O···N being 2·937 (3) Å and the angle O—H···N 166·5 (4)°. These bonds are depicted in Fig. 2 as dashed lines. In this way the Cu and O atoms form alternating chains in the cell parallel to the *c* axis. The Cu—Cu distance in this chain is 5·964 (5) Å, Cu—O 2·239 (4) and Cu···O 3·725 (4) Å.

There is no intermolecular stacking of the aromatic rings; closest distances between the tp rings are larger than 4·6 Å. The packing of the molecules is dictated by normal van der Waals contacts between the cationic chains and the bromide ions and the hydrogen bonds.

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Structure of Ethylenediammonium Orthofluoroberyllate

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Abstract. $[C_2H_{10}N_2][BeF_4]$, $M_r = 147·12$, tetragonal, $P4_12_12$, $a = 5·904$ (1), $c = 17·841$ (4) Å, $V = 621·9$ (3) Å^3, $Z = 4$, $D_x = 1·571$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 3·033$ mm^{-1}, $F(000) = 304$, room temperature, final $R = 0·038$ for 449 independent observed reflections. BeF_4 tetrahedra and $[(CH_2)_2(NH_3)_2]^{2+}$ groups alternate in planes perpendicular to the c axis. Hydrogen bonds link together all the organic groups and BeF_4 tetrahedra to form a three-dimensional network.

Introduction. Up to now, only two types of ethylenediammonium fluoroberyllates have been synthetized and characterized: a metafluoroberyllate $(CH_2)_2(NH_3)_2(BeF_3)_2$, under study, and an orthofluoroberyllate $(CH_2)_2(NH_3)_2BeF_4$, the title compound.

Experimental. Crystals are prepared by slowly adding ethylenediammonium chloride, in a stoichiometric ratio, to a water solution of silver orthofluoroberyllate obtained by the reaction

$$Ag_2CO_3 + BeCO_3 + 4HF \rightarrow Ag_2BeF_4 + 2CO_2 + 2H_2O,$$

$$Ag_2BeF_4 + 2(CH_2)NH_3Cl \rightarrow 2AgCl + (CH_2)_2(NH_3)_2BeF_4.$$

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Table 1. *Final atomic coordinates and* B_{eq}(Å^2) *values for non-*H *atoms*; *e.s.d.'s are given in parentheses*

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Be	0·7231 (6)	0·7231 (6)	0·000	1·90 (4)
F(1)	0·2462 (3)	0·5182 (3)	0·46853 (8)	3·13 (3)
F(2)	0·1065 (3)	0·2362 (3)	0·56355 (8)	3·33 (3)
N	0·2865 (4)	0·4862 (4)	0·3149 (1)	2·56 (4)
C	0·2423 (5)	0·7223 (5)	0·2914 (1)	2·65 (5)

Table 2. *Main interatomic distances* (Å), *bond angles* (°) *and hydrogen bonds* (Å,°) *in the atomic arrangement of* $(CH_2)_2(NH_3)_2BeF_4$; *e.s.d.'s are given in parentheses*

BeF_4 tetrahedron

Be	F(11)	F(12)	F(21)	F(22)
F(11)	1·542 (2)	2·533 (2)	2·515 (2)	2·498 (2)
F(12)	110·5 (2)	1·542 (3)	2·548 (4)	2·476 (4)
F(21)	109·7 (1)	108·6 (2)	1·535 (3)	2·513 (2)
F(22)	108·6 (1)	109·7 (2)	109·9 (2)	1·535 (2)

NH_3–$(CH_2)_2$–NH_3 group

N—C	1·479 (4)	N—C—C	112·7 (2)	C—C	1·505 (3)

Hydrogen bonds

N—H···F	N—H	H···F	N···F	N—H···F
N—H(1*N*)···F(1)	0·90 (2)	1·90 (2)	2·75 (3)	156 (3)
N—H(2*N*)···F(2)	1·05 (3)	1·67 (3)	2·65 (3)	174 (2)
N—H(3*N*)···F(1)	1·00 (3)	1·73 (3)	2·83 (3)	164 (3)

After filtration of AgCl and slow evaporation at room temperature, large prisms with square section are obtained. Crystal size: 0·18 × 0·19 × 0·24 mm. Density not measured. Nonius CAD-4 diffractometer, graphite monochromator. Systematic absences: $00l$, $l = 4n$; $h00$, $h = 2n$. 25 reflections ($10·0 < \theta < 14·0°$) for refining unit-cell dimensions. ω scan, scan width: 1·20°, scan speed: 1·33–2·5 ° min^{-1}, background measuring time: 8–15 s. 1086 reflections collected ($3 < \theta < 35°$), $+h$, $+k$, $+l$, $h_{max} = 9$, $k_{max} = 9$, $l_{max} = 28$. Two orientation (400 and $\bar{4}00$) and two intensity (040 and $0\bar{4}0$) control reflections measured every two hours without any significant variation. 878 independent reflections obtained after averaging Friedel pairs ($R_{int} = 0·024$). Lorentz and polarization corrections, no absorption correction. Crystal structure solved by direct methods: *MULTAN*77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Anisotropic full-matrix least-squares refinement on F, isotropic for H atoms. Unit weights. Final refinements with 449 reflections corresponding to $I > 2\sigma_I$. Final $R = 0·038$ ($wR = 0·034$), $S = 0·485$, max. $\Delta/\sigma = 0·04$, max. peak height in the final difference Fourier synthesis = 0·23 e Å^{-3}. No extinction correction. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). Enraf–Nonius (1977) *SDP* employed for all calculations. Computer used: MicroVAX II.

Discussion. Table 1 reports the final atomic coordinates.* Table 2 gives the main interatomic distances, bond angles in the BeF_4 tetrahedra and $[(CH_2)_2(NH_3)_2]$ groups and geometrical characteristics of the hydrogen bonds.

These results show that the fluoroberyllate $(CH_2)_2(NH_3)_2BeF_4$ is isotypic with the sulfate $(CH_2)_2(NH_3)_2SO_4$. However, the last structural determination of this compound was performed in 1961 (Sakurai, 1961) and is only an approximate one, mainly for the thermal factors and the hydrogen-bonding scheme.

The BeF_4 tetrahedra and $[(CH_2)_2(NH_3)_2]$ groups lie approximately in planes perpendicular to the c axis, at $z \simeq 0, \frac{1}{4}, \frac{1}{2}, \frac{3}{4}$. Fig. 1 represents the atomic arrangement in one of the planes ($z \simeq 0$). The fluoroberyllate groups are very regular, with the usual values for Be—F distances [1·542 (3) and 1·535 (3) Å] and F—Be—F angles (109·3 ± 1·2)°. In the ethylenediammonium groups, distances C—N [1·479 (4) Å], C—C [1·505 (3) Å] and the N—C—C angle [112·7 (2)°] are in good agreement with the average values found for this group.

In each plane, each ethylenediammonium group is surrounded by four BeF_4 tetrahedra, but is only linked to three of them by hydrogen bonds: two strong bonds N—H(2*N*)···F(2) with one BeF_4 tetrahedron [N···F = 2·65 (3) Å] and a weaker one, N—H(3*N*)···F(1), with each of the two others [N···F =

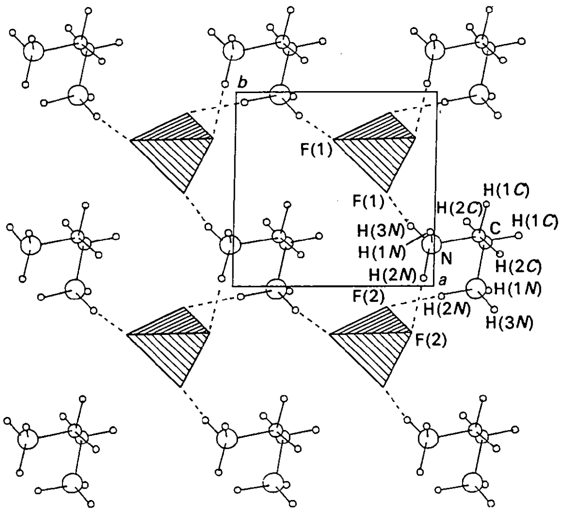

Fig. 1. Atomic arrangement of $(CH_2)_2(NH_3)_2BeF_4$ in the *ab* plane.

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, H-atom coordinates and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53712 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

2·83 (3) Å]. The ethylenediammonium groups and BeF_4 tetrahedra alternate and form chains parallel to the [110] direction. These chains are linked together by N—H(2*N*)···F(2) bonds between an ethylenediammonium group of one chain and a tetrahedron of the other.

Fig. 2 is a projection of the atomic arrangement along the *b* axis. It shows that two (001) neighbouring planes are linked together by hydrogen bonds N—H(1*N*)···F(1) between one ethylenediammonium group in one plane to a BeF_4 tetrahedron in the other [N···F = 2·75 (3) Å]. The number and the characteristics of these bonds do not account for the existence of a cleavage parallel to (001) that was proposed by Sakurai for the sulfate, but is not observed with the fluoroberyllate crystals. The drawings were performed using the *STRUPLO84* program (Fischer, 1985).

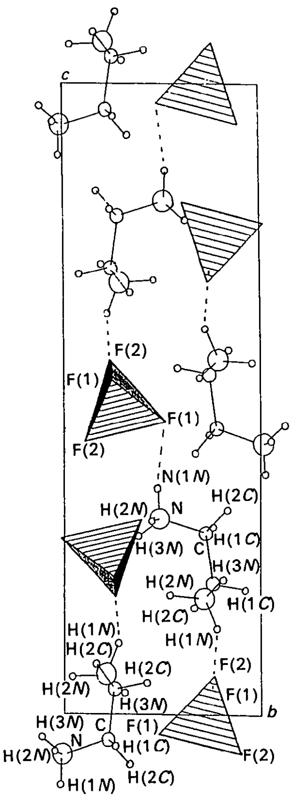

Fig. 2. Projection along the *b* axis of the atomic arrangement of $(CH_2)_2(NH_3)_2BeF_4$.

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1,1,2,2,3,3,4,4,5,5,6,6-Dodecamethyl-1,2,4,5-tetrastannacyclohexane

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Abstract. $C_{14}H_{36}Sn_4$, M_r = 679·20, monoclinic, $P2_1/n$, a = 8·236 (3), b = 13·584 (5), c = 10·738 (3) Å, β = 94·27 (3)°, V = 1198·0 (7) Å^3, Z = 2, D_x = 1·883 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 4·13 mm^{-1}, F(000) = 640, T = 291 (1) K, final R = 0·028 for 1999 unique observed [$F \geq 3{\cdot}0\sigma(F)$] diffractometer data. In the crystal the molecule resides on a centre of symmetry. The central 1,2,4,5-tetrastannacyclohexane ring has a chair conformation with an Sn—Sn bond length of 2·7753 (8) Å. The structure consists of discrete molecular units.

Introduction. Previous work (Meunier-Pieret, Van Meerssche, Gielen & Jurkschat, 1983; Preut, Bleckmann, Mitchell & Fabisch, 1984; Preut & Mitchell, 1989) indicated that while 1,2,4,5-tetrastannacyclohexanes bearing phenyl substituents at Sn prefer a chair conformation, a boat conformation is observed when the Sn atoms bear methyl substituents. In order to probe the influence of increasing substitution at the ring C atoms we have now determined the crystal structure of the title compound.

Experimental. The title compound was prepared by adding a solution of 1,3-dibromo-1,1,2,2,3,3-hexamethyl-1,3-distannapropane (0·04 mol) in ether (250 ml) to sodium (0·1 mol) in liquid ammonia

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