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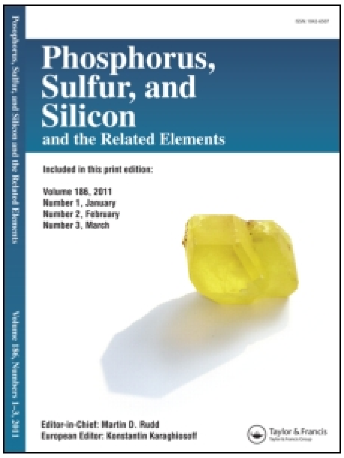

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Synthesis and Characterization of Tris[(4-fluorophenyldimethylsilyl)-methyl]tin O,O-dialkyldithiophosphates

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SYNTHESIS AND CHARACTERIZATION OF TRIS[(4-FLUOROPHENYLDIMETHYLSILYL)-METHYL]TIN O,O-DIALKYLDITHIOPHOSPHATES

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Fifteen new tris[(4-fluorophenyldimethylsilyl)methyl]tin O,O-dialkyldithiophosphates were synthesized and characterized by IR, ^1H NMR, MS spectroscopy, x-ray diffraction, and elemental analysis. The crystal structure of $[(4\text{-}FC_6H_4SiMe_2CH_2)_3Sn]_2O$ *has been determined. The structure consists of four-coordinated tin atoms in a slightly distorted tetrahedral geometry with an O atom bridge between the two Sn atoms.*

Keywords: Crystal structure; ^1H NMR; infrared spectra; mass spectra; synthesis; tris[(4-fluorophenyldimethylsilyl)methyl]tin

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Torque is a new efficient acaricide manufactured by the Shell company of America.[1] It consists of $[(PhMe_2CCH_2)_3Sn]_2O$ molecules. When a Si atom substitutes one C atom of the molecule, it is called Sila-Torque—$[(PhMe_2SiCH_2)_3Sn]_2O$. It has similar acaricidal activity as Torque, which was reported by Tacker et al. in 1986.[2] But according to our result of bioassay the Sila-Torque is not a good acaricide. Generally, an F atom appears in many recently developed hyperefficient pesticides which show high biological activity, low toxity and low residual.[3] So in this article, we introduce an F atom into Sila-Torque and synthesized a series of new compounds of $(4\text{-}FC_6H_4SiMe_2CH_2)_3SnS(S)P(OR)_2$and their intermediate $[(4\text{-}FC_6H_4SiMe_2CH_2)_3Sn]_2O$. However, the result is disappointing. The introduction of an F atom into Sila-Torque does not increase the acaricidal activity.

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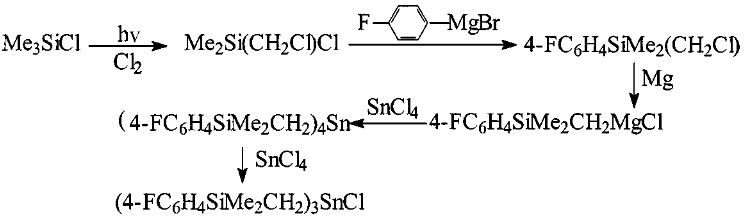
EXPERIMENTAL

Apparatus and Chemicals

IR spectra were recorded on a SHIMADZU-IR 453 spectrometer as liquid films; ^{1}H NMR spectra were measured on an AC-P200 with TMS as internal standard; mass spectra (MS) were recorded on an HP-5988 at 70 ev, and the temperature of ionization was 200°C; elemental analysis were determined on a MT-3 elementary analyzer (Yanaco, Japan).

Potassium O,O-dialkyldithiophosphate were synthesised according to Zemlyanskii and Drach[4] and Kabachmik.[5] (4-$FC_6H_4SiMe_2$ $CH_2)_3SnCl$ (m.p.: 60~62°C; $C_{27}H_{36}ClF_3Si_3Sn$ elemental analysis found (calcd.): C: 49.37% (49.43%), H: 5.29% (5.49%)) were prepared according to the route shown in Scheme 1.[6–9]



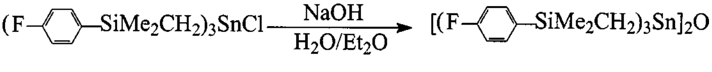
SCHEME 1

Preparation of Products

Preparation of [(4-$FC_6H_4SiMe_2CH_2)_3Sn]_2O$

m.p.: 69~70°C; $C_{54}H_{72}F_6OSi_6Sn_2$ elemental analysis found (calcd.): C: 51.56% (51.59%), H: 5.75% (5.73%).

[(4-$FC_6H_4SiMe_2CH_2)_3Sn]_2O$ was prepared according to the route shown in Scheme 2.



SCHEME 2

5 g (7.63 mmol) $(FC_6H_4SiMe_2CH_2)_3SnCl$ was dissolved in 40 ml ether, then 10 ml 10% sodium hydroxide aqueous solution was added into it. The resultant solution was continuously stirred with hydroxide aqueous solution into the flask, and refluxed the solution for 2 h once again. The ether layer was seperated and dried with anhydrous sodium sulfate.

After the desiccant was filtrated off, the crude product obtained by evaporation of ether was pure enough for analytical purpose. Yield: 83.4%.

Preparation of (4-$FC_6H_4SiMe_2CH_2)_3SnS(S)P(OR)_2$

The products were prepared according to the route shown in Scheme 3.

$$(4\text{-}FC_6H_4SiMe_2CH_2)_3SnCl + (RO)_2\overset{S}{\overset{\|}{P}}SK \longrightarrow (4\text{-}FC_6H_4SiMe_2CH_2)_3SnS\overset{S}{\overset{\|}{P}}(OR)_2$$

R = Me(1), Et(2), Pr(3), i-Pr(4), n-Bu(5), n-C_5H_{11}(6), i-C_5H_{11}(7), n-C_6H_{13}(8), n-C_7H_{15}(9), n-C_8H_{17}(10); C_6H_5(11), 4-ClC_6H_4(12), 4-MeC_6H_4(13), 3-MeC_6H_4(14), 4-t-BuC_6H_4(15)

SCHEME 3

A typical procedure was carried out as follows: 8.56 g (11 mmol) potassium O,O-dimethyldithiophosphate was added to the solution of 6.56 g (10 mmol) $(FC_6H_4SiMe_2CH_2)_3SnCl$ in 40 ml acetone. The reaction mixture was continuously stirred and refluxed for 6 h. At the end of the reaction, after filtrating, acetone was evaporated off under reduced pressure and a small volume of petroleum ether was added to the residue. Solid precipitates, most of which should be excessive potassium O,O-dimethyldithiophosphate and potassium chloride, were filtrated off. After the solvent was evaporated off, the residue was washed with petroleum ether once again. Then the pure product was obtained after evaporating off petroleum ether completely. Yield: 80.0%.

The physical constants, yields, and elemental analysis of these compounds are listed in Table I.

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RESULTS AND DISCUSSION

IR Data

The characteristic absorptions of bonds Si—CH_3 of all compounds are observed at 1225~1229 cm^{-1}, strong and sharp. The IR stretching vibration frequencies of Si—C are in the range 816~820 cm^{-1}. The asymmetric absorption vibration frequencies (ν_{asym}) of bond Sn—C are in the range 510~524 cm^{-1}, and the symmetric absorption vibration frequencies (ν_{sym}) are in the range 460~477 cm^{-1}. ν_{PS2} are located in

TABLE I Physical Constants, Yields, and Elemental Analysis of the Compounds **1–15**

No.	Formula	m.w.	Physical state	Yield (%)	Elemental analysis found (calcd.) C (%)	Elemental analysis found (calcd.) H (%)
1	$C_{29}H_{42}F_3O_2PS_2Si_3Sn$	777	colorless liquid	80.2	45.00(44.76)	5.15(5.40)
2	$C_{31}H_{46}F_3O_2PS_2Si_3Sn$	805	yellow liquid	72.3	46.16(46.21)	5.36(5.71)
3	$C_{33}H_{50}F_3O_2PS_2Si_3Sn$	833	yellow liquid	74.5	47.40(47.54)	5.89(6.00)
4	$C_{33}H_{50}F_3O_2PS_2Si_3Sn$	833	yellow liquid	80.9	47.64(47.54)	5.90(6.00)
5	$C_{35}H_{54}F_3O_2PS_2Si_3Sn$	861	yellow liquid	78.0	48.18(48.78)	6.27(6.27)
6	$C_{37}H_{58}F_3O_2PS_2Si_3Sn$	889	yellow liquid	77.5	49.80(49.94)	6.45(6.52)
7	$C_{37}H_{58}F_3O_2PS_2Si_3Sn$	889	yellow liquid	81.0	49.96(49.94)	6.39(6.52)
8	$C_{39}H_{62}F_3O_2PS_2Si_3Sn$	917	yellow liquid	79.1	51.10(51.04)	6.84(6.76)
9	$C_{41}H_{66}F_3O_2PS_2Si_3Sn$	945	yellow liquid	74.3	51.91(52.06)	7.10(6.98)
10	$C_{43}H_{70}F_3O_2PS_2Si_3Sn$	973	yellow liquid	74.0	52.97(53.03)	7.15(7.19)
11	$C_{39}H_{48}F_3O_2PS_2Si_3Sn$	901	colorless liquid	83.5	51.84(51.94)	5.14(5.11)
12	$C_{39}H_{44}Cl_2F_3O_2PS_2Si_3Sn$	970	yellow liquid	71.2	48.25(48.25)	4.55(4.54)
13	$C_{41}H_{50}F_3O_2PS_2Si_3Sn$	929	colorless liquid	80.4	52.86(52.96)	6.12(5.38)
14	$C_{41}H_{50}F_3O_2PS_2Si_3Sn$	929	colorless liquid	71.7	53.44(52.96)	6.16(5.38)
15	$C_{47}H_{62}F_3O_2PS_2Si_3Sn$	1013	colorless liquid	73.3	55.44(55.68)	5.96(6.12)

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the range 648∼675 cm^{-1} for asymmetric absorption and in the range 593∼608 cm^{-1} for symmetric absorption.

^{1}H NMR Data

The ^{1}H NMR spectra for the intermediate (4-$FC_6H_4SiMe_2CH_2)_3SnCl$ displayed two well-separated sharp single resonances at 0.28 ppm and 0.16 ppm, which were attributed to the H atom in the Si—CH_3 and Si—CH_2 groups respectively. However, all the products showed signals of the H atoms in the Si—CH_2 group at lower fields compared with the Si—CH_3 group, ranging from 0.27–0.34 ppm. We attribute these phenomena to the inductive effect of $S(S)P(OR)_2$ groups. The H atom in 4-FC_6H_4Si group exhibited two asymmetric triplets. This is attributed to the coupling between H—H and F—H on adjacent C atoms. In compound **1**, the signal of the H atom in the $S(S)P(OCH_3)_2$ group was found to be a double peak and $^3J_{P\text{—}H}$ was 15.63 Hz. Similarly, H atoms in the OCH_2 groups of other compounds exhibited multiplets due to the coupling between H and P atoms. In the diaryldithiophosphate compounds the protons' signals of FC_6H_4 and OAr overlapped as multiplets.

TABLE II ^{1}H NMR Data of the Compounds **1–15**

1	0.25 (s, 18H, $SiMe_2$), 0.27 (s, 6H, $SiCH_2$), 3.66, 3.74 (d, 6H, OCH_3, $^3J_{P-H} = 15.63$ Hz), 7.01∼7.37 (m, 12H, Ar—H)
2	0.27 (s, 18H, $SiMe_2$), 0.29(s, 6H, $SiCH_2$), 1.31(t, 6H, CH_3, $^3J_{H-H} = 7.42$ Hz), 4.07 (m, 4H, OCH_2)
3	0.26 (s, 18H, $SiMe_2$), 0.31 (s, 6H, $SiCH_2$), 0.94 (t, 6H, CH_3, $^3J_{H-H} = 7.32$ Hz), 1.67 (m, 4H, CH_2), 3.96 (m, 4H, OCH_2), 6.96∼7.40 (m, 12H, Ar—H)
4	0.28 (s, 18H, $SiMe_2$), 0.34 (s, 6H, $SiCH_2$), 1.31 (d, 12H, CH_3, $^3J_{H-H} = 6.22$ Hz), 4.75 (m, 2H, OCH)
5	0.25 (s, 18H, $SiMe_2$), 0.30 (s, 6H, $SiCH_2$), 0.90 (t, 6H, CH_3, $^3J_{H-H} = 7.22$ Hz), 1.35∼1.65 (m, 8H, CH_2), 4.02 (m, 4H, OCH_2), 7.00, 7.39 (m, 12H, Ar—H)
6	0.27 (s, 18H, $SiMe_2$), 0.31 (s, 6H, $SiCH_2$), 0.88 (t, 6H, CH_3), 1.32∼1.65 (m, 12H, CH_2), 4.00 (m, 4H, OCH_2), 7.05∼7.36 (m, 12H, Ar—H)
7	0.27 (s, 18H, $SiMe_2$), 0.31 (s, 6H, $SiCH_2$), 0.91 (d, 12H, CH_3, $^3J_{H-H} = 6.37$ Hz), 1.56 (m, 6H, CH + CH_2), 4.05 (m, 4H, OCH_2), 6.96∼7.40 (m, 12H, Ar—H)
8	0.26 (s, 18H, $SiMe_2$), 0.31 (s, 6H, $SiCH_2$), 0.88 (t, 6H, CH_3), 1.29∼1.65 (m, 16H, CH_2), 4.00 (m, 4H, OCH_2), 7.00∼7.36(m, 12H, Ar—H)
9	0.27 (s, 18H, $SiMe_2$), 0.30 (s, 6H, $SiCH_2$), 0.86 (t, 6H, CH_3), 1.26∼1.65 (m, 20H, CH_2), 4.01 (m, 4H, OCH_2), 6.96∼7.40 (m, 12H, Ar—H)
10	0.26 (s, 18H, $SiMe_2$), 0.34 (s, 6H, $SiCH_2$), 0.88 (t, 6H, CH_3), 1.25∼1.65 (m, 24H, CH_2), 4.02 (m, 4H, OCH_2), 6.96∼7.40 (m, 12H, Ar—H)
11	0.28 (s, 18H, $SiMe_2$), 0.29 (s, 6H, $SiCH_2$), 7.01∼7.35 (m, 22H, Ar—H)
12	0.19 (s, 18H, $SiMe_2$), 0.27 (s, 6H, $SiCH_2$), 7.00∼7.32 (m, 20H, Ar—H)
13	0.22 (s, 18H, $SiMe_2$), 0.28 (s, 6H, $SiCH_2$), 2.31 (s, 6H, $ArCH_3$), 6.99∼7.24 (m, 20H, Ar—H)
14	0.23 (s, 18H, $SiMe_2$), 0.27 (s, 6H, $SiCH_2$), 2.32 (s, 6H, $ArCH_3$), 6.98∼7.39 (m, 20H, Ar—H)
15	0.23 (s, 18H, $SiMe_2$), 0.28 (s, 6H, $SiCH_2$), 1.29 (s, 18H, CH_3), 6.98∼7.39 (m, 20H, Ar—H)

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M.S. Data

Compounds **1** and **11** were selected for an M.S. study. The molecular ion was not observed, but the fragment ions are in agreement with the expected structure of the compounds. The fragment ions whose weights are more than 200, all contain tin atoms and are generally cluster peaks. The ion containing silicon 4-$FC_6H_4Si^+Me_2$ (153) for compounds **1** and **11** are the base peaks. Their mass spectral data are shown in Table III. In the chemical bond breaking process there is a hydrogen transfer rearrangement, which is accomplished through a six member transition state shown in Scheme 4, and there are also methyl and phenyl transfer rearrangements which are accomplished through a four member transition state as shown in Scheme 5.

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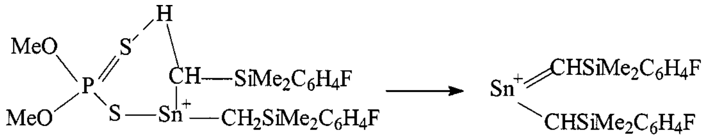
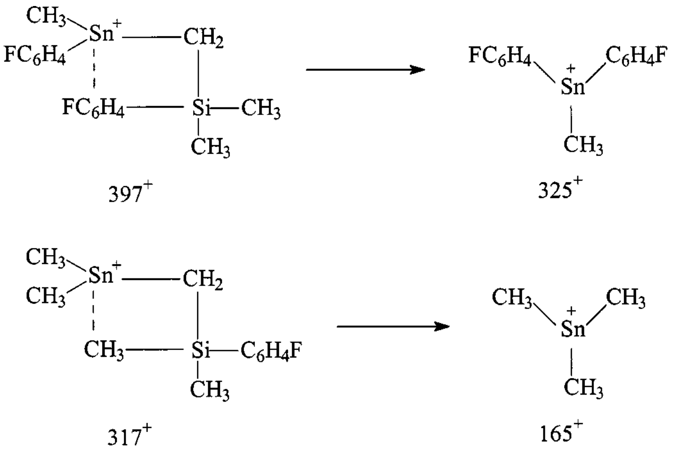
TABLE III The Mass Spectra Data of Compounds **1** and **11**

	m/e (abundance)			m/e (abundance)	
Fragment ions	1	11	Fragment ions	1	11
$M^{+\cdot}$	788(0)	902(0)	$FC_6H_4Sn^+$	215(18)	215(22)
R_3Sn^+	621(8)	621(0)	$FC_6H_4Me_2SiCH_2^+$	167(11)	167(7)
$(M\text{-}R)^+$	611(42)	735(0)	Me_3Sn^+	165(19)	165(17)
$(M\text{-}R\text{-}16)^+$	595(0)	719(24)	$(M\text{-}R_3Sn)^+$	157(14)	281(2)
$(M\text{-}R\text{-}32)^+$	579(0)	703(6)	$FC_6H_4Si^+Me_2$	153(100)	153(100)
R_2Sn^+	454(2)	454(4)	$FC_6H_4Si^+HMe$	139(44)	139(43)
$RSn^+CHSiMe_2C_6H_4F$	453(4)	453(14)	$FC_6H_4SiCH_2^+$	137(15)	137(14)
$RSn^+MeC_6H_4F$	397(18)	397(7)	$MeSn^+$	135(12)	135(11)
$(FC_6H_4)_2Sn^+Me$	325(12)	325(9)	$FC_6H_4Si^+H_2$	125(51)	125(6)
RSn^+Me_2	317(17)	317(7)	$FC_6H_4Si^+$	123(13)	123(1)
RSn^+	287(14)	287(18)	Sn^+H	121(3)	121(3)
$(M\text{-}R_3)^+$	277(44)	401(2)	Sn^+	120(3)	120(3)
$FC_6H_4Sn^+Me_2$	245(42)	245(39)	$FC_6H_4CH_2^+$	109(11)	109(10)



611⁺ 453⁺

SCHEME 4



397⁺ 325⁺

317⁺ 165⁺

SCHEME 5

TABLE IV Crystallographic Data for Compound $[(FC_6H_4SiMe_2CH_2)_3Sn]_2O$

Formula	$C_{54}H_{72}F_6OSi_6Sn_2$
Fomular weight	1257.04
Crystal size	0.35 mm × 0.25 mm × 0.20 mm
Crystal system	Triclinic
Space group	P-1
Unit cell parameters	$a = 11.442(3)$ Å, $b = 15.982(5)$ Å, $c = 17.616(5)$ Å $\alpha = 87.804(6)^\circ$, $\beta = 89.720(5)^\circ$, $\gamma = 78.806(5)^\circ$
V/(Å^3)	3157.8(16)
Z	2
$D_c/(mg \cdot m^{-3})$	1.322
Abs. coefficient (mm^{-1})	0.956
F(000)	1284
Limiting indices	$-13 < h < 12$, $-19 < k < 15$, $-17 < l < 20$
Ref. collected	13176
Final R indices	R = 0.0395, wR = 0.0699
Goodness-of-fit	0.943
Data/Parameter	11085/622
Largest diff. peak and hole/(e · Å^{-3})	0.663, −0.418

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Crystallography

Colorless crystals of compounds $[(4\text{-}FC_6H_4SiMe_2CH_2)_3Sn]_2O$ were obtained by recrystallization from petroleum/dichloromethane. Intensity data for a crystal with approximate dimensions of 0.35 × 0.25 × 0.20 mm were measured at 298(2)K on a BRUKER SMART 1000 diffractometer fitted with graphite monochromatised **Mo*K*α** radiation, $\lambda = 0.071073$ Å. A total of 11085 independent reflections were collected in the range of $1.16^\circ < \theta < 25.03^\circ$ by using $\omega/2\theta$ scan technique. Crystallographic data and final refinement details are listed in Table IV. The structure was solved from the interpretation of the patterns on synthesis using direct methods, and refined by a full-matrix least-squares procedure on F^2. Nonhydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were located in calculated

TABLE V Selected Bond Lengths (Å)

Bond	Dist.	Bond	Dist.	Bond	Dist.
Sn(1)—O(1)	1.960(3)	Sn(2)—C(11)	2.140(4)	C(19)—C(14)	1.366(7)
Sn(1)—C(61)	2.136(4)	Sn(2)—C(21)	2.140(4)	C(14)—C(15)	1.371(7)
Sn(1)—C(51)	2.148(4)	Si(1)—C(13)	1.846(6)	C(15)—C(16)	1.393(9)
Sn(1)—C(41)	2.158(4)	Si(1)—C(14)	1.857(5)	C(16)—C(17)	1.340(11)
Sn(2)—O(1)	1.960(3)	Si(1)—C(11)	1.859(4)	C(17)—C(18)	1.321(10)
Sn(2)—C(31)	2.143(4)	Si(1)—C(12)	1.865(6)	C(17)—F(1)	1.373(8)

TABLE VI Selected Bond Angles (°)

Angle	(°)	Angle	(°)
O(1)—Sn(2)—C(31)	107.59(15)	C(14)—Si(1)—C(11)	109.9(2)
O(1)—Sn(2)—C(11)	105.11(15)	C(13)—Si(1)—C(12)	108.5(3)
C(31)—Sn(2)—C(11)	114.26(17)	C(14)—Si(1)—C(12)	107.6(3)
O(1)—Sn(2)—C(21)	107.00(14)	C(11)—Si(1)—C(12)	109.4(3)
C(31)—Sn(2)—C(21)	108.84(18)	Si(1)—C(11)—Sn(2)	119.1(2)
C(11)—Sn(2)—C(21)	113.57(18)	C(19)—C(14)—Si(1)	124.0(5)
Sn(2)—O(1)—Sn(1)	140.13(16)	C(15)—C(14)—Si(1)	120.6(5)
C(13)—Si(1)—C(14)	110.3(3)	C(18)—C(17)—F(1)	118.6(10)
C(13)—Si(1)—C(11)	111.2(2)	C(16)—C(17)—F(1)	117.3(10)

positions and isotropically refined. The reference number from the Cambridge Data Centre is CCDC 173123.

The selected bond lengths and angles are listed in Table V and Table VI respectively. The molecular structure of $[(4\text{-}FC_6H_4SiMe_2CH_2)_3Sn]_2O$ is shown in Figure 1. Both Sn atoms exist in a distorted tetrahedral geometry defined by three C atoms from methylenes of the three (4-fluorophenyldimethylsilyl)methyl groups and the O atom which bridges the two Sn atoms.The average C—Sn—C angle is 112.9°and the average C—Sn—O angle is 105.8°, indicating a relatively

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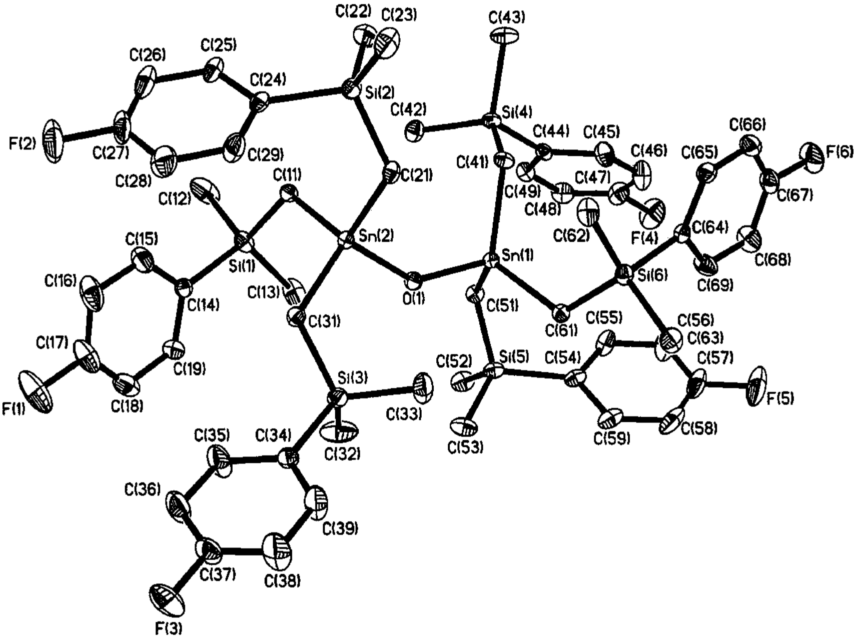

FIGURE 1 Molecular structure of $[(4\text{-}FC_6H_4SiMe_2CH_2)_3Sn]_2O$.

TABLE VII Acaricidal Activity of Some Compounds (Death Rate %)

Compds.	Conc. (ug/mL)		
	5	10	20
$[(4\text{-}FC_6H_4SiMe_2CH_2)_3Sn]_2O$	2.9	1.4	1.7
$[(PhSiMe_2CH_2)_3Sn]_2O$	0	0	0
$[(PhMe_2CCH_2)_3Sn]_2O$	85.2	96.9	100
$(4\text{-}FC_6H_4SiMe_2CH_2)_3SnS(S)P(OC_2H_5)_2$	0	0	0
$(PhMe_2SiCH_2)_3SnS(S)P(OC_2H_5)_2$	0	0	0
$(PhMe_2CCH_2)_3SnS(S)P(OC_2H_5)_2$	55.4	80.2	100

small distortion from the ideal tetrahedral angles because of repulsion between the three large (4-fluorophenyldimethylsilyl)methyl groups.

Acaricidal Activity

Unexpectedly, the introduction of an F atom into Sila-Torque does not increase the acaricidal activity. The acaricidal activity data of the lead-compounds and some of their analogs containing fluorine are listed in Table VII.

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