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Nickel Bistrifluorostannate(II) Hexahydrate, $[\text{Ni}(\text{H}_2\text{O})_6](\text{SnF}_3)_2$

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Abstract

The structure of the title compound has been solved by single crystal X-ray diffraction. Nickel is present as the hexaaquanickel cation, $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$, which is balanced in charge by discrete trigonal pyramidal $[\text{SnF}_3]^-$ complex anions in a 1:2 ratio. The non-bonding electron pairs on tin are stereochemically active.

Comment

The title compound is one of a series of complex tin(II) fluorides involving first-row transition metals of the general formula $M(\text{SnF}_3)_2 \cdot 6\text{H}_2\text{O}$ (Donaldson & Oteng, 1969). The crystal structure of the title compound represents the first of a nickel tin(II) fluoride.

The structure of the title compound contains the $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ cation which is balanced in charge by two $[\text{SnF}_3]^-$ complex anions. Unlike many complex tin(II) fluorides, the structure of the title compound contains isolated $[\text{SnF}_3]^-$ anions. This is in contrast to the network structures adopted by other complex stannous fluorides such as $\text{Cs}_2\text{Sn}_6\text{Br}_3\text{F}_{11}$ (Abrahams, Donaldson & Grimes, 1992), SnBrF_5 (Donaldson, Laughlin & Puxley, 1977; Vilminot, Granier & Cot, 1978) and Sn_4OF_6 (Abrahams, Clark, Donaldson, Khan & Southern, 1994), which show one-, two- and three-dimensional polymeric bridging structures, respectively. The non-bonding electron pairs on the Sn atoms, in common with most stannous fluorides,

show strong stereochemical activity with no evidence for direct Sn—Sn interaction.

The Sn—F bond lengths of 2.040 (3)–2.057 (6) Å are comparable with those of other systems that have isolated $[\text{SnF}_3]^-$ anions, e.g. 2.08 Å in NH_4SnF_3 (Bergerhoff & Namgung, 1978) and 2.053–2.096 Å in $\text{N}_2\text{H}_6(\text{SnF}_3)_2$ (Kaučič, Leban, Gašperšič-Škander, Ganjar & Rahten, 1988). The structure of the cobalt analogue has been determined (Maury & Gleizes, 1980) and is isostructural with the title structure, with similar Sn—F bond lengths of 2.049–2.052 Å.

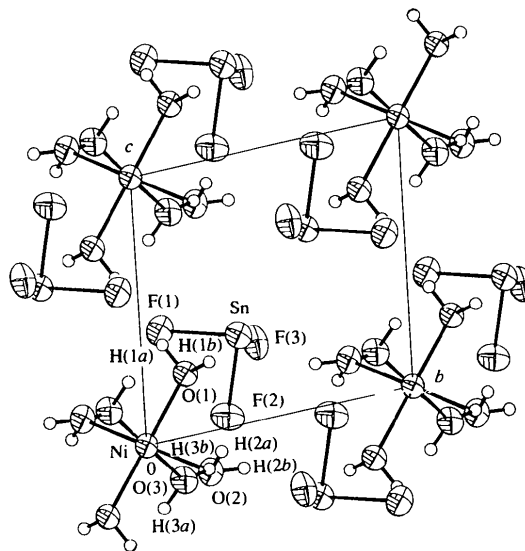


Fig. 1. Unit-cell projection of $[\text{Ni}(\text{H}_2\text{O})_6](\text{SnF}_3)_2$. Atomic displacement ellipsoids are drawn at the 50% level.

Experimental

$[\text{Ni}(\text{H}_2\text{O})_6](\text{SnF}_3)_2$ was prepared by dissolving appropriate molar quantities of NiF_2 and SnF_2 in a minimum amount of boiling water. After slow cooling, pale green triclinic prisms were formed which were collected and dried *in vacuo* over silica gel.

Crystal data

$[\text{Ni}(\text{H}_2\text{O})_6](\text{SnF}_3)_2$
 $M_r = 518.19$
 Triclinic
 $P\bar{1}$
 $a = 6.799$ (20) Å
 $b = 6.938$ (7) Å
 $c = 7.011$ (5) Å
 $\alpha = 77.51$ (7)°
 $\beta = 72.59$ (10)°
 $\gamma = 78.50$ (13)°
 $V = 304.9$ (10) Å³
 $Z = 1$
 $D_x = 2.822$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 14.142$ – 17.764 °
 $\mu = 5.675$ mm⁻¹
 $T = 291$ (2) K
 Triclinic prism
 $0.22 \times 0.20 \times 0.15$ mm
 Pale green

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer

$\omega/2\theta$ scans

Absorption correction: empirical

$T_{\min} = 0.926$, $T_{\max} = 0.999$

1547 measured reflections

1389 independent reflections

1283 observed reflections

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0101$

$\theta_{\text{max}} = 27.49^\circ$

$h = -8 \rightarrow 8$

$k = -8 \rightarrow 9$

$l = 0 \rightarrow 9$

2 standard reflections

frequency: 60 min

intensity decay: none

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0214$

$wR(F^2) = 0.0559$

$S = 1.133$

1389 reflections

95 parameters

H—O distances fixed at

0.95 (2) Å

$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2 + 0.1582P]$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.581 \text{ e } \text{Å}^{-3}$

$\Delta\rho_{\text{min}} = -0.642 \text{ e } \text{Å}^{-3}$

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.0396 (21)

Atomic scattering factors

from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Molecular graphics: *SNOOPI* (Davies, 1983). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BR1092). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Sn(1)	0.33287 (3)	0.36293 (3)	0.32999 (3)	0.03870 (12)
Ni(1)	0	0	0	0.03075 (14)
F(1)	0.2798 (4)	0.0733 (3)	0.4110 (3)	0.0513 (5)
F(2)	0.4214 (3)	0.3049 (4)	0.0397 (3)	0.0484 (5)
F(3)	0.0376 (3)	0.4248 (3)	0.2900 (4)	0.0493 (5)
O(1)	0.1695 (4)	-0.1571 (3)	-0.2308 (3)	0.0383 (5)
O(2)	0.1654 (4)	0.2311 (3)	-0.1493 (4)	0.0382 (5)
O(3)	0.2160 (4)	-0.1306 (4)	0.1574 (4)	0.0395 (5)

Table 2. Selected geometric parameters (Å, °)

Sn(1)—F(1)	2.040 (3)	Ni(1)—O(3)	2.050 (5)
Sn(1)—F(2)	2.047 (3)	Ni(1)—O(2)	2.054 (4)
Sn(1)—F(3)	2.057 (6)	Ni(1)—O(1)	2.069 (3)
F(1)—Sn(1)—F(2)	84.78 (12)	O(3)—Ni(1)—O(1 ¹)	91.3 (2)
F(1)—Sn(1)—F(3)	85.4 (2)	O(3 ¹)—Ni(1)—O(1 ¹)	88.7 (2)
F(2)—Sn(1)—F(3)	84.42 (14)	O(2)—Ni(1)—O(1 ¹)	91.98 (15)
O(3)—Ni(1)—O(2)	92.6 (2)	O(2 ¹)—Ni(1)—O(1 ¹)	88.02 (15)
O(3 ¹)—Ni(1)—O(2)	87.4 (2)		

Symmetry code: (i) $-x, -y, -z$.

Table 3. Contact distances (Å)

O(1)···F(1 ⁱ)	2.653 (4)	O(2)···F(3 ⁱⁱⁱ)	2.657 (5)
O(1)···F(3 ⁱⁱ)	2.708 (6)	O(3)···F(1)	2.674 (7)
O(2)···F(2)	2.660 (6)	O(3)···F(2 ^{iv})	2.635 (4)

Symmetry codes: (i) $x, y, z - 1$; (ii) $-x, -y, -z$; (iii) $-x, 1 - y, -z$; (iv) $1 - x, -y, -z$.

The structure was solved by Patterson methods using *SHELXS86* (Sheldrick, 1985) and refined using *SHELXL93* (Sheldrick, 1993). In the final refinement H—O distances were fixed at 0.95 (2) Å and anisotropic displacement parameters were refined for all non-H atoms.

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Strontium Diiron(III) Pyrophosphate

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Abstract

$\text{SrFe}_2^{\text{III}}(\text{P}_2\text{O}_7)_2$ [strontium diiron(III) bis(diphosphate)] was prepared using high-temperature fusion and was found to crystallize in the triclinic space group $P\bar{1}$.