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Dabconium orthofluoroberyllate hemihydrate

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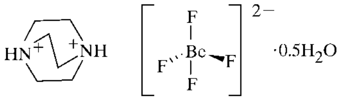
Alternating $C_6H_{14}N_2^{2+}$ and $[BeF_4]^{2-}$ units within the *ac* plane of 1,4-diazoniabicyclo[2.2.2]octane tetrafluoroberyllium hemihydrate, $(C_6H_{14}N_2)[BeF_4]\cdot0.5H_2O$, combine to form double chains *via* hydrogen bonding. These double chains connect through bifurcated hydrogen bonds to water molecules, which lie on twofold axes, producing a two-dimensional sheet. Very weak hydrogen-bond interactions, along the *b* axis, join the sheets to produce a three-dimensional network.

Comment

The title compound, (I), has been prepared as a precursor in the formation of beryllium-containing framework materials. Cocrystallization of the templating agent as a beryllium salt aids in the formation of these frameworks.



(I)

The beryllium coordination geometry is almost regular tetrahedral, with the four Be—F bond distances ranging from 1.524 (2) to 1.572 (2) Å, and the F—Be—F angles ranging from 105.8 (1) to 113.7 (1)°. These values are in good agreement with other $[BeF_4]^{2-}$ geometries in the literature (Le Fur *et al.*, 1991; Srivastava *et al.*, 1999; Tedenac *et al.*, 1971). In the doubly protonated 1,4-diazabicyclo[2.2.2]octane (dabco), the C—N distances of 1.485 (3) Å and C—C distances of 1.509 (3) Å are typical of the average values found for this group (Fig. 1 and Table 1).

Each $[BeF_4]^{2-}$ unit is linked to two dabconium moieties and one water molecule through bifurcated N—H···F and O—H···F interactions, which have *D*···*A* distances in the range 2.5932 (16)–3.0737 (17) Å (Table 2) [weaker interactions, namely C—H···F contacts characterized by C···F distances of 3.091 (2)–3.339 (2) Å, have not been tabulated]. Thus, one-dimensional hydrogen-bonded chains consisting of $C_6H_{14}N_2^{2+}$ and $[BeF_4]^{2-}$ units run parallel to the *b* axis. These chains are linked in the *ab* plane by weak hydrogen-bonding interactions involving water molecules, linking pairs of parallel chains (Fig. 2) into hydrogen-bonded double-chain units. These units lie parallel to one another, intersecting the normal to the *ac* plane. Water molecules lie between the hydrogen-bonded double-chain units, where each parallel set is rotated through 90° to give an alternating pattern.

Other possible hydrogen bonds have been found using *PLATON* (Spek, 1990, 1998), which indicates very weak C—H···F interactions between the dabconium C—H groups and the F atoms of the $[BeF_4]^{2-}$ dianion. For each water link there

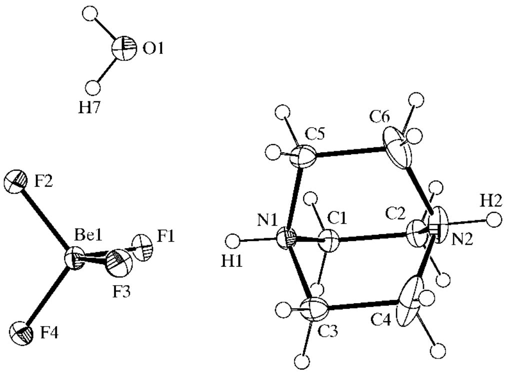


Figure 1
The three discrete moieties in the title compound. Displacement ellipsoids are shown at the 30% probability level.

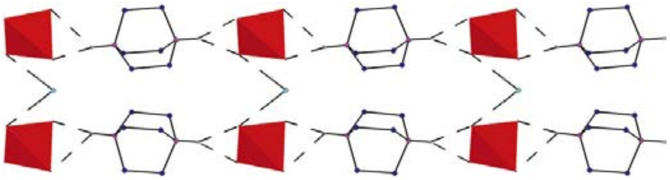


Figure 2
The hydrogen-bonding arrangement forming the double chain, viewed along the *bc* plane.

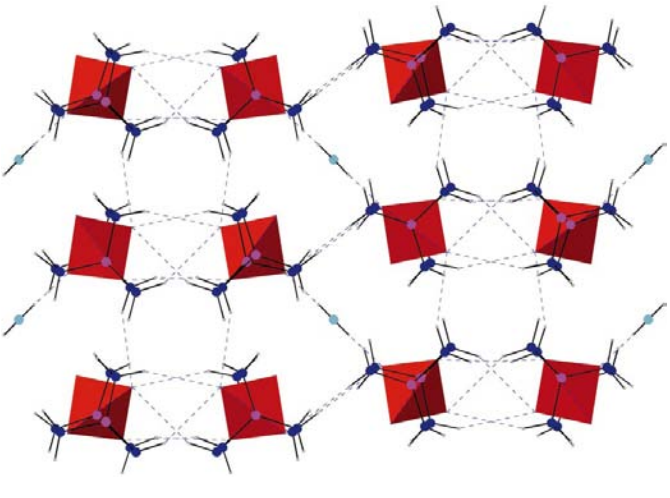


Figure 3
The three-dimensional hydrogen-bonded network, viewed down the *b* axis.

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are two C—H···F crosslinks between the parallel double chains (Fig. 3). Overall, these hydrogen bonds result in the formation of a three-dimensional network.

Experimental

Beryllium(II) fluoride (0.1 g, 0.002 mol) was dissolved in an acidic aqueous solution of distilled water (2 ml) and 30% hydrofluoric acid (0.085 ml, 0.002 mol). 1,4-Diazabicyclo[2.2.2]octane (dabco) (0.224 g, 0.002 mol) was added to give an overall molar ratio of 1:1:1. The solution was placed in a 23 ml Teflon-lined autoclave and heated at 423 K for 24 h. The resulting solution was placed in a plastic sample vial and the solvent allowed to evaporate. The title compound crystallized as a colourless crystalline solid. Finally, the product was recovered by filtration and dried in air.

Crystal data

$(C_6H_{14}N_2)[BeF_4]\cdot 0.5H_2O$
$M_r = 208.21$
Orthorhombic, *Pbcn*
$a = 19.523$ (4) Å
$b = 9.2726$ (19) Å
$c = 9.939$ (2) Å
$V = 1799.3$ (6) Å^3
$Z = 8$
$D_x = 1.537$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 14 284 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.15$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.12 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
14 284 measured reflections
2041 independent reflections
1814 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.051$
$\theta_{\rm max} = 27.5°$
$h = -24 \rightarrow 25$
$k = -12 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.117$
$S = 1.07$
2040 reflections
135 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 1.245P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.39$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.40$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Be1—F1	1.567 (2)	Be1—F3	1.524 (2)
Be1—F2	1.572 (2)	Be1—F4	1.546 (2)
F1—Be1—F2	108.27 (13)	F2—Be1—F3	111.08 (12)
F1—Be1—F3	107.60 (12)	F2—Be1—F4	105.86 (12)
F1—Be1—F4	110.24 (12)	F3—Be1—F4	113.69 (13)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F1$^{\rm i}$	0.90 (2)	1.71 (2)	2.5932 (16)	166 (2)
N1—H1···F3$^{\rm i}$	0.90 (2)	2.48 (2)	3.0737 (17)	123 (2)
N2—H2···F2$^{\rm ii}$	0.89 (3)	1.93 (3)	2.7320 (18)	149 (2)
N2—H2···F4$^{\rm ii}$	0.89 (3)	2.10 (3)	2.7927 (18)	134 (2)
O1—H7···F2	0.82 (2)	1.97 (2)	2.7881 (15)	176 (3)

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

Two of the three dabconium ethylene groups showed slight disorder. Each group has one C atom with an elongated displacement parameter perpendicular to the C—C and C—N bonds. A static disorder model was investigated as an alternative to the dynamic disorder model but the latter was preferred due to the instability of the former model during refinement. H atoms were initially located in difference maps and their positions idealized. Thereafter they were refined riding on their parent atoms, with C—H distances of 0.99 Å and $U_{\rm iso}$(H) values of $1.2U_{\rm eq}$(C) for Csp^3 atoms. The H atoms of the N—H and O—H groups were located and freely refined.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *COLLECT*, *DENZO* (Otwinowski & Minor, 1997) and *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1497). Services for accessing these data are described at the back of the journal.

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