metal-organic compounds

Propane-1,2-diammonium tetrafluoroberyllate

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Cocrystallization of the inorganic $[BeF_4]^{2-}$ unit with the organic moiety $[NH_3CH_2CH(NH_3)CH_3]^{2+}$ results in the three-dimensional network of the title compound, $(C_3H_{12}N_2)[BeF_4]$ or $C_3H_{12}N_2^{2+} \cdot BeF_4^{2-}$, created by hydrogen bonds between the protonated ammonium groups and the highly electronegative F atoms of the anion. The structure is described in terms of layers related to each other by crystallographic centres of symmetry.

Comment

Three-dimensional tetrahedral Be frameworks are very rare (Le Fur *et al.*, 1991). Using various organic templating units, Be salts have been synthesized to provide precursors for the production of fluoro-frameworks (Le Fur *et al.*, 1991; Anderson *et al.*, 1973). Aqueous HF solutions, under either hydrothermal or ambient conditions, have proved to be satisfactory for the synthesis of such salts and the title compound, (I), is an example.

(I)
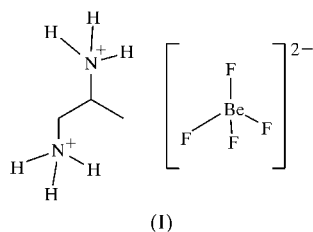
The coordination of Be in (I) is in the form of a slightly distorted tetrahedron (Fig. 1 and Table 1). The Be—F bond lengths [1.5352 (17)–1.5752 (17) Å] and the F—Be—F angles [106.86 (11)–111.12 (11)°] show little variation and are in good agreement with other similar geometries (Srivastava *et al.*, 1999; Tedenac *et al.*, 1971; Collins *et al.*, 1983; Hahn & Chung, 1972). In the doubly protonated 1,2-diaminopropane group, average C—N [1.492 (1) Å] and C—C [1.521 (2) Å] distances (Table 1) are in good agreement with normal values (Orpen *et al.*, 1992).

Each BeF$_4$ tetrahedron in (I) is linked to six doubly protonated 1,2-diaminopropane units through N—H···F
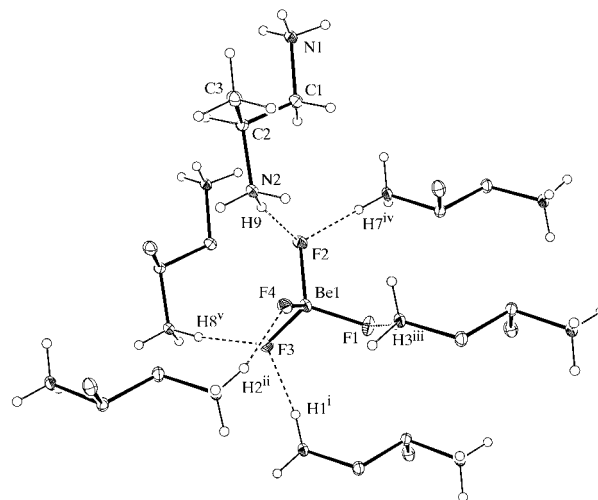
Figure 1
The structure of (I), showing the role of the $[BeF_4]^{2-}$ anion in hydrogen-bond formation (dashed lines). Displacement ellipsoids are drawn at the 50% probability level. H atoms connected to the C atoms of symmetry-related cations have been omitted for clarity, while the remaining H atoms are shown as open circles. The labelling scheme encompasses all non-H atoms of the asymmetric unit and selected other atoms [symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (v) $1 + x, y, z$].
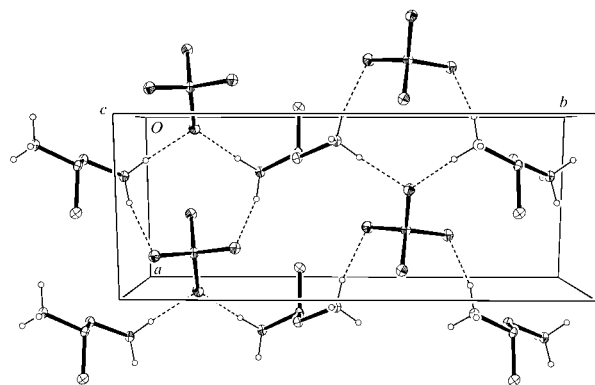
Figure 2
A portion of a hydrogen-bonded layer in (I), viewed parallel to (001), $\frac{c}{2}$ thick and centred on $z = \frac{1}{4}$.
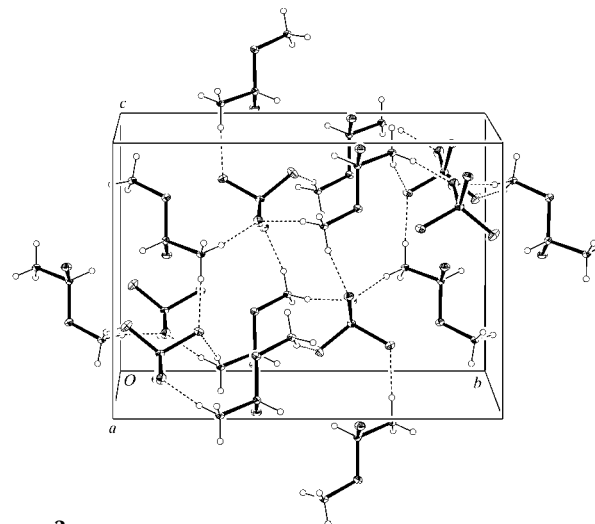
Figure 3
The cell of (I) viewed along **a**.

hydrogen bonds, characterized by H···F distances of 1.78 (2)–1.91 (2) Å (Table 2 and Figs. 1–3). Four of these interactions, namely those involving atoms H1, H3, H8 and H9, connect the ions to form sheets perpendicular to (001) (Fig. 2), in which $R_4^3(10)$ and $R_4^4(18)$ connectivity is evident (Motherwell *et al.*, 1999). These sheets, related to each other by crystallographic centres of symmetry and stacked in the *c* direction, are connected by two further hydrogen bonds (Fig. 3), involving atoms H2 and H7, to create, as the smallest and simplest examples, centrosymmetric $R_4^4(12)$ and non-centrosymmetric $R_6^5(19)$ connectivities.

Experimental

BeF$_2$ (0.100 g, 0.0022 mol) was dissolved in an acidic aqueous mixture of distilled water (2 ml) and 30% hydrofluoric acid (0.085 ml, 0.002 mol). 1,2-Diaminopropane (0.17 ml, 0.002 mol) was added to give an overall molar ratio of 1:1:1. The resulting solution was placed in a plastic sample vial and left to concentrate slowly by evaporation, yielding colourless crystals of (I), which were recovered by filtration and air dried.

Crystal data

(C$_3$H$_{12}$N$_2$)[BeF$_4$]	$D_x = 1.55$ Mg m^{-3}
$M_r = 161.16$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1114
$a = 5.5355$ (11) Å	reflections
$b = 13.560$ (3) Å	$\theta = 2.9$–27.5°
$c = 9.6048$ (19) Å	$\mu = 0.17$ mm^{-1}
$\beta = 106.73$ (3)°	$T = 120$ (2) K
$V = 690.4$ (3) Å^3	Block, colourless
$Z = 4$	0.14 × 0.08 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1530 independent reflections
φ and ω scans to fill the Ewald sphere	1294 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.045$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	$\theta_{max} = 27.5°$
	$h = -6 \rightarrow 7$
$T_{min} = 0.745$, $T_{max} = 0.993$	$k = -14 \rightarrow 17$
3623 measured reflections	$l = -12 \rightarrow 9$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.033P)^2$
$R(F) = 0.030$	$+ 0.118P]$
$wR(F^2) = 0.077$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
1530 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
135 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
H atoms: see below	

All H atoms, with one exception (H6), were located in difference maps and refined isotropically. Atom H6, attached to tertiary atom C2, which could not be refined satisfactorily in this manner, was fixed in a position with C—H = 1.00 Å and refined using a riding model with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 1990, 1998).

Table 1
Selected geometric parameters (Å, °).

Be1—F1	1.5352 (17)	Be1—F4	1.5492 (17)
Be1—F2	1.5752 (17)	N1—C1	1.4849 (16)
Be1—F3	1.5603 (19)	N2—C2	1.4969 (15)
N1—C1—C2	112.17 (10)	F1—Be1—F4	109.50 (11)
N2—C2—C3	107.73 (10)	F2—Be1—F3	109.67 (11)
N2—C2—C1	106.04 (10)	F2—Be1—F4	106.86 (11)
F1—Be1—F2	110.97 (10)	F3—Be1—F4	108.59 (10)
F1—Be1—F3	111.12 (11)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F3^{vi}	0.90 (2)	1.86 (2)	2.7461 (13)	169.8 (13)
N1—H2···F4^{vii}	0.88 (2)	1.78 (2)	2.6446 (13)	165.4 (15)
N1—H3···F1^{viii}	0.90 (2)	1.89 (2)	2.7646 (15)	163.5 (15)
N2—H7···F2^{ix}	0.88 (2)	1.87 (2)	2.7507 (14)	175.5 (14)
N2—H8···F3^{x}	0.90 (2)	1.87 (2)	2.7611 (13)	171.8 (13)
N2—H9···F2	0.94 (2)	1.91 (2)	2.8240 (16)	163.3 (14)

Symmetry codes: (vi) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (vii) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (viii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ix) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (x) $x - 1, y, z$.

References

Anderson, M. R., Brown, I. D. & Vilminot, S. (1973). *Acta Cryst.* B**29**, 2625–2627.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.
Collins, D. M., Mahar, M. C. & Whitehurst, F. W. (1983). *Acta Cryst.* B**39**, 303–306.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hahn, Th. & Chung, S. J. (1972). *Mater. Res. Bull.* **7**, 1209–1218.
Le Fur, B. Y., Aléonard, S. & Gorius, M. F. (1991). *Acta Cryst.* C**47**, 949–951.
Motherwell, W. D. S., Shields, G. P. & Allen, F. H. (1999). *Acta Cryst.* B**55**, 1044–1056.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1992). *International Tables for Crystallography*, Vol. C, edited by A. J. C. Wilson, pp. 176–177. Dordrecht: Kluwer Academic Publishers.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Spek, A. L. (1998). *PLATON*. University of Utrecht, The Netherlands.
Srivastava, R. C., Klooster, W. T. & Koetzle, T. F. (1999). *Acta Cryst.* B**55**, 17–23.
Tedenac, J. C., Vilminot, S., Cot, L., Norbert, A. & Maurin, M. (1971). *Mater. Res. Bull.* **6**, 183–188.