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1*H*-Benzimidazolium Hydrogen L-Tartrate Dihydrate

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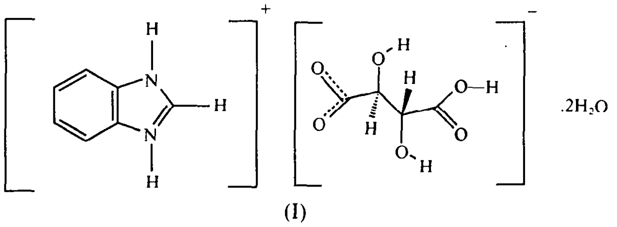
Abstract

The structure of the title compound, $C_7H_7N_2^+.C_4H_5O_6^-.2H_2O$, is dominated by infinite chains of hydrogen L-tartrate anions linked in a head-to-tail manner by a short hydrogen bond, O···O = 2.576 (4) Å. The cation and the water molecules provide several crosslinks between adjacent anionic chains, creating a complex three-dimensional hydrogen-bond network.

Comment

The structures and properties of salts of tartaric acid have captivated scientists from a remarkably diverse range of disciplines, including solid-state physics (Fousek, 1991), wine-making technology (Clark, Fuglesang & Gump, 1988) and chiral synthesis (Marshall & Luke, 1991). This family of compounds is also beginning to attract significant attention because of its ability to exhibit nonlinear optical effects (Aakeröy, Hitchcock & Seddon, 1992; Zyss, Pecaut, Levy & Masse, 1993). Furthermore, the possibilities of using infinite sheets of hydrogen tartrate anions as building blocks ('scaffolding') for crystal engineering have also been explored recently (Aakeröy & Hitchcock, 1993).

We are currently conducting an extensive structural investigation of hydrogen tartrate salts in order to determine the role played by the cation in inducing, or discriminating between, different anionic aggregate types. In addition, the physico-chemical characteristics of the cations within hydrated *versus* anhydrous hydrogen tartrate salts are being evaluated with a view to predicting and controlling the structural features of novel, unknown crystalline materials (Aakeröy, Rzepa & Seddon, 1994). The synthesis and crystallographic study of the title compound (I) forms part of this investigation. The title compound was prepared by mixing equimolar ethanolic solutions of 1*H*-benzimidazole and L-tartaric acid. After evaporation of the solvent, the product was collected by filtration and recrystallized from water.



(I)

The cation and anion do not display any unusual structural features (Fig. 1). The cation exhibits the expected planar geometry and the C—C—C—C torsion angle of the anion is close to 180°.

The anions are arranged in infinite chains, parallel to **b**, *via* a short O—H···O hydrogen bond, with an O—H···O angle of 160 (3)°, which links adjacent anions in a head-to-tail fashion. However, there are no direct hydrogen bonds between adjacent chains, which prevents the formation of the commonly

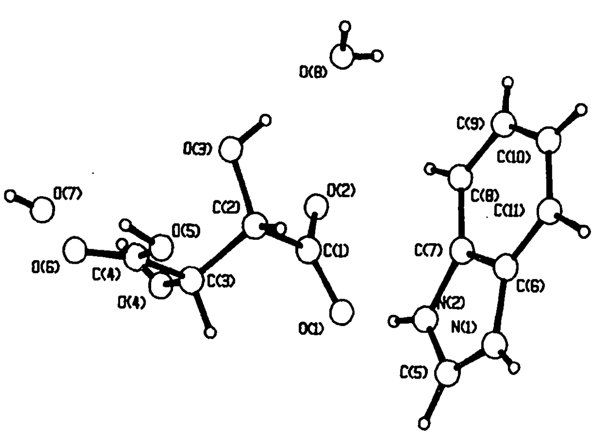


Fig. 1. View of the title compound showing the atom-numbering scheme.

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occurring two-dimensional hydrogen tartrate layer (Aakeröy & Hitchcock, 1993). The cation provides the expected crosslink between adjacent chains *via* two hydrogen bonds, N(1)···O(2)($-1+x$, y, z) 2.749 (4) and N(2)···O(1) 2.805 (4) Å (Fig. 2). A very similar cation–anion interaction, where the cation again acts as a crosslink between adjacent anionic aggregates, has also been observed in 1*H*-imidazolium hydrogen L-tartrate (Aakeröy & Hitchcock, 1993). The two water molecules provide an additional six hydrogen bonds thereby generating a complex three-dimensional hydrogen-bonded network throughout the structure.

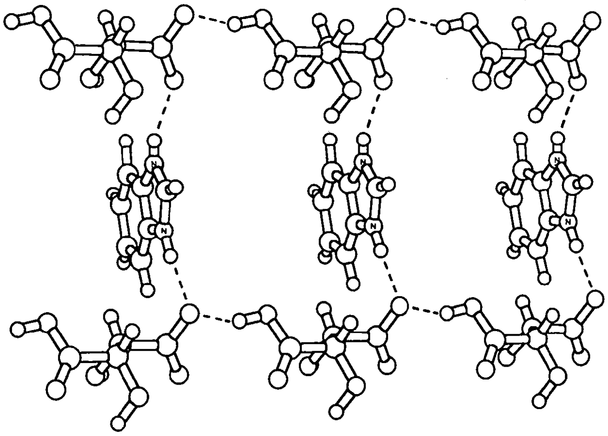

Fig. 2. A view (down the *c* axis) of the cationic crosslinking of adjacent anionic chains (chains parallel to **b**). Hydrogen bonds are represented by dashed lines. Water molecules removed for clarity.

Experimental

Crystal data

$C_7H_7N_2^+.C_4H_5O_6^-$.-
2H₂O
$M_r = 304.3$
Monoclinic
$P2_1$
$a = 9.218$ (1) Å
$b = 7.279$ (2) Å
$c = 10.957$ (3) Å
$\beta = 110.43$ (2)°
$V = 689.0$ (3) Å^3
$Z = 2$

$D_x = 1.47$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 7$–$10°$
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Tablet
0.20 × 0.15 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ-2θ scans
Absorption correction: none
1313 measured reflections
1313 independent reflections
1113 observed reflections [$I > 2\sigma(I)$]

$\theta_{max} = 25°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 8$
$l = -13 \rightarrow 13$
2 standard reflections
frequency: 60 min
intensity variation: 3%

Refinement

Refinement on F
$R = 0.030$
$wR = 0.039$
$S = 1.6$
1113 reflections
237 parameters
Only coordinates of H atoms refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.3$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O(1)	0.4660 (2)	0.6457	0.3261 (1)	0.032 (1)
O(2)	0.6324 (2)	0.6465 (3)	0.2198 (2)	0.037 (1)
O(3)	0.6102 (2)	0.2814 (3)	0.1809 (2)	0.041 (1)
O(4)	0.7750 (2)	0.3445 (3)	0.4593 (2)	0.044 (1)
O(5)	0.4848 (2)	−0.0016 (3)	0.3441 (2)	0.034 (1)
O(6)	0.7378 (2)	−0.0164 (3)	0.4523 (3)	0.063 (1)
O(7)	1.0591 (2)	0.1863 (3)	0.4776 (2)	0.050 (1)
O(8)	0.4816 (2)	0.4335 (3)	−0.0553 (2)	0.049 (1)
N(1)	−0.1029 (2)	0.5741 (4)	0.1669 (2)	0.041 (1)
N(2)	0.1455 (2)	0.5873 (4)	0.2203 (2)	0.040 (1)
C(1)	0.5486 (2)	0.5663 (4)	0.2712 (2)	0.025 (1)
C(2)	0.5410 (2)	0.3568 (4)	0.2665 (2)	0.028 (1)
C(3)	0.6228 (3)	0.2791 (4)	0.4023 (2)	0.030 (1)
C(4)	0.6208 (2)	0.0692 (4)	0.4016 (2)	0.030 (1)
C(5)	0.0281 (3)	0.5822 (5)	0.2641 (2)	0.042 (1)
C(6)	−0.0743 (3)	0.5752 (5)	0.0506 (2)	0.035 (1)
C(7)	0.0854 (3)	0.5843 (4)	0.0853 (2)	0.034 (1)
C(8)	0.1562 (3)	0.5901 (6)	−0.0081 (2)	0.048 (1)
C(9)	0.0610 (3)	0.5851 (6)	−0.1356 (2)	0.053 (1)
C(10)	−0.1007 (3)	0.5764 (6)	−0.1709 (2)	0.051 (1)
C(11)	−0.1705 (3)	0.5688 (5)	−0.0789 (2)	0.046 (1)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(1)	1.263 (3)	C(8)—C(9)	1.366 (3)
O(3)—C(2)	1.416 (3)	C(9)—C(10)	1.405 (4)
O(4)—C(3)	1.405 (3)	C(10)—C(11)	1.374 (5)
O(5)—C(4)	1.297 (3)	O(2)—C(1)	1.249 (3)
O(6)—C(4)	1.201 (3)	N(1)—C(5)	1.303 (3)
N(1)—C(6)	1.388 (4)	N(2)—C(7)	1.388 (3)
N(2)—C(5)	1.327 (4)	C(1)—C(2)	1.526 (4)
C(2)—C(3)	1.523 (3)	C(6)—C(7)	1.388 (3)
C(3)—C(4)	1.528 (4)	C(7)—C(8)	1.393 (4)
C(6)—C(11)	1.387 (3)		
C(5)—N(2)—C(7)	108.2 (2)	O(2)—C(1)—C(2)	118.7 (2)
O(1)—C(1)—C(2)	116.4 (2)	O(3)—C(2)—C(3)	108.6 (2)
O(3)—C(2)—C(1)	112.6 (2)	C(1)—C(2)—C(3)	109.7 (2)
O(4)—C(3)—C(2)	112.7 (2)	O(4)—C(3)—C(4)	110.5 (2)
O(5)—C(4)—O(6)	125.4 (3)	C(2)—C(3)—C(4)	111.4 (2)
O(6)—C(4)—C(3)	120.6 (2)	O(5)—C(4)—C(3)	114.0 (2)
N(1)—C(6)—C(7)	105.8 (2)	N(1)—C(5)—N(2)	110.2 (2)
C(7)—C(6)—C(11)	121.4 (3)	N(1)—C(6)—C(11)	132.9 (2)
N(2)—C(7)—C(8)	131.9 (2)	N(2)—C(7)—C(6)	106.4 (2)
C(7)—C(8)—C(9)	116.8 (2)	C(6)—C(7)—C(8)	121.6 (2)
C(9)—C(10)—C(11)	121.6 (2)	C(8)—C(9)—C(10)	121.6 (3)
C(5)—N(1)—C(6)	109.3 (2)	C(6)—C(11)—C(10)	117.0 (2)
O(1)—C(1)—O(2)	124.9 (2)		

Table 3. *Hydrogen-bonding geometry* (Å)

D—H···A	D···A	D—H···A	D···A
N(1)—H···O(2^{i})	2.749 (4)	O(8)—H···O(2^{iii})	2.719 (4)
N(2)—H···O(1)	2.805 (4)	O(8)—H···O(3^{iv})	2.865 (4)

O(3)—H···O(8)	2.681 (4)	O(7)—H···O(4^{v})	2.876 (4)
O(4)—H···O(7)	2.803 (4)	O(7)—H···O(6^{vi})	2.788 (4)
O(5)—H···O(1^{ii})	2.576 (4)		

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

Calculations were performed using *SHELXS*86 (Sheldrick, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71713 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1071]

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1-Methyl-5-nitro-2-phenylimidazole and 2-(*p*-Aminophenyl)-1-methyl-5-nitroimidazole

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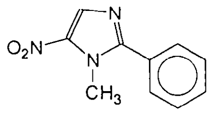
Abstract

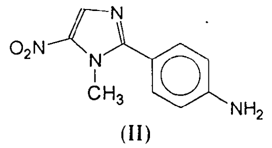
1-Methyl-5-nitro-2-phenylimidazole (I), $C_{10}H_9N_3O_2$, and 2-(*p*-aminophenyl)-1-methyl-5-nitroimidazole (II), $C_{10}H_{10}N_4O_2$, show similar bond distances and angles. Some differences are observed in the bond distances and angles in the phenyl rings due to the presence of the amino substituent and hydrogen bonds in (II).

Comment

Perspective views showing the atomic numbering scheme for (I) and (II) are given in Figs. 1 and 2. In both structures the imidazole and phenyl rings are planar within experimental error. The dihedral angles between the phenyl and imidazole rings in (I) and (II) are 138.58 (6) and 143.1 (1)°, respectively. The angle between the nitro group and the imidazole plane is 8.1 (2)° in (I) and 6.4 (3)° in (II).



(I)



(II)

The amino and nitro groups in (II) are involved in hydrogen bonding [N24—H241···O502^{i} 2.11 (5) Å, 151 (3)°; N24—H242···O501^{ii} 2.33 (4) Å, 167 (4)°; symmetry codes: (i) $x-1, y, z$; (ii) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$]. Each molecule of (II) is surrounded by four other molecules. This results in folded layers parallel to (010).

The bond lengths and bond angles in both compounds are similar, except for those in the phenyl rings. A comparison of the angles in the phenyl rings shows the influence of the amino substituent in (II). The differences found are

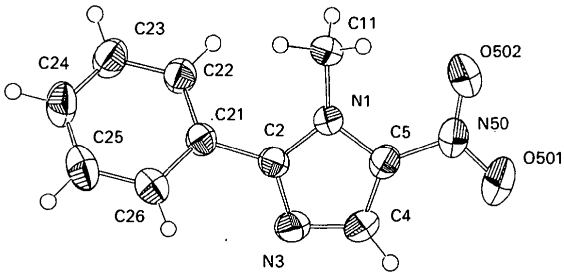


Fig. 1. A perspective view of molecule (I) with atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.

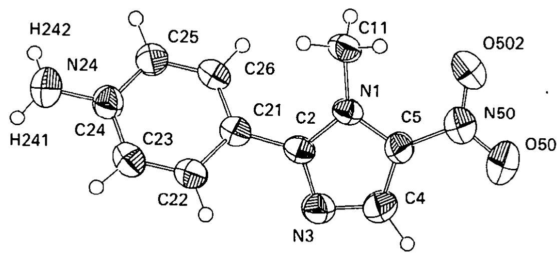


Fig. 2. A perspective view of molecule (II) with atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.