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1*H*-Benzo[*d*]imidazol-3-ium (*Z*)-3-carboxyprop-2-enoate

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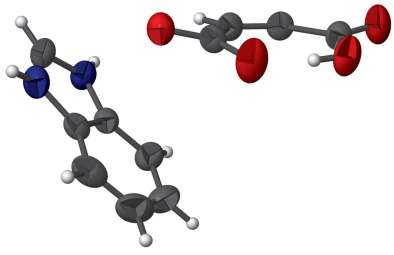
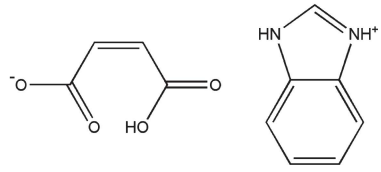
In the anion of the title molecular salt, $C_7H_7N_2^+.C_4H_3O_4^-$, an O—H···O hydrogen bond generates an $S(7)$ graph-set motif while a pair of intermolecular N—H···O and C—H···O hydrogen bonds generate an $R_4^4(10)$ ring-motif. Adjacent anions and cations are further connected through N—H···O hydrogen bonds into infinite chains along [101] and these chains are linked by C—H···O hydrogen bonds, forming a three-dimensional network.

3D view



Chemical scheme



Structure description

Benzimidazole derivatives exhibit various biological effects including antidiabetic (Subudhi *et al.*, 2007) and antimicrobial (El-masry *et al.*, 2000) activity. Herewith we report the synthesis and crystal structure of the title compound whose geometric parameters are comparable to those found in similar structures (Amudha *et al.*, 2015; Krishnamurthy *et al.*, 2015).

The asymmetric unit, Fig. 1, contains a 1*H*-benzo[*d*]imidazol-3-ium cation, protonated at the benzoimidazole N2 atom, and a (*Z*)-3-carboxyprop-2-enoate anion with the OH group of one carboxylic acid deprotonated. The benzoimidazole ring system is almost planar [r.m.s. deviation = 0.016 (2) Å from the best-fit mean plane]. In the anion, an O4—H4*A*···O2 hydrogen bond (Table 1), generates an $S(7)$ graph-set motif, Fig. 2.

In the crystal, a pair of N2—H2*A*···O1 and C7—H7···O1 hydrogen bonds generate an $R_4^4(10)$ ring motif. Atom N2 acts as a bifurcated N—H···(O,O) donor, forming a very long N2—H2*A*···O2 hydrogen bond enclosing an $R_1^2(4)$ ring, Fig. 2 and Table 1. In the crystal, adjacent anions and cations are connected through N—H···O hydrogen bonds (Table 1) into infinite chains along [101]. These chains are further linked by C—H···O hydrogen bonds, forming a three-dimensional network, Fig. 3.

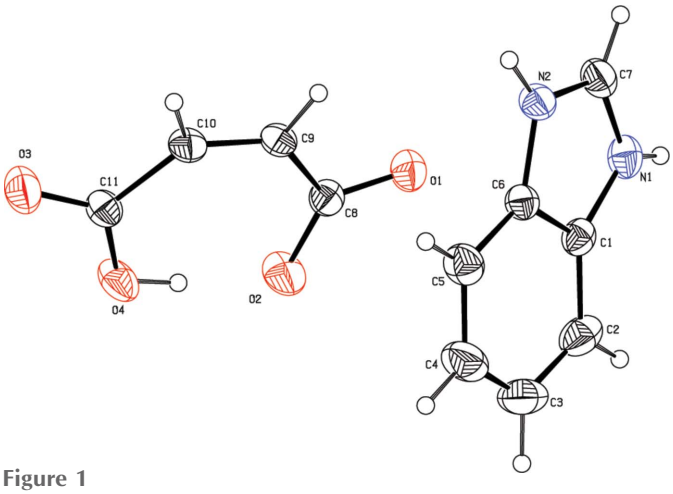


Figure 1
The structure of the title salt, showing the atom labelling and 30% probability displacement ellipsoids.

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O2	0.86 (2)	1.54 (2)	2.395 (2)	173 (2)
N1—H1···O3^{i}	0.87 (1)	1.85 (1)	2.716 (2)	170 (2)
N1—H1···O4^{i}	0.87 (1)	2.59 (2)	3.2215 (19)	130 (2)
N2—H2A···O1^{ii}	0.87 (1)	1.89 (1)	2.761 (2)	173 (2)
N2—H2A···O2^{ii}	0.87 (1)	2.58 (2)	3.2157 (19)	130 (2)
C7—H7···O1^{iii}	0.93	2.29	3.193 (2)	164

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_7N_2^+\cdot C_4H_3O_4^-$
M_r	234.21
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	295
a, b, c (Å)	12.8062 (18), 5.4759 (8), 15.840 (2)
β (°)	92.709 (4)
V (Å^3)	1109.5 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.968, 0.979
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14385, 2814, 1758
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.671
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.118, 1.01
No. of reflections	2814
No. of parameters	165
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.16

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

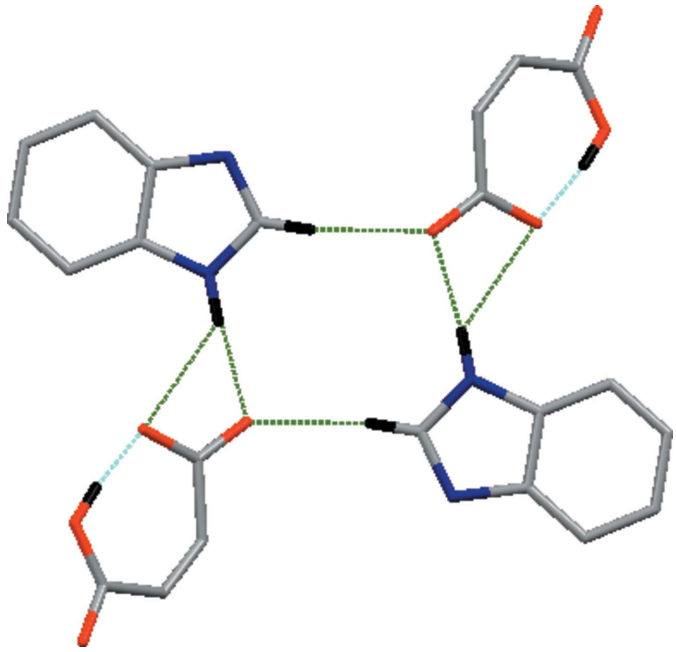


Figure 2
A partial view of the crystal packing of the title compound, showing the ring-set motifs.

Synthesis and crystallization

Benzimidazole (6 g m) and maleic acid (5.88 g m) were taken in an equimolar ratio (1:1) and dissolved in water at room temperature. The resulting solution was stirred well for about six h using a magnetic stirrer and then the solution was filtered and allowed to evaporate at room temperature. Slow evaporation of the solvent yielded crystals suitable for X-ray diffraction analysis over 15 days.

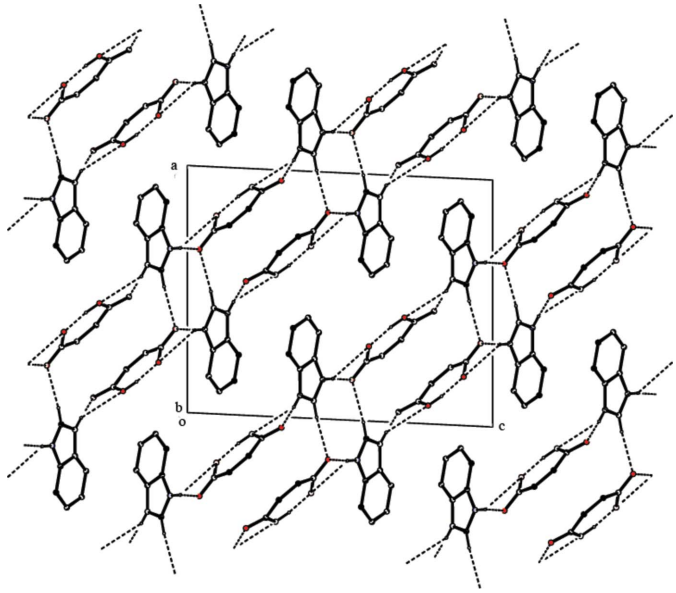


Figure 3
The crystal packing of the title molecular salt viewed along the b axis. The hydrogen bonds are shown as dashed lines (see Table 1). With the exception of H7, C-bound H atoms have been omitted for clarity.

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161025 [https://doi.org/10.1107/S2414314616010257]

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Crystal data

$C_7H_7N_2^+·C_4H_3O_4^-$
M_r = 234.21
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 12.8062 (18) Å
b = 5.4759 (8) Å
c = 15.840 (2) Å
β = 92.709 (4)°
V = 1109.5 (3) Å^3
Z = 4
$F(000)$ = 488
D_x = 1.402 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2814 reflections
θ = 2.6–28.4°
μ = 0.11 mm^{-1}
T = 295 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.968, T_{max} = 0.979
14385 measured reflections
2814 independent reflections
1758 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 28.5°, θ_{min} = 2.6°
h = −17→17
k = −7→7
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.118
S = 1.01
2814 reflections
165 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.3092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 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	0.80286 (14)	0.4571 (3)	0.63117 (10)	0.0408 (4)
C2	0.74325 (18)	0.2787 (4)	0.66778 (12)	0.0577 (5)
H2	0.7736	0.1541	0.7005	0.069*
C3	0.63754 (19)	0.2955 (4)	0.65332 (14)	0.0698 (6)
H3	0.5946	0.1791	0.6767	0.084*
C4	0.59243 (16)	0.4824 (4)	0.60453 (15)	0.0678 (6)
H4	0.5201	0.4862	0.5956	0.081*
C5	0.65108 (14)	0.6604 (4)	0.56929 (12)	0.0539 (5)
H5	0.6205	0.7862	0.5374	0.065*
C6	0.75809 (12)	0.6443 (3)	0.58345 (10)	0.0386 (4)
C7	0.92803 (14)	0.6872 (4)	0.58628 (11)	0.0495 (5)
H7	0.9943	0.7480	0.5768	0.059*
C8	0.75802 (13)	0.2061 (3)	0.41264 (10)	0.0407 (4)
C9	0.73360 (13)	0.4304 (3)	0.36290 (10)	0.0404 (4)
H9	0.7734	0.5666	0.3785	0.048*
C10	0.66372 (13)	0.4662 (3)	0.29959 (11)	0.0418 (4)
H10	0.6611	0.6247	0.2785	0.050*
C11	0.58908 (13)	0.2933 (3)	0.25709 (11)	0.0427 (4)
N1	0.90930 (12)	0.4909 (3)	0.63094 (9)	0.0487 (4)
N2	0.83986 (11)	0.7854 (3)	0.55688 (9)	0.0446 (4)
O1	0.83660 (9)	0.2067 (2)	0.46103 (8)	0.0498 (3)
O2	0.69760 (11)	0.0217 (2)	0.40536 (9)	0.0652 (4)
O3	0.54288 (11)	0.3592 (3)	0.19178 (9)	0.0649 (4)
O4	0.57443 (10)	0.0823 (2)	0.28870 (9)	0.0587 (4)
H1	0.9574 (13)	0.392 (3)	0.6511 (14)	0.081 (7)*
H2A	0.8352 (15)	0.913 (3)	0.5238 (11)	0.063 (6)*
H4A	0.6146 (17)	0.056 (5)	0.3328 (11)	0.094*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0504 (10)	0.0373 (9)	0.0346 (9)	−0.0001 (8)	0.0026 (7)	−0.0051 (7)
C2	0.0855 (15)	0.0431 (11)	0.0456 (10)	−0.0063 (10)	0.0129 (10)	0.0011 (8)
C3	0.0801 (16)	0.0646 (14)	0.0670 (14)	−0.0266 (12)	0.0281 (12)	−0.0085 (11)
C4	0.0453 (11)	0.0846 (17)	0.0745 (15)	−0.0131 (12)	0.0149 (10)	−0.0164 (13)
C5	0.0436 (10)	0.0602 (13)	0.0578 (11)	0.0035 (9)	0.0019 (8)	−0.0036 (10)
C6	0.0414 (9)	0.0356 (9)	0.0388 (9)	−0.0016 (7)	0.0017 (7)	−0.0042 (7)
C7	0.0417 (10)	0.0541 (12)	0.0519 (11)	−0.0044 (8)	−0.0062 (8)	−0.0003 (9)
C8	0.0468 (10)	0.0352 (9)	0.0399 (9)	−0.0012 (8)	0.0016 (7)	−0.0030 (7)
C9	0.0476 (10)	0.0280 (8)	0.0454 (9)	−0.0105 (7)	0.0011 (8)	−0.0021 (7)

C10	0.0491 (10)	0.0271 (8)	0.0491 (10)	−0.0059 (7)	0.0011 (8)	0.0024 (7)
C11	0.0416 (9)	0.0373 (9)	0.0488 (10)	−0.0016 (7)	−0.0016 (8)	−0.0027 (8)
N1	0.0494 (10)	0.0488 (9)	0.0466 (9)	0.0067 (8)	−0.0104 (7)	0.0028 (7)
N2	0.0439 (8)	0.0397 (8)	0.0495 (9)	−0.0034 (7)	−0.0041 (7)	0.0066 (7)
O1	0.0499 (7)	0.0509 (8)	0.0478 (7)	0.0009 (6)	−0.0066 (6)	0.0001 (6)
O2	0.0779 (10)	0.0358 (7)	0.0795 (10)	−0.0180 (7)	−0.0221 (8)	0.0195 (7)
O3	0.0719 (9)	0.0587 (9)	0.0617 (9)	−0.0106 (7)	−0.0233 (7)	0.0066 (7)
O4	0.0590 (9)	0.0393 (7)	0.0759 (10)	−0.0154 (6)	−0.0191 (7)	0.0058 (7)

Geometric parameters (Å, º)

C1—N1	1.376 (2)	C7—H7	0.9300
C1—C6	1.382 (2)	C8—O1	1.2359 (19)
C1—C2	1.384 (3)	C8—O2	1.274 (2)
C2—C3	1.365 (3)	C8—C9	1.485 (2)
C2—H2	0.9300	C9—C10	1.327 (2)
C3—C4	1.392 (3)	C9—H9	0.9300
C3—H3	0.9300	C10—C11	1.484 (2)
C4—C5	1.366 (3)	C10—H10	0.9300
C4—H4	0.9300	C11—O3	1.222 (2)
C5—C6	1.381 (2)	C11—O4	1.277 (2)
C5—H5	0.9300	N1—H1	0.871 (9)
C6—N2	1.383 (2)	N2—H2A	0.873 (9)
C7—N1	1.315 (2)	O4—H4A	0.860 (10)
C7—N2	1.316 (2)		
N1—C1—C6	106.51 (15)	N2—C7—H7	124.8
N1—C1—C2	131.48 (17)	O1—C8—O2	122.11 (16)
C6—C1—C2	122.01 (17)	O1—C8—C9	118.15 (15)
C3—C2—C1	116.38 (19)	O2—C8—C9	119.73 (15)
C3—C2—H2	121.8	C10—C9—C8	129.87 (15)
C1—C2—H2	121.8	C10—C9—H9	115.1
C2—C3—C4	121.7 (2)	C8—C9—H9	115.1
C2—C3—H3	119.2	C9—C10—C11	130.20 (15)
C4—C3—H3	119.2	C9—C10—H10	114.9
C5—C4—C3	122.0 (2)	C11—C10—H10	114.9
C5—C4—H4	119.0	O3—C11—O4	121.68 (16)
C3—C4—H4	119.0	O3—C11—C10	118.12 (16)
C4—C5—C6	116.65 (19)	O4—C11—C10	120.19 (15)
C4—C5—H5	121.7	C7—N1—C1	108.51 (14)
C6—C5—H5	121.7	C7—N1—H1	124.3 (15)
C5—C6—C1	121.27 (17)	C1—N1—H1	126.8 (15)
C5—C6—N2	132.43 (17)	C7—N2—C6	108.26 (15)
C1—C6—N2	106.29 (14)	C7—N2—H2A	124.9 (13)
N1—C7—N2	110.43 (16)	C6—N2—H2A	126.7 (13)
N1—C7—H7	124.8	C11—O4—H4A	112.0 (17)
N1—C1—C2—C3	−178.05 (18)	O1—C8—C9—C10	169.98 (18)

C6—C1—C2—C3	0.8 (3)	O2—C8—C9—C10	−11.0 (3)
C1—C2—C3—C4	−0.1 (3)	C8—C9—C10—C11	−1.3 (3)
C2—C3—C4—C5	−0.7 (3)	C9—C10—C11—O3	−169.26 (18)
C3—C4—C5—C6	0.9 (3)	C9—C10—C11—O4	10.8 (3)
C4—C5—C6—C1	−0.2 (3)	N2—C7—N1—C1	0.0 (2)
C4—C5—C6—N2	178.47 (18)	C6—C1—N1—C7	0.34 (19)
N1—C1—C6—C5	178.42 (16)	C2—C1—N1—C7	179.36 (18)
C2—C1—C6—C5	−0.7 (3)	N1—C7—N2—C6	−0.3 (2)
N1—C1—C6—N2	−0.53 (18)	C5—C6—N2—C7	−178.24 (19)
C2—C1—C6—N2	−179.66 (16)	C1—C6—N2—C7	0.54 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*A*···O2	0.86 (2)	1.54 (2)	2.395 (2)	173 (2)
N1—H1···O3^{i}	0.87 (1)	1.85 (1)	2.716 (2)	170 (2)
N1—H1···O4^{i}	0.87 (1)	2.59 (2)	3.2215 (19)	130 (2)
N2—H2*A*···O1^{ii}	0.87 (1)	1.89 (1)	2.761 (2)	173 (2)
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