}	0.89	1.89	2.7764 (13)	171
C5—H5···O3^{v}	0.93	2.53	3.424 (2)	163
C3—H3···*Cg*2^{vi}	0.93	2.98	3.6907 (16)	134

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

and the layers are linked *via* C—H···O hydrogen bonds, forming a three-dimensional structure (Table 1 and Fig. 3). There are also C—H···π interactions present involving inversion-related benzimidazolium cations (Table 1).

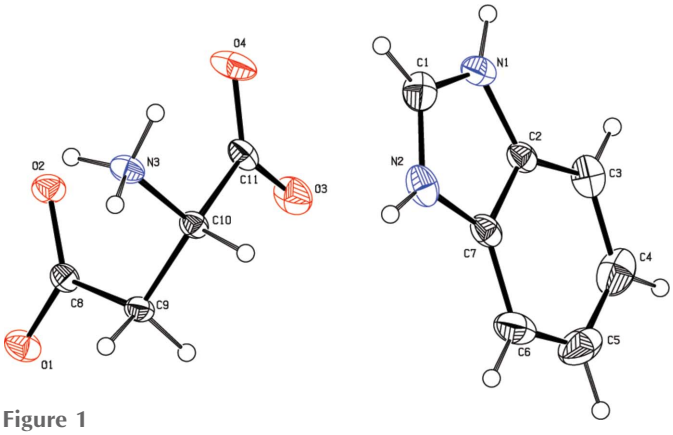


Figure 1
The molecular structure of the title molecular salt, showing the atom labelling and 30% probability displacement ellipsoids.

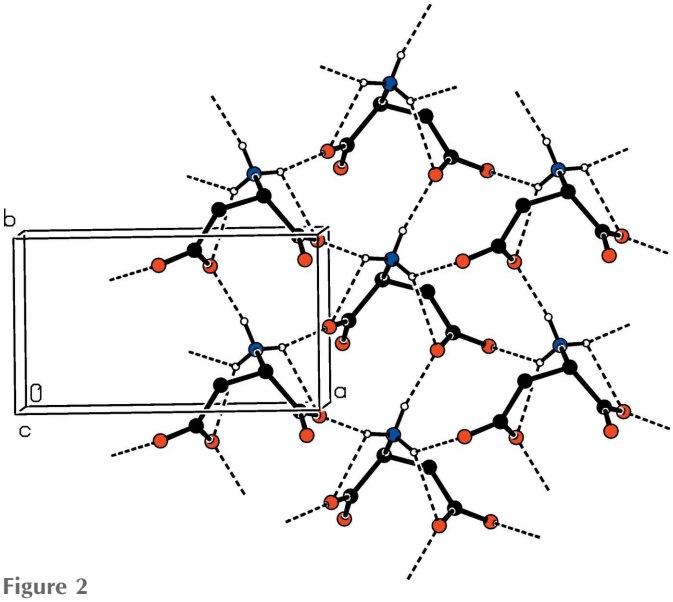


Figure 2
A partial view of the crystal packing of the title compound, viewed along the *c* axis. The N—H···O hydrogen bonds linking the anions are shown as dashed lines (see Table 1), and the cations have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_7N_2^+\cdot C_4H_6NO_4^-$
M_r	251.24
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	295
a, *b*, *c* (Å)	8.9612 (3), 5.0796 (2), 12.5535 (4)
β (°)	102.438 (1)
V (Å^3)	558.02 (3)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.12
Crystal size (mm)	0.26 × 0.24 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.971, 0.977
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15479, 2797, 2659
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.688
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.028, 0.073, 1.04
No. of reflections	2797
No. of parameters	164
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.17

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Synthesis and crystallization

Benzimidazole (3 g) and L-aspartic acid (3.35 g) were dissolved in deionized water in a 1:1 molar ratio and stirred well for about 4 h. The homogeneous solution was filtered and

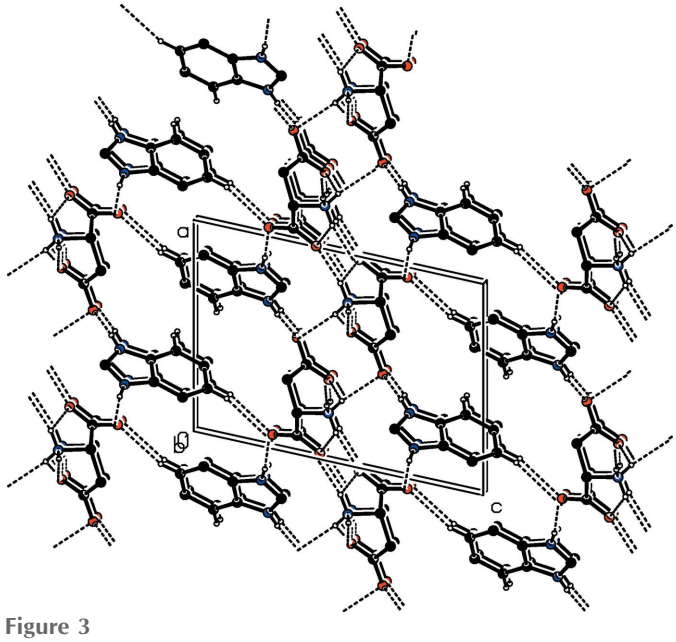


Figure 3
The crystal packing of the title compound, viewed along the *b* axis. The hydrogen bonds are shown as dashed lines (see Table 1), and the majority of the C-bound H atoms have been omitted for clarity.

allowed to evaporate slowly at room temperature. Crystals of the title compound suitable for X-ray diffraction analysis were obtained within a week.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors acknowledge the SAIF, IIT, Madras, for the data collection.

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full crystallographic data

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Benzimidazolium L-aspartate

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1*H*-benzo[*d*]imidazol-3-ium 2-azaniumylsuccinate

Crystal data

$C_7H_7N_2^+·C_4H_6NO_4^-$
M_r = 251.24
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 8.9612 (3) Å
b = 5.0796 (2) Å
c = 12.5535 (4) Å
β = 102.438 (1)°
V = 558.02 (3) Å^3
Z = 2
$F(000)$ = 264
D_x = 1.495 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9965 reflections
θ = 2.3–28.8°
μ = 0.12 mm^{-1}
T = 295 K
Block, colourless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.971, T_{max} = 0.977
15479 measured reflections
2797 independent reflections
2659 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 29.3°, θ_{min} = 2.3°
h = −12→12
k = −6→6
l = −17→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.04
2797 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.0908P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.77000 (15)	0.8587 (3)	0.30109 (11)	0.0413 (3)
H1	0.7795	0.9053	0.3739	0.050*
C2	0.70087 (12)	0.6516 (2)	0.14622 (10)	0.0296 (2)
C3	0.63857 (16)	0.4813 (3)	0.06173 (12)	0.0427 (3)
H3	0.5719	0.3472	0.0710	0.051*
C4	0.6799 (2)	0.5205 (4)	−0.03576 (13)	0.0563 (4)
H4	0.6406	0.4100	−0.0942	0.068*
C5	0.7792 (2)	0.7209 (4)	−0.04961 (13)	0.0594 (5)
H5	0.8049	0.7400	−0.1171	0.071*
C6	0.84112 (17)	0.8932 (3)	0.03336 (14)	0.0504 (4)
H6	0.9071	1.0277	0.0233	0.061*
C7	0.79934 (13)	0.8547 (2)	0.13322 (10)	0.0325 (2)
C8	1.41921 (12)	0.4035 (2)	0.38084 (9)	0.0252 (2)
C9	1.33481 (12)	0.6359 (2)	0.31878 (9)	0.0257 (2)
H9A	1.4023	0.7874	0.3290	0.031*
H9B	1.3109	0.5942	0.2416	0.031*
C10	1.18811 (12)	0.7093 (2)	0.35329 (9)	0.0249 (2)
H10	1.1365	0.8433	0.3022	0.030*
C11	1.07617 (12)	0.4790 (2)	0.35027 (10)	0.0294 (2)
N1	0.68539 (12)	0.6614 (2)	0.25308 (9)	0.0352 (2)
H1A	0.6306	0.5578	0.2831	0.042*
N2	0.83924 (13)	0.9809 (2)	0.23273 (10)	0.0405 (3)
H2	0.8986	1.1151	0.2474	0.049*
N3	1.22318 (11)	0.8297 (2)	0.46337 (8)	0.0293 (2)
H3A	1.2919	0.7315	0.5079	0.044*
H3B	1.1382	0.8390	0.4890	0.044*
H3C	1.2605	0.9909	0.4593	0.044*
O1	1.53603 (9)	0.32202 (19)	0.34933 (7)	0.0364 (2)
O2	1.37046 (9)	0.30887 (17)	0.45807 (7)	0.03213 (18)
O3	1.05177 (11)	0.3459 (2)	0.26439 (8)	0.0457 (2)
O4	1.01562 (12)	0.4487 (2)	0.42891 (9)	0.0497 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0402 (7)	0.0411 (7)	0.0413 (6)	0.0072 (6)	0.0056 (5)	−0.0072 (6)
C2	0.0237 (5)	0.0283 (5)	0.0377 (6)	0.0031 (4)	0.0084 (4)	0.0024 (5)
C3	0.0390 (7)	0.0341 (7)	0.0516 (8)	0.0022 (6)	0.0022 (6)	−0.0049 (6)
C4	0.0658 (11)	0.0570 (10)	0.0425 (8)	0.0206 (9)	0.0038 (7)	−0.0090 (7)
C5	0.0665 (10)	0.0744 (12)	0.0426 (8)	0.0330 (10)	0.0234 (7)	0.0149 (8)

C6	0.0406 (7)	0.0497 (8)	0.0675 (9)	0.0119 (6)	0.0259 (7)	0.0256 (7)
C7	0.0244 (5)	0.0273 (6)	0.0462 (6)	0.0024 (4)	0.0082 (4)	0.0057 (5)
C8	0.0234 (5)	0.0214 (5)	0.0310 (5)	−0.0023 (4)	0.0064 (4)	−0.0010 (4)
C9	0.0241 (5)	0.0260 (5)	0.0290 (5)	−0.0012 (4)	0.0098 (4)	0.0040 (4)
C10	0.0229 (5)	0.0218 (5)	0.0306 (5)	−0.0023 (4)	0.0075 (4)	0.0023 (4)
C11	0.0210 (5)	0.0253 (5)	0.0423 (6)	−0.0026 (4)	0.0078 (4)	0.0013 (5)
N1	0.0318 (5)	0.0357 (5)	0.0413 (5)	0.0011 (4)	0.0150 (4)	0.0029 (5)
N2	0.0315 (5)	0.0291 (5)	0.0572 (7)	−0.0031 (4)	0.0017 (5)	−0.0041 (5)
N3	0.0300 (5)	0.0243 (4)	0.0369 (5)	−0.0049 (4)	0.0142 (4)	−0.0034 (4)
O1	0.0321 (4)	0.0341 (4)	0.0468 (5)	0.0067 (4)	0.0168 (4)	0.0021 (4)
O2	0.0338 (4)	0.0277 (4)	0.0366 (4)	−0.0025 (4)	0.0115 (3)	0.0067 (4)
O3	0.0422 (5)	0.0446 (6)	0.0506 (5)	−0.0180 (5)	0.0103 (4)	−0.0142 (5)
O4	0.0459 (5)	0.0505 (6)	0.0616 (6)	−0.0187 (5)	0.0317 (5)	−0.0050 (5)

Geometric parameters (Å, º)

C1—N2	1.3177 (19)	C8—O1	1.2657 (13)
C1—N1	1.3211 (18)	C8—C9	1.5227 (15)
C1—H1	0.9300	C9—C10	1.5166 (15)
C2—N1	1.3789 (16)	C9—H9A	0.9700
C2—C3	1.3890 (18)	C9—H9B	0.9700
C2—C7	1.3901 (17)	C10—N3	1.4816 (14)
C3—C4	1.368 (2)	C10—C11	1.5365 (15)
C3—H3	0.9300	C10—H10	0.9800
C4—C5	1.387 (3)	C11—O4	1.2342 (15)
C4—H4	0.9300	C11—O3	1.2511 (15)
C5—C6	1.382 (3)	N1—H1A	0.8600
C5—H5	0.9300	N2—H2	0.8600
C6—C7	1.3972 (19)	N3—H3A	0.8900
C6—H6	0.9300	N3—H3B	0.8900
C7—N2	1.3809 (17)	N3—H3C	0.8900
C8—O2	1.2427 (13)		
N2—C1—N1	111.40 (12)	C8—C9—H9A	108.7
N2—C1—H1	124.3	C10—C9—H9B	108.7
N1—C1—H1	124.3	C8—C9—H9B	108.7
N1—C2—C3	131.10 (12)	H9A—C9—H9B	107.6
N1—C2—C7	106.66 (11)	N3—C10—C9	110.19 (9)
C3—C2—C7	122.23 (12)	N3—C10—C11	110.09 (9)
C4—C3—C2	116.82 (15)	C9—C10—C11	114.02 (9)
C4—C3—H3	121.6	N3—C10—H10	107.4
C2—C3—H3	121.6	C9—C10—H10	107.4
C3—C4—C5	121.56 (16)	C11—C10—H10	107.4
C3—C4—H4	119.2	O4—C11—O3	126.64 (11)
C5—C4—H4	119.2	O4—C11—C10	118.17 (11)
C6—C5—C4	122.33 (14)	O3—C11—C10	115.13 (10)
C6—C5—H5	118.8	C1—N1—C2	107.65 (11)
C4—C5—H5	118.8	C1—N1—H1A	126.2

C5—C6—C7	116.47 (15)	C2—N1—H1A	126.2
C5—C6—H6	121.8	C1—N2—C7	107.69 (12)
C7—C6—H6	121.8	C1—N2—H2	126.2
N2—C7—C2	106.60 (11)	C7—N2—H2	126.2
N2—C7—C6	132.80 (13)	C10—N3—H3A	109.5
C2—C7—C6	120.59 (13)	C10—N3—H3B	109.5
O2—C8—O1	124.80 (11)	H3A—N3—H3B	109.5
O2—C8—C9	118.66 (10)	C10—N3—H3C	109.5
O1—C8—C9	116.53 (10)	H3A—N3—H3C	109.5
C10—C9—C8	114.21 (9)	H3B—N3—H3C	109.5
C10—C9—H9A	108.7		

N1—C2—C3—C4	−178.37 (13)	C8—C9—C10—N3	70.83 (12)
C7—C2—C3—C4	0.71 (19)	C8—C9—C10—C11	−53.54 (13)
C2—C3—C4—C5	−0.1 (2)	N3—C10—C11—O4	9.95 (15)
C3—C4—C5—C6	−0.4 (3)	C9—C10—C11—O4	134.37 (12)
C4—C5—C6—C7	0.4 (2)	N3—C10—C11—O3	−172.69 (10)
N1—C2—C7—N2	−0.28 (13)	C9—C10—C11—O3	−48.26 (14)
C3—C2—C7—N2	−179.56 (11)	N2—C1—N1—C2	0.53 (15)
N1—C2—C7—C6	178.49 (11)	C3—C2—N1—C1	179.06 (13)
C3—C2—C7—C6	−0.79 (18)	C7—C2—N1—C1	−0.13 (13)
C5—C6—C7—N2	178.63 (14)	N1—C1—N2—C7	−0.71 (15)
C5—C6—C7—C2	0.24 (19)	C2—C7—N2—C1	0.59 (14)
O2—C8—C9—C10	−5.63 (14)	C6—C7—N2—C1	−177.96 (14)
O1—C8—C9—C10	174.79 (10)		

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C2–C7 ring.

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