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2,2′-(Ethane-1,2-diyl)dibenzimidazolium bis(perchlorate) tetrahydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.069
wR factor = 0.218
Data-to-parameter ratio = 14.6

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

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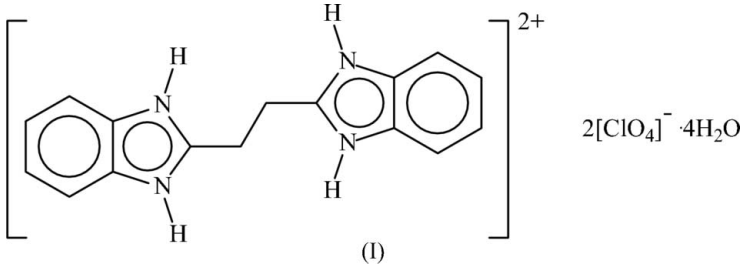
The cation, anion and water molecules of the title compound, $C_{16}H_{16}N_4^{2+}\cdot 2ClO_4^-\cdot 4H_2O$, are linked by hydrogen bonds into a three-dimensional network. The cation lies on a center of inversion, which is located at the mid-point of the $-CH_2CH_2-$ bond.

Comment

The 2,2′-(ethane-1,2-diyl)dibenzimidazole nitrogen heterocycle, whose synthesis has been described (Dudd *et al.*, 2003), affords crystalline compounds with metal salts; with some, the nitrogen heterocycle binds to the metal atom (Albada *et al.*, 1999, 2000), whereas in others it does not bind, existing instead as a protonated dication (Broughton *et al.*, 1998; Matthews *et al.*, 2003). An attempt to synthesize an erbium perchlorate complex gave 2,2′-(ethane-1,2-diyl)dibenzimidazolium bis(perchlorate) tetrahydrate, (I) (Fig. 1). The cation lies on a center of inversion, and its four nitrogen-bound H atoms are engaged in hydrogen bonding with the water molecules. The cation, anion and water molecules are linked by hydrogen bonds (Table 1) into a three-dimensional network.



(I)

Experimental

To a hot solution of 2,2′-(ethane-1,2-diyl)dibenzimidazole (0.07 g, 0.30 mmol) in ethanol (5 ml) was added a solution of erbium(III) perchlorate (0.34 g, 0.60 mmol) in acetonitrile (5 ml), giving a clear light-red solution. The solution was filtered and pale-yellow crystals were isolated from the filtrate after 10 d.

Crystal data

$C_{16}H_{16}N_4^{2+}\cdot 2ClO_4^-\cdot 4H_2O$
M_r = 535.29
Monoclinic, $P2_1/n$
a = 7.0898 (6) Å
b = 17.204 (2) Å
c = 9.9894 (9) Å
β = 110.072 (2)°
V = 1144.4 (2) Å^3
Z = 2
D_x = 1.553 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 295 (2) K
Block, pale yellow
0.20 × 0.16 × 0.11 mm

Data collection

Bruker APEX-II area-detector diffractometer
φ and ω scans
Absorption correction: none
7440 measured reflections
2601 independent reflections
1988 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.218$
$S = 1.05$
2601 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1223P)^2 + 0.991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.99$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H1w1···O1	0.84 (1)	2.13 (2)	2.915 (4)	154 (4)
O1w—H1w2···O2w^{i}	0.84 (1)	2.29 (2)	3.083 (4)	158 (4)
O2w—H2w1···O1^{ii}	0.85 (1)	2.11 (2)	2.907 (4)	157 (5)
O2w—H2w2···O1w^{iii}	0.85 (1)	2.13 (4)	2.897 (4)	150 (7)
N1—H1n···O1w	0.85 (1)	2.02 (1)	2.853 (4)	169 (4)
N2—H2n···O2w	0.85 (1)	1.96 (1)	2.804 (4)	172 (3)

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

The displacement parameters of the O atoms of the perchlorate were restrained to be nearly isotropic. The carbon-bound H atoms were placed at calculated positions (C—H = 0.93 and 0.97 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) set at $1.2U_{eq}$(C). The water and amino H atoms were located in a difference Fourier map, and were refined with distance restraints of O—H = N—H = 0.85 (1) Å and H···H = 1.39 (1) Å; their displacement parameters were freely refined.

Data collection: *APEX-2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

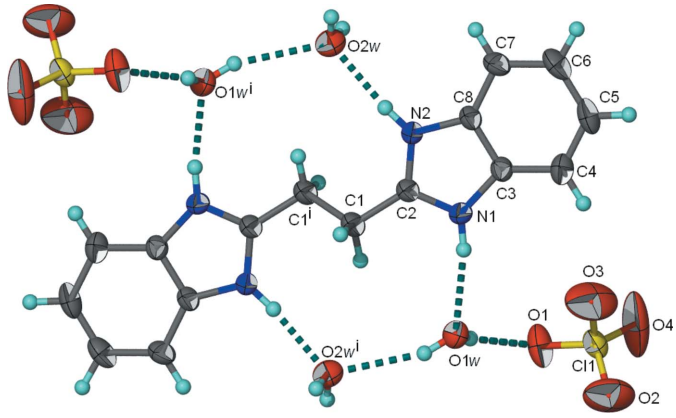


Figure 1
The formula unit of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

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