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Benzotriazolium perchlorate monohydrate

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The crystal structure of the title compound, $C_6H_6N_3^+\cdot ClO_4^-\cdot H_2O$, consists of cations, anions and water molecules linked by N—H···O and O—H···O hydrogen bonds into sheets of alternating $R_4^4(12)$ and $R_6^6(20)$ rings, which form chains running along the $[10\bar{1}]$ direction.

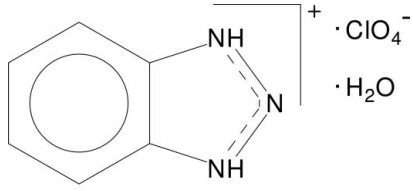
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.105
Data-to-parameter ratio = 12.7

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

Comment

Benzotriazole and its derivatives are used mainly as corrosion inhibitors for metals, antifreeze agents, antidust agents for photography and polymer stabilizers, as well as in industrial cooling systems. They are also widely used in chemical syntheses (Patsalides & Robards, 1985; Gruden *et al.*, 2001; Cancilla, *et al.*, 2003; Katritzky & Rogovoy, 2003). The title compound, (I), was investigated as part of a structural study on hydrogen-bonding patterns in *N*-heterocyclic perchlorate salts (Sieroń, 2005*a*,*b*, 2007).



(I)

In (I), there are benzotriazolium cations, perchlorate anions and water molecules (Fig. 1). All the interatomic distances and angles are normal (Allen *et al.*, 1987). One of the H atoms of the benzotriazolium ion and one of the water molecule are

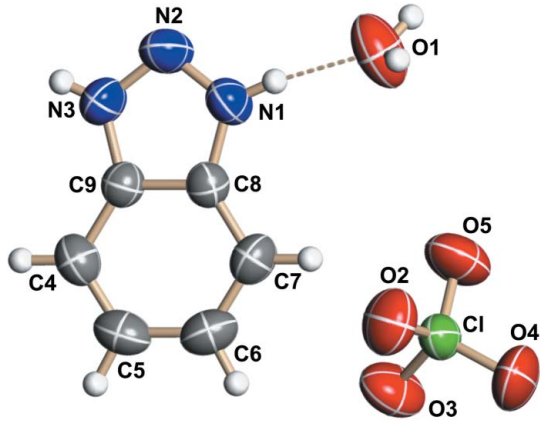


Figure 1
The asymmetric unit of (I) with the atom-numbering scheme. The displacement ellipsoids for the non-H atoms are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

engaged in a bifurcated unsymmetrical (strong and weak) hydrogen bond. Each of these hydrogen bonds involves two perchlorate O atoms (Table 1). A bifurcation is confirmed by the sums of angles about atoms H3 and H11, which are 354 and 359°, respectively (Jeffrey & Saenger, 1991). The ions and water molecules are linked by N—H···O and O—H···O hydrogen bonds, forming fused 12- and 20-membered cetrosymmetric rings, described by the graph-set notations as $R_4^4(12)$ and $R_6^6(20)$, respectively (Etter *et al.*, 1990). The substructure based on these motifs propagates along the $[10\bar{1}]$ direction (Fig. 2).

In addition, the benzotriazole rings are engaged in π–π stacking interactions, with distances between ring centroids of *ca* 3.7 Å, and with perpendicular distances between equivalent planes of *ca* 3.5 Å. The closest interatomic distances C5···C9($2-x, 1-y, -z$) and C7···C9($1-x, 1-y, -z$) are 3.495 (3) and 3.498 (3) Å, respectively.

Experimental

The title compound was prepared by dissolving benzotriazole (1 mmol) in perchloric acid (4 mmol) in water (20 ml). After a few days, prism-shaped colourless crystals up to 1 mm in size were obtained at room temperature.

Crystal data

$C_6H_6N_3^+\cdot ClO_4^-\cdot H_2O$
M_r = 237.60
Triclinic, $P\bar{1}$
a = 7.4227 (2) Å
b = 8.0524 (2) Å
c = 8.8973 (3) Å
α = 74.023 (3)°
β = 74.335 (3)°
γ = 87.246 (2)°
V = 492.07 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 K
0.50 × 0.50 × 0.30 mm

Data collection

Kuma KM-4 CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.827, T_{max} = 0.893
5415 measured reflections
1925 independent reflections
1842 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.105
S = 1.09
1925 reflections
151 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.82 (3)	1.83 (3)	2.646 (2)	171 (3)
N3—H3···O2^{i}	0.75 (3)	2.32 (3)	2.920 (3)	139 (3)
N3—H3···O4^{ii}	0.75 (3)	2.54 (3)	3.027 (3)	124 (2)
O1—H12···O2^{iii}	0.82	2.21	2.972 (3)	154
O1—H11···O3^{iv}	0.82	2.18	2.943 (3)	155
O1—H11···O3^{v}	0.82	2.52	3.031 (3)	121

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $x+1, y+1, z-1$; (iii) $-x, -y+1, -z+1$; (iv) $x, y+1, z$; (v) $-x+1, -y+1, -z+1$.

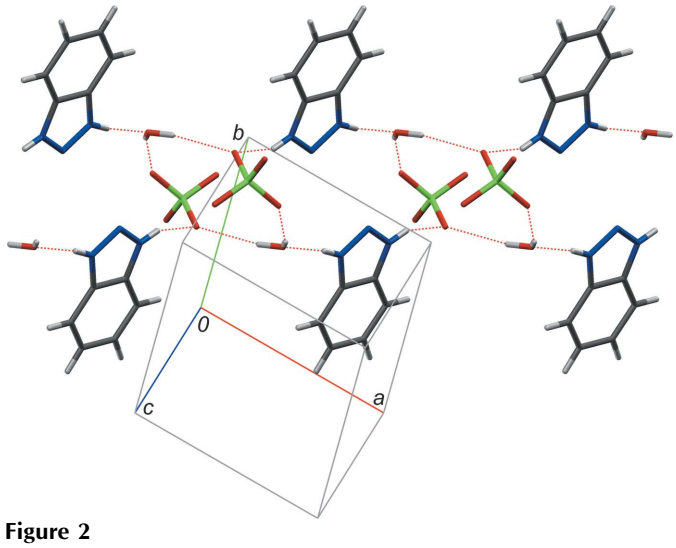


Figure 2
Packing diagram showing hydrogen bonds (dashed lines) running along the $[10\bar{1}]$ direction.

All H atoms were initially located in a difference Fourier map. The aryl H atoms were repositioned with idealized geometry keeping C—H = 0.93 Å and they were refined using a riding model. The H atoms of the water molecule were refined with the O—H distances restrained to 0.82 Å initially, and then refined using a riding model. The positions of the amine H atoms and the atomic displacement parameters of all the H atoms were refined freely.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2001); molecular graphics: *SHELXTL* and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2003).

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