Crystal structure of triammonium heptafluorogermanate, $(NH_4)_3GeF_7$

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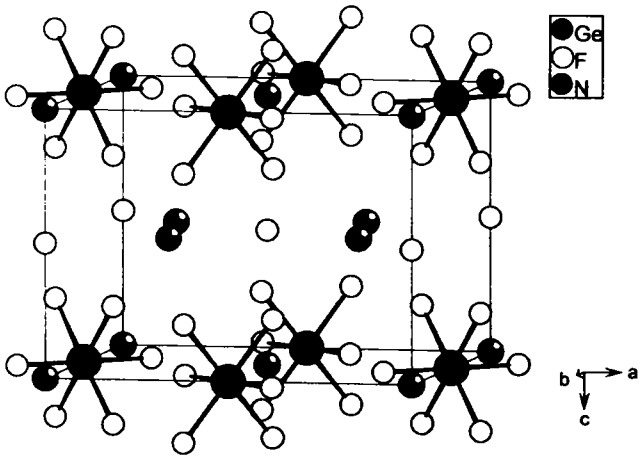

$F_7GeH_{12}N_3$, tetragonal, *P4/mbm* (No. 127), a =8.210(1) Å, c =5.984(1) Å, V =403.3 Å^3, Z =2, $R(F)$ =0.055, $R_w(F^2)$ =0.140.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless, irregular, size 0.1 x 0.1 x 0.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	38.68 cm^{-1}
Diffractometer:	Stoe IPDS
Scan mode:	50 exposures, $\Delta\varphi = 3°$
$T_{measurement}$:	293 K
$2\theta_{max}$:	54.94°
$N(hkl)_{unique}$:	275
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	23
Programs:	SHELXS-86, SHELXL-93

Source of material: Colorless single crystals of $(NH_4)_3GeF_7$ were obtained during the reaction of Ge powder with $(NH_4)HF_2$ (molar ratio 1:4) in a sealed Monel ($Cu_{32}Ni_{68}$) ampoule at 573 K. At higher temperature (> 673 K), $(NH_4)GeF_6$ (see ref. 2) is the main product.

The crystal structure of triammonium heptafluorogermanate contains as the isotypic $(NH_4)_3SiF_7$ (see ref. 3) isolated $[GeF_6]$ octahedra [$d(Ge^{4+}–F^-)$ = 175.6 pm (F2); 175.8 pm (F3)] and lonesome F- ions (F1).

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ge	2*d*	0	1/2	0	0.0433(7)	U_{11}	0.0373(8)	–0.0031(6)	0	0
F(1)	2*b*	0	0	1/2	0.075(4)	U_{11}	0.079(7)	0	0	0
F(2)	4*g*	0.3488(8)	*x*+1/2	0	0.195(9)	U_{11}	0.113(8)	–0.13(1)	0	0
F(3)	8*k*	0.1008(6)	*x*+1/2	0.219(1)	0.151(5)	U_{11}	0.099(4)	–0.054(6)	–0.041(3)	U_{13}
N(1)	2*a*	0	0	0	0.10(1)	U_{11}	0.18(3)	0	0	0
N(2)	4*h*	0.293(1)	*x*+1/2	1/2	0.104(5)	U_{11}	0.050(5)	–0.044(7)	0	0

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