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2-Dihydroxymethyl-3*H*-benzoimidazol-1-ium chloride monohydrate

Alexander J. Blake,* Stuart J. Hill and Peter Hubberstey

School of Chemistry, University of Nottingham, University Park, Nottingham NG7 2RD, England

Correspondence e-mail: a.j.blake@nottingham.ac.uk

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.100
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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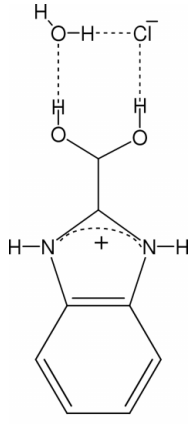
The title compound, $C_8H_9N_2O_2^+\cdot Cl^-\cdot H_2O$, comprises $C_8H_9N_2O_2^+$ cations, chloride anions and lattice water molecules. Individual components are linked into an $R_3^2(8)$ hydrogen-bonded ring to form the crystallographic asymmetric unit. The asymmetric units are linked by hydrogen-bonding contacts to form an extended three-dimensional structure.

Comment

The crystallographic asymmetric unit of 2-dihydroxymethyl-3*H*-benzoimidazol-1-ium chloride monohydrate [1*H*-benzimidazole-2-methanediol monohydrochloride monohydrate], (I), contains a single 2-dihydroxymethyl-3*H*-benzoimidazol-1-ium cation, one chloride anion and one water molecule linked by one O—H···O and two O—H···Cl hydrogen bonds to form an $R_3^2(8)$ ring (Fig. 1). The short interatomic distances and near-linear interatomic angles of these hydrogen bonds (O1—H11···Cl1, O211—H211···Cl1 and O212—H212···O1; see Table 1) are indicative of their significance.



(I)

The long-range structure can be considered in a number of different ways. Our preferred description is that of mutually perpendicular asymmetric units linked by O—H···Cl (O1—H12···Cl1^{i}; see Table 1) and N—H···O (N3—H3···O212^{iii}; see Table 1) contacts in the form of an $R_5^4(18)$ motif (Fig. 2) to generate a crosslinked double-stranded ribbon (Fig. 3) which propagates in the *b* direction. The ribbons are linked by pairs of centrosymmetrically related N—H···Cl contacts (N1—H1···Cl1^{ii}; see Table 1) in the form of an $R_4^2(14)$ motif (Fig. 4) to give a two-dimensional sheet structure parallel to the (010) plane. The sheets, the thickness of which is equivalent to the length of the *a* axis, interdigitate with a limited π–π interaction between crystallographically centrosymmetrically related

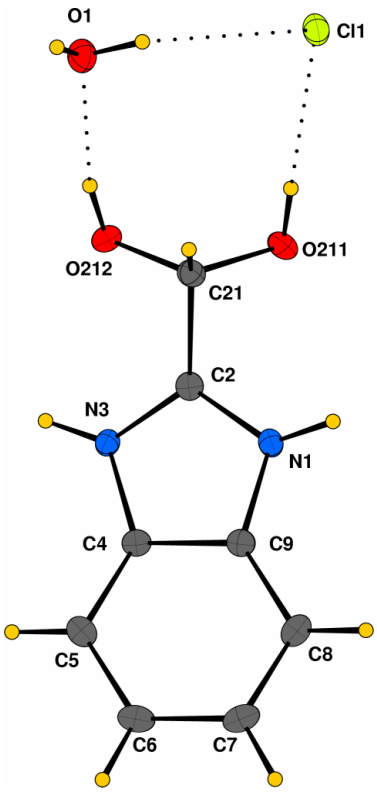


Figure 1
The asymmetric unit of the title compound showing the atom-numbering scheme and hydrogen-bonding contacts. Displacement ellipsoids are drawn at the 50% probability level.

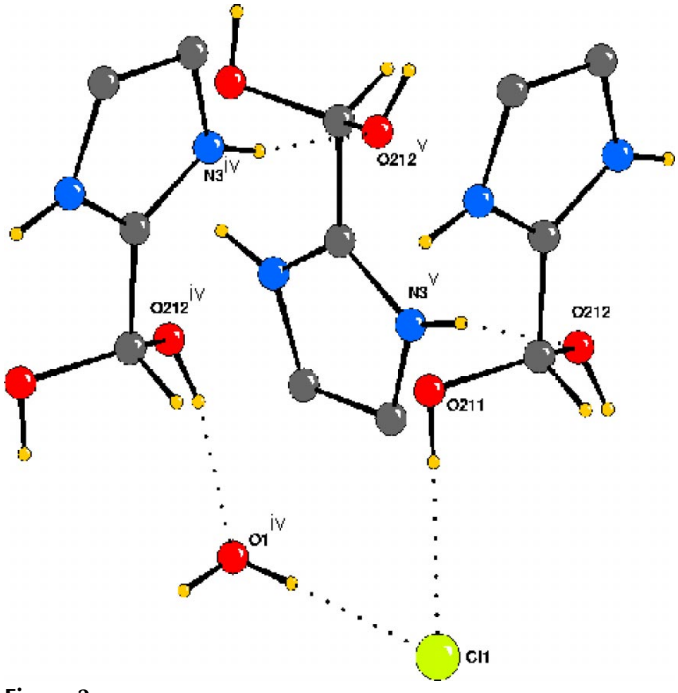


Figure 2
The $R_5^4(18)$ motif within which mutually perpendicular asymmetric units are hydrogen-bonded to form a crosslinked double-stranded ribbon. Atoms are coloured as follows: C black, N blue, O red, Cl green, H small yellow circles. Benzene rings have been omitted for clarity. [Symmetry codes: (iv) x, $1+y$, z; (v) $1-x$, $1/2+y$, $3/2-z$.]

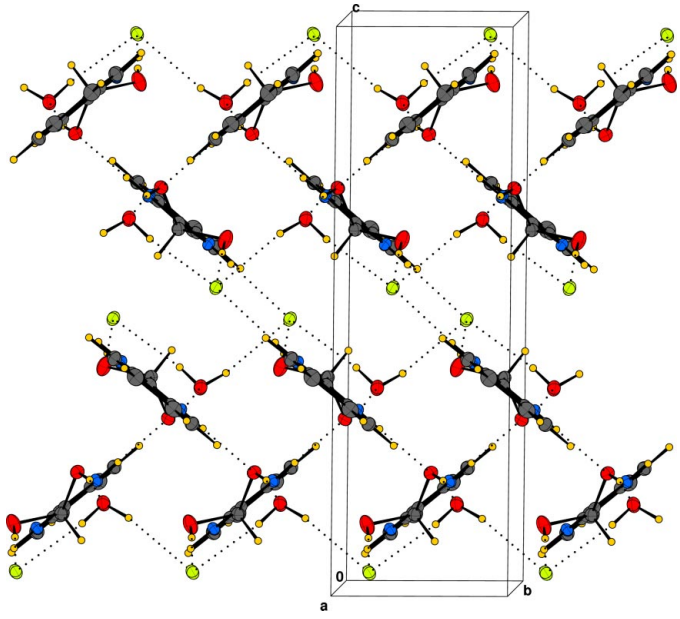


Figure 3
A projection of the structure approximately on to the (100) plane showing the combination of crosslinked double-stranded ribbons to form two-dimensional sheets. Atoms are coloured as in Fig. 2.

aromatic units (Fig. 5). The interplanar separation [3.642 (3) Å], C7···C8($2-x$, $1-y$, $2-z$) distance [3.779 (4) Å] and H7···C8 distance (3.647 Å) are indicative of the weakness of this π–π interaction.

Experimental

The title compound was obtained as a by-product of the reaction of 1,2-phenylenediamine (2.44 g, 22.5 mmol) with iminodiacetic acid (1.50 g, 11.2 mmol) in aqueous hydrochloric acid (6 *M*, 40 ml). This reaction is a variant of the method of Casella *et al.* (1996) for the preparation of bis[1-methylbenzimidazol-2-yl)methyl]amine hydrochloride. Crystals suitable for diffraction studies were grown from acetonitrile solution.

Crystal data

$C_8H_9N_2O_2^+\cdot Cl^-\cdot H_2O$
M_r = 218.64
Monoclinic, $P2_1/c$
a = 12.513 (4) Å
b = 4.977 (3) Å
c = 16.388 (9) Å
β = 103.38 (6)°
V = 992.9 (9) Å^3
Z = 4
D_x = 1.463 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 reflections
θ = 13.3–17.4°
μ = 0.37 mm^{-1}
T = 150 (2) K
Tablet, colourless
0.48 × 0.29 × 0.19 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
ω/θ scans
Absorption correction: none
1724 measured reflections
1724 independent reflections
1470 reflections with $I > 2\sigma(I)$
θ_{max} = 25.0°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.100$
$S = 1.21$
1724 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.848P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···Cl1	0.83 (2)	2.32 (2)	3.132 (2)	169 (3)
O211—H211···Cl1	0.82 (2)	2.25 (2)	3.069 (2)	178 (3)
O212—H212···O1	0.82 (2)	1.86 (2)	2.664 (3)	167 (3)
O1—H12···Cl1^{i}	0.83 (2)	2.36 (2)	3.173 (2)	168 (3)
N1—H1···Cl1^{ii}	0.88 (2)	2.27 (2)	3.129 (2)	165 (2)
N3—H3···O212^{iii}	0.88 (2)	1.95 (2)	2.819 (3)	170 (2)

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

H atoms were found from ΔF syntheses (O and N) or placed geometrically (C); thereafter they were refined respectively with distance restraints (N—H = 0.88, O—H = 0.82 and water H···H = 1.33 Å) and using a riding model (C—H = 0.93 and 0.98 Å for aromatic and methine H, respectively).

Data collection: *STADI*4 (Stoe & Cie, 1995); cell refinement: *Stadi*4; data reduction: *X-RED* (Stoe & Cie, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2001).

We thank EPSRC for the award of a diffractometer and for a maintenance grant to SJH.

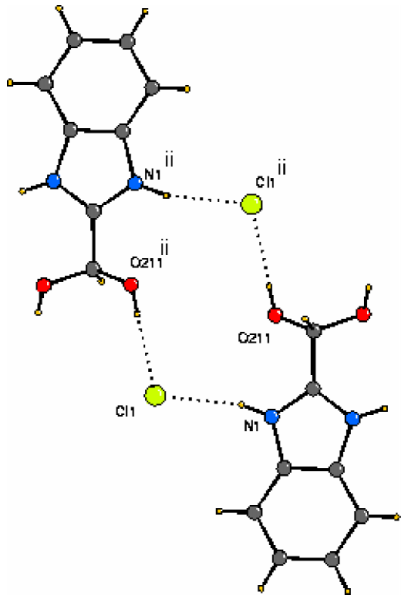


Figure 4
The $R_4^2(14)$ motif by which the ribbons are linked to form a sheet structure. Atoms are coloured as in Fig. 2. [Symmetry code: (ii) $1-x, 2-y, 2-z$.]

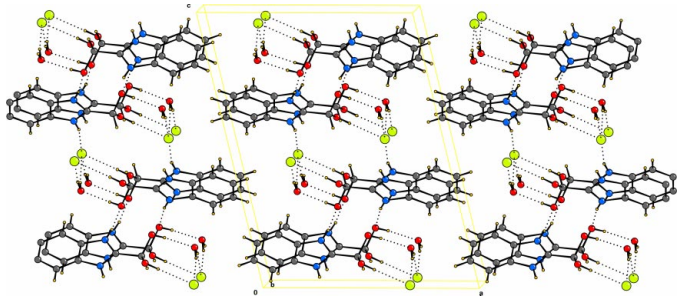


Figure 5
A projection of the structure approximately on to the (010) plane showing the interdigitation of the sheets. Atoms are coloured as in Fig. 2.

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