Acta Cryst. (1973). B29, 2961

The Crystal Structure of Hydrazinium Fluoroberyllate

BY M. R. ANDERSON, S. VILMINOT* AND I. D. BROWN

Institute for Materials Research, McMaster University, Hamilton, Ontario, Canada, L8S 4M1

(*Received* 31 *July* 1973; *accepted* 1 *August* 1973)

Abstract. Hydrazinium fluoroberyllate, $(N_2H_6)BeF_4$, is monoclinic, space group $P2_1/c$, with $a=5{\cdot}568$ (2),† $b=7{\cdot}305$ (2), $c=9{\cdot}910$ (4) Å, and $\beta=98{\cdot}25$ (3)°, $Z=4$. The structure was solved by direct methods and refined by least-squares calculations to give R(weighted) = 0·043 for 770 X-ray reflexions measured from a crystal sealed in a dried quartz capillary tube. The structure consists of BeF_4^{2-} tetrahedra (mean Be–F = 1·547 Å) and $N_2H_6^{2+}$ ions linked by hydrogen bonds.

Introduction. $(N_2H_6)BeF_4$ was prepared by the action of hydrofluoric acid on a mixture of $N_2H_4.H_2O$ and $Be(OH)_2$ in aqueous solution (Tédenac, Vilminot, Cot, Norbert & Maurin, 1971). The crystals were washed with water and dried by warming under vacuum before being sealed in dried quartz capillaries to prevent surface decomposition. The space group $P2_1/c$ was confirmed by systematic absences (Table 1) observed on precession photographs. All X-ray diffraction measurements were made at room temperature on a Syntex four-circle automatic diffractometer with Mo $K\alpha$ radiation monochromated by reflexion from a graphite crystal. The lattice parameters (Table 1) were refined by a least-squares analysis of the 2θ measurements of fifteen reflexions. The intensities of 770 independent reflexions with $\sin\theta/\lambda<0{\cdot}60$ were measured and corrected for Lorentz and polarization effects. No absorption correction was made, the maximum error in F introduced by its neglect being less than 1 %.

Table 1. *Crystallographic data for* $(N_2H_6)BeF_4$

Crystal system	Monoclinic
Space group	$P2_1/c$
a	5·568 (2) Å
b	7·305 (2)
c	9·910 (4)
β	98·25 (3)°
Z	4
D_{calc}	1·983 g cm^{-3}
Absorption coefficient for Mo $K\alpha$	0·28 mm^{-1}
Crystal size	0·1 × 0·1 × 0·15 mm
Wavelength Mo $K\alpha$	0·71069 Å
Systematic absences	$h0l$ $l=2n+1$
	$0k0$ $k=2n+1$

The structure was solved with the direct methods programs *PHASE* and *SINGEN* of the X-RAY 71 system. Initially 90 reflexions were correctly phased and used to calculate a three-dimensional electron-density map from which all non-hydrogen atoms were located. After a least-squares refinement of these atoms with the program *CRYLSQ*, all hydrogen atoms were located from difference maps. Further refinement led to an $R_1[=\sum(||F_o|-|F_c||)/\sum|F_o|]$ of 0·044. There was no evidence of extinction and a final refinement gave $R_2[=(\sum w(|F_o|-|F_c|)^2/\sum w|F_o|^2)^{1/2}]$ of 0·043, where

* On leave from Laboratoire de Chimie Minérale, Chimie des Matériaux E.R.A. 314, Faculté des Sciences, Place Eugène Bataillon, 34 Montpellier, France.

† Throughout this paper standard errors in the last quoted figures are shown in parentheses.

Table 2. *Parameters derived from the final least-squares refinement*

Expressions used for the temperature factors are:

$\exp[-2\pi^2\times10^{-3}(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)]$ and $\exp[-2\pi^2\times10^{-3}\,U(2\sin\theta/\lambda)^2]$.

	x	y	z	U or U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Be	0·7545 (11)	0·2993 (8)	0·0634 (6)	16 (3)	19 (3)	18 (3)	2 (3)	5 (2)	2 (3)
F(1)	0·5276 (4)	0·1835 (4)	0·0835 (3)	25 (1)	27 (2)	25 (2)	−9 (1)	4 (1)	−1 (1)
F(2)	−0·0182 (5)	0·1752 (4)	0·0943 (3)	31 (2)	23 (2)	21 (1)	8 (1)	3 (1)	2 (1)
F(3)	0·7825 (5)	0·4541 (3)	0·1701 (3)	29 (1)	17 (1)	29 (2)	1 (1)	0 (1)	−3 (1)
F(4)	0·7247 (5)	0·1372 (4)	0·4137 (3)	27 (1)	34 (2)	23 (1)	1 (1)	2 (1)	−7 (1)
N(1)	0·3593 (9)	0·3602 (7)	0·2982 (5)	26 (2)	32 (3)	29 (3)	7 (2)	12 (2)	10 (2)
N(2)	0·1510 (8)	0·3097 (7)	0·3566 (5)	21 (2)	25 (2)	30 (2)	3 (2)	8 (2)	8 (2)
H(1)	0·419 (11)	0·483 (10)	0·330 (7)	24 (19)					
H(2)	0·486 (11)	0·300 (8)	0·324 (6)	13 (16)					
H(3)	0·294 (11)	0·374 (9)	0·206 (7)	28 (18)					
H(4)	0·032 (11)	0·360 (9)	0·315 (6)	17 (18)					
H(5)	0·142 (11)	0·187 (10)	0·350 (7)	25*					
H(6)	0·179 (11)	0·332 (9)	0·443 (7)	25*					

* Not refined.

$w=(1\cdot14-0\cdot073|F_o|+0\cdot0014|F_o|^2)^{-1}$.* Final atomic positions and temperature factors are given in Table 2.

Discussion. Views of the structure along **a** and **b** are given in Figs. 1 and 2. The crystal contains nearly regular BeF_4^{2-} tetrahedra and $N_2H_6^{2+}$ ions (Table 3) held together by a three-dimensional system of one bifurcated, one trifurcated and four single hydrogen bonds (Table 4). Three of the fluorine atoms form two hydrogen bonds and one bond to Be [mean Be–F = 1·544 (5) Å] and one fluorine atom forms three hydrogen bonds and a rather longer bond to Be [Be–F = 1·557 (8) Å].

Table 3. *Bond distances* (Å) *and angles* (°)

BeF_4 tetrahedron			
Be—F(1)	1·557 (8)	F(1)–Be—F(2)	108·1 (4)
Be—F(2)	1·552 (10)	F(1)–Be—F(3)	108·4 (4)
Be—F(3)	1·540 (7)	F(1)–Be—F(4)	108·0 (4)
Be—F(4)	1·540 (8)	F(2)–Be—F(3)	107·1 (4)
		F(2)–Be—F(4)	109·8 (4)
		F(3)–Be—F(4)	115·3 (4)
N_2H_6 ion			
N(1)–N(2)	1·417 (16)	N(2)–N(1)–H(1)	111 (4)
N(1)–H(1)	1·00 (7)	N(2)–N(1)–H(2)	116 (4)
N(1)–H(2)	0·84 (6)	N(2)–N(1)–H(3)	102 (4)
N(1)–H(3)	0·94 (7)	H(1)–N(1)–H(2)	98 (5)
		H(1)–N(1)–H(3)	106 (5)
		H(2)–N(1)–H(3)	123 (6)
N(2)–H(4)	0·81 (6)	N(1)–N(2)–H(4)	109 (5)
N(2)–H(5)	0·90 (7)	N(1)–N(2)–H(5)	106 (4)
N(2)–H(6)	0·86 (7)	N(1)–N(2)–H(6)	108 (4)
		H(4)–N(2)–H(5)	112 (6)
		H(4)–N(2)–H(6)	116 (5)
		H(5)–N(2)–H(6)	105 (6)

Table 4. *Hydrogen-bond lengths* (Å) *and angles* (°)

D–H···A	D–H (Å)	H···A (Å)	D–A (Å)	∠D–H···A (°)
N(1)–H(1)···F(1)	1·00 (7)	1·70 (7)	2·67 (1)	165 (6)
N(1)–H(2)···F(4)	0·84 (6)	1·91 (6)	2·73 (3)	165 (6)
N(1)–H(3)···F(1)	0·94 (7)	2·35 (7)	2·77 (2)	106 (5)
N(1)–H(3)···F(2)		2·41 (7)	3·02 (4)	122 (5)
N(1)–H(3)···F(4)		2·25 (7)	2·91 (1)	126 (5)
N(2)–H(4)···F(3)	0·81 (6)	1·97 (7)	2·77 (4)	164 (6)
N(2)–H(5)···F(3)	0·90 (7)	1·77 (7)	2·64 (1)	163 (6)
N(2)–H(6)···F(1)	0·86 (7)	2·23 (7)	2·85 (5)	129 (6)
N(2)–H(6)···F(2)		1·98 (8)	2·66 (3)	135 (6)

* The observed and calculated structure factors have been deposited with the National Lending Library, England, as Supplementary Publication Number SUP 30189 (6 pp.) and are also given by Anderson (1973). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

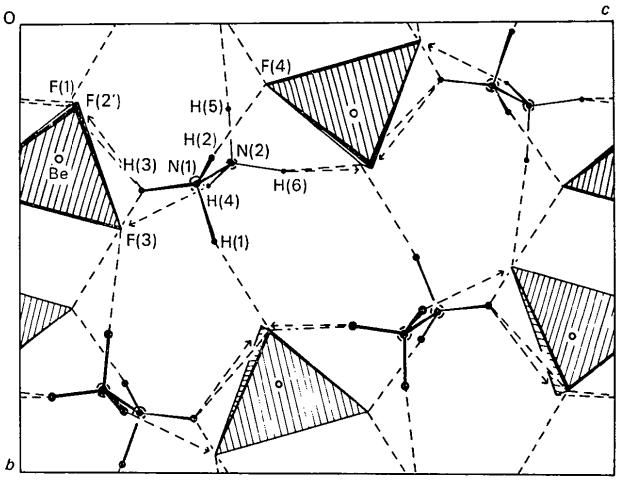

Fig. 1. Structure of $(N_2H_6)BeF_4$ projected down **a**.

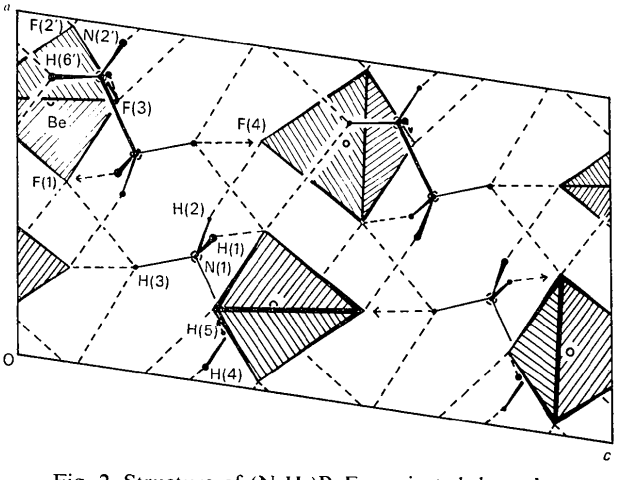

Fig. 2. Structure of $(N_2H_6)BeF_4$ projected down **b**.

We thank the National Research Council of Canada for an operating grant and one of us (S.V.) thanks the Centre National de la Recherche Scientifique (France) for financial support.

References

ANDERSON, M. R. (1973). Ph. D. Thesis, McMaster Univ.
TÉDENAC, J. C., VILMINOT, S., COT, L., NORBERT, A. & MAURIN, M. (1971). *Mater. Res. Bull.* **6**, 183–188.