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Acta Cryst. (1993). C**49**, 1335–1336

Structure of Guanidinium Hexafluorogallate, $[C(NH_2)_3]_3GaF_6$

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(*Received* 26 *June* 1992; *accepted* 11 *January* 1993)

Abstract

The guanidinium cation is planar within experimental error and does not exhibit any symmetry. The two inequivalent GaF_6 anions have $\bar{3}$ symmetry. The cation is strongly hydrogen bonded to the GaF_6 anions utilizing all H atoms. The molecular packing can be described as a framework of GaF_6 ions (where Ga atoms occupy octahedral voids) hydrogen bonded to guanidinium cations lying approximately parallel to (100) planes.

Comment

This work is part of a series of X-ray diffraction and NMR studies of crystal structure and ionic motion in various guanidinium salts (Pająk, Grottel & Kozioł, 1982; Kozak, Grottel, Kozioł & Pająk, 1987; Grottel, Kozak, Kozioł & Pająk, 1989).

The title compound is isostructural with guanidinium hexafluoroaluminate (Grottel, Kozak, Małuszyńska & Pająk, 1992). No essential differences in crystal and molecular structures of the aluminium derivative [$Pa\bar{3}$, $a = 13.953$ (2) Å] and the gallium analogue have been observed, except for a longer Ga—F bond length of 1.901 (1) Å, compared to the length of the Al—F bond of 1.818 (1) Å.

Experimental

Crystal data

$[C(NH_2)_3]_3[GaF_6]$
$M_r = 363.95$
Cubic
$Pa\bar{3}$
$a = 14.073$ (1) Å
$V = 2787.1$ (1) Å^3
$Z = 8$
$D_x = 1.735$ Mg m^{-3}
$D_m = 1.74$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 15 reflections
$\theta = 22.3$–$26.5°$
$\mu = 3.44$ mm^{-1}
$T = 293$ K
Cube
0.2 × 0.2 × 0.1 mm
Transparent

Data collection

Syntex $P2_1$ diffractometer
$\theta/2\theta$ scans
2272 measured reflections
638 independent reflections
526 observed reflections
$[I \geq 1.96\sigma(I)]$
$R_{int} = 0.049$
$\theta_{max} = 58°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 17$
3 standard reflections
monitored every 100 reflections
intensity variation: ±2%

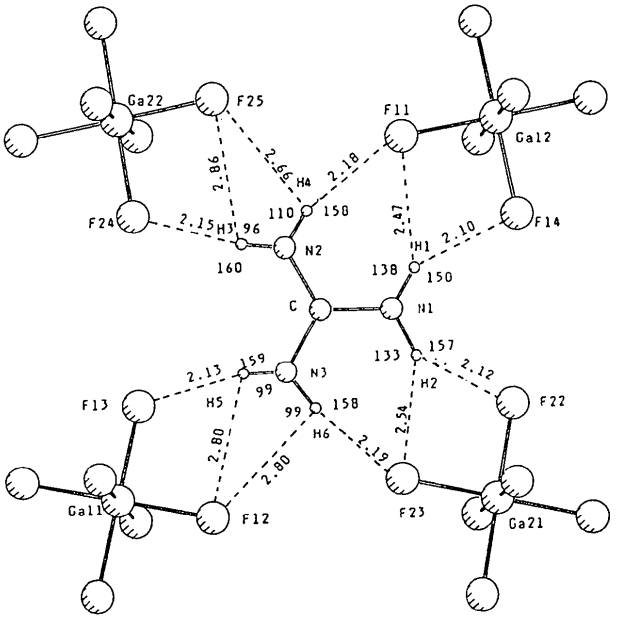

Fig. 1. The hydrogen-bond geometry (Å, °) of $[C(NH_2)_3]_3GaF_6$. σ(H···F) = 0.02 Å, σ(N—H···F) = 2°. Symmetry codes are: Ga11 $-\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$; Ga21 0, $\frac{1}{2}, \frac{1}{2}$; Ga12 0, 1, $\frac{1}{2}$; Ga22 $-\frac{1}{2}$, 1, $\frac{1}{2}$; F12 $y-1$, $-z+\frac{1}{2}$, $x+\frac{1}{2}$; F13 $x-\frac{1}{2}$, y, $-z+\frac{1}{2}$; F23 $-y+\frac{1}{2}$, $-z+1$, $x+\frac{1}{2}$; F22 none; F14 $1+x$, $\frac{3}{2}-y$, $\frac{1}{2}+z$; F24 $x-\frac{1}{2}$, $-y+\frac{3}{2}$, $-z+1$; F25 $y-1$, $-z+\frac{3}{2}$, $x+\frac{1}{2}$.

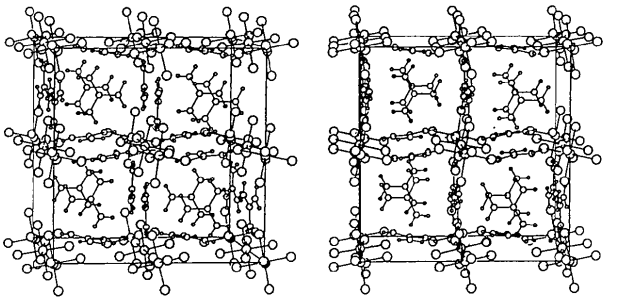

Fig. 2. Stereoscopic view of the until-cell contents.

Refinement

Refinement on F
Final $R = 0.034$
$wR = 0.045$
$S = 0.9$
525 reflections
83 parameters
All H-atom parameters refined
$w = 1/(\sigma_F^2 + 0.0045F^2)$
$(\Delta/\sigma)_{max} = 0.06$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.68$ e Å^{-3}
Extinction correction: $F_c^* = F_c(1-xF_c^2/\sin\theta)$
Extinction coefficient: $x = 3.1 \times 10^{-4}$
Atomic scattering factors from *SHELX*76

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2)

Ga(1) and Ga(2) each have site symmetry $\bar{3}$ and site-occupancy factor $\frac{1}{6}$; the respective values for all the other atoms are 1 and 1. H atoms were treated isotropically; for the other atoms $U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}$.

	x	y	z	U_{iso}/U_{eq}
Ga(1)	0	0.5000	0	0.146 (3)
Ga(2)	0	0.5000	0.5000	0.150 (4)
F(1)	0.0269 (1)	0.6302 (1)	0.0237 (1)	0.266 (5)
F(2)	0.0205 (1)	0.6309 (1)	0.4737 (1)	0.279 (5)
C	−0.2337 (2)	0.7514 (1)	0.5261 (3)	0.284 (10)
N(1)	−0.1410 (2)	0.7518 (2)	0.5235 (1)	0.410 (10)
N(2)	−0.2802 (2)	0.8312 (2)	0.5398 (2)	0.426 (10)
N(3)	−0.2794 (2)	0.6699 (2)	0.5166 (2)	0.417 (10)
H(1)	−0.106 (3)	0.803 (2)	0.528 (2)	0.487 (9)
H(2)	−0.104 (3)	0.704 (3)	0.515 (2)	0.541 (10)
H(3)	−0.333 (3)	0.827 (2)	0.537 (2)	0.310 (8)
H(4)	−0.251 (2)	0.880 (2)	0.543 (3)	0.398 (10)
H(5)	−0.333 (3)	0.672 (3)	0.513 (2)	0.497 (11)
H(6)	−0.246 (2)	0.630 (3)	0.503 (2)	0.426 (13)

Table 2. *Geometric parameters* (Å, °)

C—N(1)	1.305 (4)	N(3)—H(6)	0.75 (4)
C—N(2)	1.314 (3)	Ga(1)—F(1)	1.901 (1)
C—N(3)	1.322 (4)	Ga(2)—F(2)	1.901 (1)
N(1)—H(1)	0.87 (4)		
N(1)—H(2)	0.85 (4)	F(1)···F(1)	2.704 (2)
N(2)—H(3)	0.74 (4)	F(1)···F(1)	2.671 (2)
N(2)—H(4)	0.80 (3)	F(2)···F(2)	2.704 (2)
N(3)—H(5)	0.75 (5)	F(2)···F(2)	2.672 (2)
N(1)—C—N(2)	119.9 (2)	H(3)—N(2)—H(4)	126 (3)
N(1)—C—N(3)	119.2 (2)	C—N(3)—H(5)	118 (3)
N(2)—C—N(3)	120.9 (3)	C—N(3)—H(6)	111 (3)
C—N(1)—H(1)	124 (2)	H(5)—N(3)—H(6)	129 (2)
C—N(1)—H(2)	127 (3)	F(1)—Ga(1)—F(1)	90.7 (3)
H(1)—N(1)—H(2)	108 (4)	F(1)—Ga(1)—F(1)	180.0 (3)
C—N(2)—H(3)	115 (2)	F(2)—Ga(2)—F(2)	90.7 (3)
C—N(2)—H(4)	119 (2)	F(2)—Ga(2)—F(2)	180.0 (3)

Guanidinium hexafluorogallate was obtained by the following reaction (Szczepański, 1990):

$$3[C(NH_2)_3]_2CO_3 + Ga_2O_3 + 12HF \rightarrow 2[C(NH_2)_3]_3GaF_6 + 3CO_2 + 6H_2O.$$

The substrates were treated with an excess of hydrofluoric acid (40% aqueous solution) and heated to complete dissolution. Density was measured by flotation in CCl_4 and CH_3I. The background and integrated intensities were obtained by the peak-profile-analysis method of Lehman & Larsen (1974) using *PRARA* (Jaskólski, 1982). Corrections for Lorentz and polarization effects were applied. No absorption correction was made. The H-atom positions were calculated and refined isotropically. Programs used: *SHELX*76 (Sheldrick, 1976); *PLUTO* (Motherwell & Clegg, 1978); *PARST* (Nardelli, 1983); *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1986).

The author wishes to thank Professor Z. Pająk for suggesting this structure determination. This work was supported by the National Research Committee under grant No. 200729101.

Lists of structure factors, anisotropic thermal parameters and hydrogen-bond geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71010 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1015]

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Acta Cryst. (1993). C**49**, 1336–1339

Structure of Dipotassium *trans*-Diaquabis(oxalato-*O*,*O*′)nickelate(II)–Water (1/4)

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(*Received* 8 *September* 1992; *accepted* 25 *January* 1993)

Abstract

The crystal structure of the title compound was studied by single-crystal X-ray diffraction in order to determine the coordination geometry around the Ni^{II} atom in the anionic complex. The crystal structure comprises centrosymmetric $[Ni(C_2O_4)_2(H_2O)_2]^{2-}$