

# catena-Poly[[trimethyltin(IV)]- $\mu$ -3,4-difluorobenzeneseleninato- $\kappa^2$ O:O']

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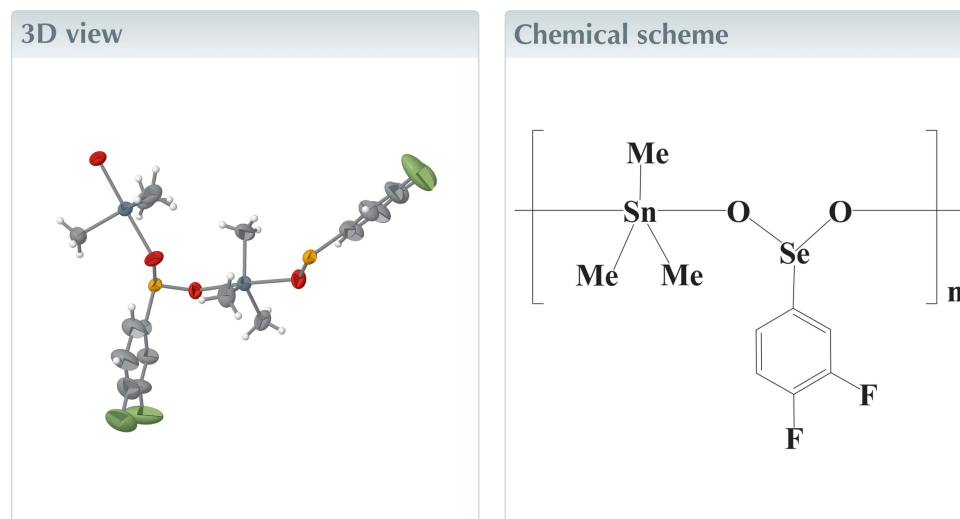
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Keywords: crystal structure; organotin complex; 3,4-difluorobenzeneseleninic acid; trimethyltin(IV); polymeric chain.

CCDC reference: 1578937

Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

The title compound,  $[\text{Sn}(\text{CH}_3)_3(\text{C}_6\text{H}_3\text{F}_2\text{O}_2\text{Se})]_n$ , was prepared by treatment of 3,4-difluorobenzeneseleninic acid and trimethyltin chloride with sodium ethoxide in methanol. In the polymeric crystal structure, infinite chains, with the  $\text{Sn}^{\text{IV}}$  atom in a trigonal-bipyramidal  $\text{C}_3\text{O}_2$  coordination environment involving methyl ligands and the bridging 3,4-difluorobenzeneseleninate anion, are present. The chains extend parallel to [010] and are linked through slipped  $\pi$ - $\pi$  interactions and weak  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds into a three-dimensional network.



## Structure description

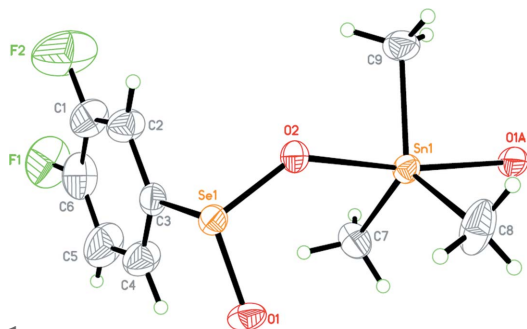
In recent years, organotin complexes have been attracting attention due to their significant number of industrial applications and their biological activities (Dubey & Roy, 2003; Gielen, 2002). As part of our ongoing investigations in this field (Ma *et al.*, 2011), we have synthesized the title compound and present its crystal structure here.

As can be seen from Fig. 1, the asymmetric unit of the title compound consists of one  $[(\text{CH}_3)_3\text{Sn}]$  moiety and a deprotonated 3,4-difluorobenzeneseleninate anion that bridges adjacent  $\text{Sn}^{\text{IV}}$  atoms. The geometric index  $\tau_5$  (Addison *et al.*, 1984) of Sn1 is 0.83, indicating a distorted trigonal-bipyramidal coordination environment whereby two O atoms from two 3,4-difluorobenzeneseleninate anions occupy the axial positions [ $\text{O}2-\text{Sn}1-\text{O}1A(-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}) = 171.53(11)^\circ$ ] and the methyl C atoms occupy the equatorial sites ( $\text{C}-\text{Sn}1-\text{C}$  angle sum is  $359.9^\circ$ ). The OSeO units of the 3,4-difluorobenzeneseleninate anion link adjacent  $[\text{Me}_3\text{Sn}]^+$  moieties into a zigzag chain structure extending parallel to [010] (Fig. 2). As a result of weak  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bonding interactions (Table 1) and slipped  $\pi$ - $\pi$  interactions between the difluorobenzene rings of adjacent chains (plane-to-plane distance = 3.538 Å), a three-dimensional network is established (Fig. 3).

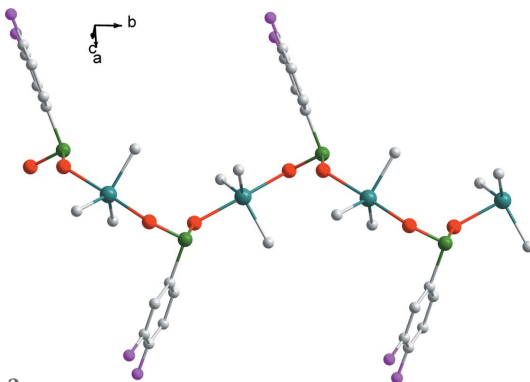
**Table 1**  
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2\cdots O2^i$	0.93	2.59	3.370 (7)	142

Symmetry code: (i)  $-x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$ .



**Figure 1**  
The coordination environment of the  $\text{Sn}^{\text{IV}}$  atom, showing displacement ellipsoids at the 30% probability level. [Symmetry code: (A)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$ .]



**Figure 2**  
View of the polymeric chain structure in the title compound running parallel to [010]. H atoms have been omitted for clarity.

### Synthesis and crystallization

All reactions were carried out under a nitrogen atmosphere using standard Schlenk techniques. The title compound was synthesized by dissolving 3,4-difluorobenzeneseleninic acid (0.225 g, 1.0 mmol) and sodium ethoxide (0.068 g, 1.0 mmol) in methanol (30 ml) under stirring for 30 min. Trimethyltin chloride (0.199 g, 1.0 mmol) was then added and the mixture stirred for a further 12 h at 323 K. The reaction mixture was filtered and the solvent gradually evaporated under vacuum until a colourless solid was obtained. The resulting product was recrystallized from diethyl ether to give transparent colourless crystals of the title compound (yield 80%, m.p. 413–415 K). Analysis calculated for  $\text{C}_9\text{H}_{12}\text{O}_2\text{F}_2\text{SeSn}$ : C 27.87, H 3.12%; found: C 27.67, H 3.38%.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

**Table 2**  
Experimental details.

Crystal data	$[\text{Sn}(\text{CH}_3)_3(\text{C}_6\text{H}_3\text{F}_2\text{O}_2\text{Se})]$
Chemical formula	387.84
$M_r$	Monoclinic, $C2/c$
Crystal system, space group	298
Temperature (K)	18.0109 (15), 10.5246 (8), 14.0791 (11)
$a, b, c$ (Å)	$\beta$ (°) 95.443 (2)
	$V$ (Å <sup>3</sup> ) 2656.8 (4)
	$Z$ 8
	Radiation type Mo $K\alpha$
	$\mu$ (mm <sup>-1</sup> ) 4.67
	Crystal size (mm) 0.45 × 0.25 × 0.18
Data collection	
Diffractometer	Bruker APEXIII CCD area detector
Absorption correction	Multi-scan (SADABS; Bruker, 2016)
$T_{\text{min}}, T_{\text{max}}$	0.228, 0.487
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	7837, 3199, 2336
$R_{\text{int}}$	0.048
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.665
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.033, 0.093, 1.04
No. of reflections	3199
No. of parameters	139
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.98, -0.82

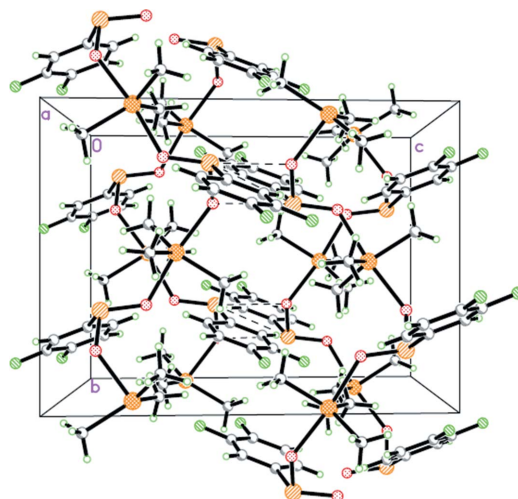
Computer programs: APEX3 (Bruker, 2016), SAINT (Bruker, 2016), SHELXS97 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), Mercury (Macrae *et al.*, 2008) and publCIF (Westrip, 2010).

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### References

Addison, A. W., Rao, T. N., Reedijk, J., van Rijn, J. & Verschoor, G. C. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356.



**Figure 3**  
A perspective view along [100], showing the crystal packing of the title compound.

- Bruker (2016). *APEX3*, *SAINTE* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dubey, S. K. & Roy, U. (2003). *Appl. Organomet. Chem.* **17**, 3–8.
- Gielen, M. (2002). *Appl. Organomet. Chem.* **16**, 481–494.
- Ma, C. L., Guo, M. J., Ru, J., Zhang, R. F. & Wang, Q. F. (2011). *Inorg. Chim. Acta*, **378**, 213–217.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## full crystallographic data

*IUCrData* (2017). 2, x171457 [https://doi.org/10.1107/S2414314617014572]

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catena-Poly[[trimethyltin(IV)]- $\mu$ -3,4-difluorobenzeneseleninato- $\kappa^2$ O:O']*Crystal data*

[Sn(CH<sub>3</sub>)<sub>3</sub>(C<sub>6</sub>H<sub>3</sub>F<sub>2</sub>O<sub>2</sub>Se)]

$M_r = 387.84$

Monoclinic,  $C2/c$

$a = 18.0109$  (15) Å

$b = 10.5246$  (8) Å

$c = 14.0791$  (11) Å

$\beta = 95.443$  (2)°

$V = 2656.8$  (4) Å<sup>3</sup>

$Z = 8$

$F(000) = 1472$

$D_x = 1.939$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 3059 reflections

$\theta = 2.6$ – $27.9$ °

$\mu = 4.67$  mm<sup>-1</sup>

$T = 298$  K

Block, colorless

$0.45 \times 0.25 \times 0.18$  mm

*Data collection*

Bruker APEXIII CCD area detector  
diffractometer

Radiation source: fine-focus sealed tube  
phi and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2016)

$T_{\min} = 0.228$ ,  $T_{\max} = 0.487$

7837 measured reflections

3199 independent reflections

2336 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 28.2$ °,  $\theta_{\min} = 2.6$ °

$h = -23 \rightarrow 23$

$k = -13 \rightarrow 10$

$l = -18 \rightarrow 17$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.093$

$S = 1.04$

3199 reflections

139 parameters

0 restraints

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 2.010P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.017$

$\Delta\rho_{\max} = 0.98$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.82$  e Å<sup>-3</sup>

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.27668 (2)	0.51508 (3)	0.69692 (2)	0.03983 (12)
Se1	0.19978 (3)	0.81568 (4)	0.60742 (3)	0.03991 (14)
F1	-0.1054 (2)	0.6237 (5)	0.4668 (4)	0.1246 (16)
F2	0.0018 (3)	0.6159 (7)	0.3566 (4)	0.171 (3)
O1	0.1875 (2)	0.8348 (3)	0.7241 (2)	0.0519 (8)
O2	0.25046 (18)	0.6830 (3)	0.6011 (2)	0.0447 (8)
C1	0.0180 (4)	0.6642 (7)	0.4431 (5)	0.082 (2)
C2	0.0887 (3)	0.7068 (6)	0.4718 (4)	0.0660 (15)
H2	0.1265	0.7041	0.4313	0.079*
C3	0.1010 (3)	0.7533 (5)	0.5628 (3)	0.0479 (11)
C4	0.0444 (3)	0.7563 (7)	0.6227 (4)	0.0735 (17)
H4	0.0544	0.7872	0.6845	0.088*
C5	-0.0279 (4)	0.7138 (8)	0.5922 (6)	0.105 (3)
H5	-0.0665	0.7163	0.6315	0.126*
C6	-0.0376 (3)	0.6687 (7)	0.5012 (6)	0.085 (2)
C7	0.1736 (3)	0.5143 (5)	0.7559 (4)	0.0643 (16)
H7A	0.1438	0.5842	0.7304	0.096*
H7B	0.1820	0.5224	0.8240	0.096*
H7C	0.1481	0.4359	0.7401	0.096*
C8	0.3657 (4)	0.6155 (6)	0.7734 (5)	0.088 (2)
H8A	0.3673	0.5947	0.8399	0.132*
H8B	0.3579	0.7052	0.7652	0.132*
H8C	0.4120	0.5920	0.7496	0.132*
C9	0.2920 (3)	0.4116 (5)	0.5720 (4)	0.0658 (15)
H9A	0.2847	0.3228	0.5834	0.099*
H9B	0.3416	0.4253	0.5547	0.099*
H9C	0.2565	0.4397	0.5211	0.099*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.0463 (2)	0.03474 (18)	0.03778 (19)	0.00020 (13)	0.00044 (13)	0.00274 (13)
Se1	0.0516 (3)	0.0288 (2)	0.0381 (3)	-0.00090 (18)	-0.0022 (2)	0.00034 (18)
F1	0.053 (2)	0.167 (4)	0.147 (4)	-0.030 (3)	-0.023 (2)	0.000 (4)
F2	0.113 (4)	0.271 (8)	0.125 (4)	-0.044 (4)	-0.014 (3)	-0.087 (5)
O1	0.074 (2)	0.0423 (18)	0.0393 (18)	-0.0108 (16)	0.0059 (16)	-0.0103 (15)
O2	0.0548 (19)	0.0376 (17)	0.0414 (18)	0.0073 (14)	0.0029 (15)	0.0071 (14)
C1	0.075 (4)	0.092 (5)	0.072 (4)	-0.010 (4)	-0.027 (4)	-0.021 (4)
C2	0.057 (3)	0.082 (4)	0.057 (3)	-0.006 (3)	-0.001 (3)	-0.014 (3)
C3	0.049 (3)	0.042 (3)	0.050 (3)	0.005 (2)	-0.009 (2)	0.000 (2)
C4	0.057 (3)	0.105 (5)	0.058 (4)	0.000 (4)	0.004 (3)	-0.005 (4)
C5	0.077 (5)	0.138 (7)	0.093 (6)	-0.006 (5)	-0.023 (4)	-0.002 (6)
C6	0.042 (4)	0.097 (5)	0.115 (6)	-0.013 (3)	-0.009 (4)	0.014 (5)
C7	0.064 (4)	0.060 (3)	0.072 (4)	0.009 (3)	0.022 (3)	0.015 (3)
C8	0.094 (5)	0.060 (4)	0.099 (5)	-0.020 (3)	-0.046 (4)	0.015 (4)

C9	0.099 (5)	0.049 (3)	0.050 (3)	0.014 (3)	0.013 (3)	-0.002 (3)
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*Geometric parameters (Å, °)*

Sn1—C7	2.104 (5)	C3—C4	1.383 (7)
Sn1—C9	2.108 (5)	C4—C5	1.404 (9)
Sn1—C8	2.125 (6)	C4—H4	0.9300
Sn1—O2	2.247 (3)	C5—C6	1.363 (10)
Sn1—O1 <sup>i</sup>	2.262 (3)	C5—H5	0.9300
Se1—O2	1.675 (3)	C7—H7A	0.9600
Se1—O1	1.690 (3)	C7—H7B	0.9600
Se1—C3	1.944 (5)	C7—H7C	0.9600
F1—C6	1.355 (7)	C8—H8A	0.9600
F2—C1	1.326 (7)	C8—H8B	0.9600
O1—Sn1 <sup>ii</sup>	2.262 (3)	C8—H8C	0.9600
C1—C6	1.352 (10)	C9—H9A	0.9600
C1—C2	1.374 (8)	C9—H9B	0.9600
C2—C3	1.370 (7)	C9—H9C	0.9600
C2—H2	0.9300		
C7—Sn1—C9	121.1 (2)	C5—C4—H4	119.3
C7—Sn1—C8	116.9 (3)	C6—C5—C4	115.6 (7)
C9—Sn1—C8	121.8 (3)	C6—C5—H5	122.2
C7—Sn1—O2	95.57 (16)	C4—C5—H5	122.2
C9—Sn1—O2	86.66 (16)	C1—C6—F1	117.8 (7)
C8—Sn1—O2	91.06 (19)	C1—C6—C5	123.0 (6)
C7—Sn1—O1 <sup>i</sup>	91.36 (17)	F1—C6—C5	119.3 (7)
C9—Sn1—O1 <sup>i</sup>	85.58 (16)	Sn1—C7—H7A	109.5
C8—Sn1—O1 <sup>i</sup>	90.13 (19)	Sn1—C7—H7B	109.5
O2—Sn1—O1 <sup>i</sup>	171.53 (11)	H7A—C7—H7B	109.5
O2—Se1—O1	105.85 (16)	Sn1—C7—H7C	109.5
O2—Se1—C3	100.84 (18)	H7A—C7—H7C	109.5
O1—Se1—C3	98.87 (19)	H7B—C7—H7C	109.5
Se1—O1—Sn1 <sup>ii</sup>	121.34 (16)	Sn1—C8—H8A	109.5
Se1—O2—Sn1	135.08 (17)	Sn1—C8—H8B	109.5
F2—C1—C6	117.2 (6)	H8A—C8—H8B	109.5
F2—C1—C2	120.8 (7)	Sn1—C8—H8C	109.5
C6—C1—C2	122.0 (6)	H8A—C8—H8C	109.5
C3—C2—C1	117.1 (6)	H8B—C8—H8C	109.5
C3—C2—H2	121.5	Sn1—C9—H9A	109.5
C1—C2—H2	121.5	Sn1—C9—H9B	109.5
C2—C3—C4	121.0 (5)	H9A—C9—H9B	109.5
C2—C3—Se1	119.0 (4)	Sn1—C9—H9C	109.5
C4—C3—Se1	120.0 (4)	H9A—C9—H9C	109.5
C3—C4—C5	121.4 (6)	H9B—C9—H9C	109.5
C3—C4—H4	119.3		
O2—Se1—O1—Sn1 <sup>ii</sup>	123.4 (2)	Se1—C3—C4—C5	179.0 (6)

C3—Se1—O1—Sn1 <sup>ii</sup>	-132.6 (2)	C3—C4—C5—C6	0.8 (11)
O1—Se1—O2—Sn1	18.4 (3)	F2—C1—C6—F1	0.0 (11)
C3—Se1—O2—Sn1	-84.1 (3)	C2—C1—C6—F1	-179.6 (7)
F2—C1—C2—C3	-179.0 (7)	F2—C1—C6—C5	178.7 (8)
C6—C1—C2—C3	0.6 (10)	C2—C1—C6—C5	-0.9 (12)
C1—C2—C3—C4	0.3 (9)	C4—C5—C6—C1	0.2 (12)
C1—C2—C3—Se1	-179.7 (5)	C4—C5—C6—F1	178.8 (7)
C2—C3—C4—C5	-1.0 (10)		

Symmetry codes: (i)  $-x+1/2, y-1/2, -z+3/2$ ; (ii)  $-x+1/2, y+1/2, -z+3/2$ .

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C2—H2 $\cdots$ O2 <sup>iii</sup>	0.93	2.59	3.370 (7)	142

Symmetry code: (iii)  $-x+1/2, -y+3/2, -z+1$ .