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BIMETALLIC ORGANOMETALLIC COMPOUNDS WITH TIN—LANTHANIDE BONDS

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Summary

New bimetallic organometallic compounds of lanthanides $[(Me_3SiCH_2)_3Sn]_3$-Ln · DME have been prepared by interaction of tris(trimethylsilylmethyl)tin hydride with $[(Me_3Si)_2N]_3Ln$ (Ln = Pr, Nd) in 1,2-dimethoxyethane (DME). Reactions of triphenylgermane and triethyltin hydride with tris[bis(trimethylsilyl)amido]-praseodymium and -neodymium was shown to be complicated by the cleavage of an ether bond of DME to afford compounds of the composition $(R_3M)_nLn(OCH_2CH_2OMe)_{3-n}$ · DME ($n = 1$ or 2; R = Et, M = Sn; R = Ph, M = Ge). The compounds obtained are weakly coloured solids, sensitive to oxygen and moisture. It has been shown that reactions of these compounds with hydrogen chloride, 1,2-dibromoethane, benzoyl peroxide, silver trifluoroacetate and other reagents proceed with cleavage of the tin—lanthanide bonds. The mechanism of the reactions being studied is discussed.

Introduction

In recent years there has been a growing interest in the organometallic chemistry of the *f*-elements. Cyclopentadienyl derivatives of the lanthanides and actinides are the most studied. Relatively few investigations of σ-bonded organolanthanides have been made [1—10]. Bimetallic organometallic compounds of the lanthanides, containing metal—lanthanide bonds, were reported only quite recently. The compounds with germanium— and tin—lanthanide covalent bonds were prepared by Schumann and coworkers according to the equation [11]

$$LnCl_3 + 3\ LiM(C_6H_5)_3 \xrightarrow{THF} Ln[M(C_6H_5)_3]_3 + 3\ LiCl$$

M = Ge; Sn; Ln = Pr, Nd, Gd, Er

However, the compounds were not isolated in a pure state, because of great experimental difficulties.

Compounds with phosphorus—lanthanide bonds are also known [12,13].

Earlier, we showed [14,15] that bis[tris(pentafluorophenyl)germyl]- and -stannyl]mercury react with metallic lanthanides in DME solution to form polymetallic organometallic complexes of the type $[(C_6F_5)_3M]_3Ln \cdot \{Hg[M(C_6F_5)_3]_2\}_n \cdot 3\,DME$, where M = Ge, Sn; Ln = Pr, Nd, Ho, Lu; $n = 1$ or 2. We did not manage to prepare non-fluorinated complexes by this method. We report here the synthesis and investigation of some chemical properties of bimetallic organometallic compounds with hydrocarbon radicals containing tin—praseodymium and tin—neodymium bonds.

Results and discussion

While studying the possible preparation routes of bimetallic organometallic compounds of the lanthanides we have found that reaction of tris(trimethylsilylmethyl)tin hydride with tris[bis(trimethylsilyl)amidopraseodynium and -neodymium in DME solution at 100°C leads to the formation of hexamethyldisilazane and organotin complexes of the lanthanides (eq. 1).

$$3(Me_3SiCH_2)_3SnH + [(Me_3Si)_2N]_3Ln \xrightarrow[100^\circ C,\ 15\ h]{DME} 3(Me_3Si)_2NH + [(Me_3SiCH_2)_3Sn]_3Ln \cdot DME \qquad (1)$$

Ln = Pr, Nd

Yields of the compounds isolated are rather high (60—75%). It has also been established that reactions of triphenylgermane and triethyltin hydride with above mentioned amidoderivatives of praseodymium and neodymium proceed more complicatedly. Together with the formation of the metal—lanthanide bond, cleavage of the 1,2-dimethoxyethane ether bond is observed. As a result the compounds of the composition $(R_3M)_nLn(OCH_2CH_2OMe)_{3-n} \cdot (DME)_x$ (with R = Et and M = Sn $n = 1, x = 0$; with R = Ph and M = Ge $n = 2, x = 1$) were isolated.

In connection with this it is necessary to note that at first we were mistaken to suppose [16] that in the reaction of triethyltin hydride with $[(Me_3Si)_2N]_3Ln$ the composition of complexes being formed is analogous to those which are formed according to eq. 1.

The abstraction of a methyl group from 1,2-dimethoxyethane by different organometallic compounds was reported earlier. In particular it has been shown [17] that Ph_3SiLi cleaves the Me—O bond in DME to form methyltriphenylsilane in an 84.5% yield. Recently it was noted [18] that the reaction of magnesium metal with $Hg(SiMe_2Ph)_2$ in DME is accompanied by cleavage of the solvent. As a result of this reaction the authors obtained the unusual compound of magnesium $[Mg_4(OCH_2CH_2OMe)_6 \cdot 2\,DME] \cdot [Hg(SiMe_2Ph)_3]_2$.

In the case of triphenyltin hydride we failed to prepare bimetallic organometallic compounds of the lanthanides. Only hexaphenyldistannane was isolated from the reaction mixture in high yield. The amidoderivatives of the lanthanides probably catalyze the decomposition of triphenyltin hydride. It was confirmed by reaction with catalytic amounts of $[(Me_3Si)_2N]_3Ln$ at room temperature.

All compounds obtained are weakly-coloured solids decomposing in the temperature range 230—290°C. They are sensitive to oxygen and moisture, almost insoluble both in polar and nonpolar solvents, and highly thermally stable. The complexes were identified by elemental analyses, infrared spectra and some chemical reactions given below. Unfortunately the insolubility of the complexes precluded molecular weight determinations and X-ray analysis. Therefore the given structures of the complexes are only supposed. The high decomposition points and insolubility of compounds may testify to their possible polymeric character.

The thermal decomposition of $[(Me_3SiCH_2)_3Sn]_3Pr \cdot DME$ (I) in the absence of solvents proceeds at 250°C to afford methyl[tris(trimethylsilylmethyl)]-stannane, hexakis(trimethylsilylmethyl)distannane and unidentified products containing praseodymium, which may be explained by equation 2

$$[(Me_3SiCH_2)_3Sn]_3Pr \cdot DME \xrightarrow[5\ h]{250°C} 2(Me_3SiCH_2)_3SnMe + 1/2(Me_3SiCH_2)_6Sn_2 + \text{unidentified products} \quad (2)$$

The formation of $(Me_3SiCH_2)_3SnMe$ is apparently due to the abstraction of methyl group from the coordinately linked DME.

Hydrolysis of compounds with excess of water leads to the destruction of complexes as described by eq. 3

$$2[(Me_3SiCH_2)_3Sn]_3Ln \cdot DME + 7\ H_2O \xrightarrow[10\ h]{50°C} 4(Me_3SiCH_2)_3SnMe + [(Me_3SiCH_2)_3Sn]_2O + 2\ Ln(OH)_3 \quad (3)$$

The formation of methyl[tris(trimethylsilylmethyl)]stannane in this reaction is unexpected and the mechanism of this process is not entirely clear. It seems likely that in this case the abstraction of methyl groups from the coordinately linked DME also occurs.

The reaction with hydrogen chloride leads to the different products, depending on the reagent ratio. Thus, at a molar ratio of I and HCl 1 : 4 the reaction proceeds according to eq. 4

$$[(Me_3SiCH_2)_3Sn]_3Pr \cdot DME + 4\ HCl \xrightarrow[20°C]{THF} 2(Me_3SiCH_2)_3SnMe + PrCl_3 + (Me_3SiCH_2)_3SnCl + 2\ H_2 \quad (4)$$

At the reagent ratio 1 : 6 the reaction can be described by eq. 5

$$[(Me_3SiCH_2)_3Sn]_3Pr \cdot DME + 6\ HCl \xrightarrow[20°C]{THF} 3(Me_3SiCH_2)_3SnCl + PrCl_3 + 2\ H_2 + 2\ CH_4 \quad (5)$$

Methyl[tris(trimethylsilylmethyl)]stannane is also formed in this reaction at the intermediate step, but it further reacts with hydrogen chloride to give methane. It was confirmed by the reaction of $(Me_3SiCH_2)_3SnMe$ with hydrogen chloride. The composition of residues after the abstraction of methyl groups from DME is not entirely clear yet because of difficulties with their isolation.

It is known [19] that reactions of bimetallic organometallic compounds with organic halides, halogens, benzoyl peroxide and other reagents proceed with the cleavage of metal—metal bonds. We have found that analogous reactions of the lanthanide complexes also proceed with the cleavage of the tin—lanthanide bonds. Thus, the reaction of I with 1,2-dibromoethane leads to the formation of hexakis(trimethylsilylmethyl)distannane, praseodymium bromide and ethylene (eq. 6)

$$2[(Me_3SiCH_2)_3Sn]_3Pr \cdot DME + 3\ BrCH_2CH_2Br \xrightarrow[120°C]{benzene} 3[(Me_3SiCH_2)_3Sn]_2 + 2\ PrBr_3 + 3\ CH_2{=}CH_2 \qquad (6)$$

Apparently, in the initial steps of this reaction tris(trimethylsilylmethyl)tin bromide is formed which reacts further at the Sn—Pr bond to give distannane and praseodymium bromide.

The reaction of complexes obtained with benzoyl peroxide at 50° C yields the unsymmetrical derivative of the lanthanide $(Me_3SiCH_2)_3Sn\text{-}Ln(OCOPh)_2$ and tris(trimethylsilylmethyl)tin benzoate (eq. 7).

$$[(Me_3SiCH_2)_3Sn]_3Ln \cdot DME + 2(PhCOO)_2 \xrightarrow[50°C]{benzene} 2(Me_3SiCH_2)_3SnOCOPh + (Me_3SiCH_2)_3Sn{-}Ln(OCOPh)_2 \qquad (7)$$

Ln = Pr, Nd

Silver trifluoroacetate easily reacts with complexes at room temperature according to eq. 8. Yields of products are rather high.

$$[(Me_3SiCH_2)_3Sn]_3Ln \cdot DME + 6\ CF_3COOAg \xrightarrow[20°C]{THF} 6\ Ag + 3(Me_3SiCH_2)_3SnOCOCF_3 + (CF_3COO)_3Ln \cdot 2\ THF + DME \quad (8)$$

Ln = Pr, Nd

We suppose that the reaction with silver trifluoroacetate as well as the analogous reactions of bimetallic organomercurials [20] proceeds via the formation of intermediate complex of organometallic compound with silver trifluoroacetate.

So, as it is seen from the reactions discussed above, bimetallic organometallic compounds with tin—lanthanide bonds have rather high reactivity together with the thermal stability. We are presently continuing our studies.

Experimental

All experiments were carried out in evacuated sealed systems. THF, DME and other solvents were purified, rigorously dried and immediately before use distilled under argon from lithium aluminium tetrahydride. The melting points were determined in evacuated sealed capillaries. GLC analyses were carried out on a Tsvet-129 chromatograph with a heat conductivity detector using 100 × 0.4 cm column packed with Reoplex 400 (15%) on Chromatone N-AW-DMCS, with helium as the carrier gas. IR spectra were recorded on a UR-20 spectrophotometer.

The IR spectra of the complexes obtained exhibit absorption bands at 1250, 1020, 840 and 720 cm^{-1}, characteristic of Me_3SiCH_2Sn fragment. In addition, in the IR spectra there are absorption bands at 1460, 1100 and 860 cm^{-1} characteristic of the IR spectrum of 1,2-dimethoxyethane. The carbon—oxygen stretching frequency of DME equal to 1120 cm^{-1} is shifted to the region of lower frequencies (1100 cm^{-1}) in the IR spectra of the lanthanide complexes, as expected on coordination.

Tris[tris(trimethylsilylmethyl)stannyl]praseodymium 1,2-dimethoxyethane (I)

To a solution of 2.00 g (3.2 mmol) of tris[bis(trimethylsilyl)amido]praseodymium (II) in 20 ml of DME was added 3.42 g (9.0 mmol) of tris(trimethylsilylmethyl)tin hydride. The mixture was heated at 100°C for 15 h. The solvent was removed by recondensation in vacuo. The pale yellow residue was washed with hexane several times and dried in vacuo to give 3.00 g (73.2%) of $[Me_3SiCH_2)_3Sn]_3Pr \cdot DME$ (I), dec. 270—274°C. (Found: C, 34.16; H, 7.74; Pr, 10.39. $C_{40}H_{109}O_2PrSi_9Sn_3$ calcd.: C, 35.02; H, 8.00; Pr, 10.27%.) GLC analysis of volatile products revealed the presence of 1.30 g (90.3%) of hexamethyldisilazane.

Tris[tris(trimethylsilylmethyl)stannyl]neodymium 1,2-dimethoxyethane (III)

Following the above procedure, 1.30 g (2.1 mmol) of tris[bis(trimethylsilyl)amido]neodymium (IV) in 20 ml of DME on treatment with 2.77 g (7.3 mmol) of $(Me_3SiCH_2)_3SnH$ afforded 1.80 g (63.2%) of $[(Me_3SiCH_2)_3Sn]_3Nd \cdot DME$ (III), dec. 290—291°C. (Found: C, 35.23; H, 7.82; Nd, 11.00. $C_{40}H_{109}NdO_2Si_9Sn_3$ calcd.: C, 34.92; H, 7.98; Nd, 10.48%.) GLC analysis of volatile products showed the presence of 0.92 g (90.0%) of hexamethyldisilazane.

Bis(2-methoxyethanolato)triethylstannylpraseodymium

A mixture of 3.84 g (5.8 mmol) of II and 4.00 g (19.3 mmol) of triethyltin hydride in 25 ml of DME was heated at 100°C for 20 h. DME was replaced with hexane in the usual manner. $Et_3Sn{-}Pr(OCH_2CH_2OMe)_2$ (1.20 g, 41.7%) was isolated, dec. 225—230°C. (Found: C, 28.72; H, 6.09; Pr, 28.02. $C_{12}H_{29}O_4PrSn$ calcd.: C, 29.00; H, 5.88; Pr, 28.35%.) Distillation of the organic layer afforded 1.00 g (25.1%) of hexaethyldistannane, b.p. 160—161°C/23 mmHg, n_D^{28} 1.5370 (lit. [21] b.p. 160°C/23 mmHg, n_D^{28} 1.5374). GLC analysis of the organic layer showed that methyltriethyltin (0.46 g, 20.0%) was present. GLC analysis of the volatile products showed the presence of 1.20 g (42.8%) of hexamethyldisilazane.

Bis(2-methoxyethanolato)triethylstannylneodymium

Following the above procedure, 2.60 g (4.2 mmol) of IV on treatment with a solution of 2.70 g (13.0 mmol) of triethyltin hydride in 20 ml of DME afforded 1.10 g (46.1%) of $Et_3Sn{-}Nd(OCH_2CH_2OMe)_2$, dec. 250°C. (Found: C, 27.97; H, 6.00; Nd, 29.05. $C_{12}H_{29}NdO_4Sn$ calcd.: C, 28.28; H, 5.84; Nd, 28.83%.) GLC analysis of the organic layer gave 0.68 g (25.4%) of hexaethyldistannane and 0.40 g (22.0%) of methyltriethyltin. In addition, 0.96 g (48.5%) of hexamethyldisilazane was identified in the volatile products by GLC.

Reaction of triphenyltin hydride with II

(a). To 2.80 g (8.0 mmol) of triphenyltin hydride was added 1.60 g (2.6 mmol) of II in 20 ml of DME. The slightly exothermic reaction was complete in 3—5 min at ca. 20°C and 85 ml (98.4%) of hydrogen was evolved. The crystalline product which precipitated was recrystallized from hexane to give 2.30 g (82.4%) of hexaphenyldistannane, m.p. 236—238°C. A mixture of Ph_6Sn_2 with an authentic sample gave no melting point depression.

(b). A solution of 0.20 g (0.3 mmol) of II in 20 ml of DME was added to 1.50 g (4.3 mmol) of triphenyltin hydride. After standing at 20°C for 3—5 min the reaction was complete and 45 ml (97.2%) of hydrogen was evolved. Work-up in the usual fashion afforded 1.28 g (85.9%) of hexaphenyldistannane, m.p. (mixed) 237—238°C.

Thermal decomposition of I

2.04 g (1.5 mmol) of I was heated at 250°C for 5 h in an evacuated ampoule. The reaction mixture was extracted several times with hexane. Distillation of the extract gave 1.09 g (95.6%) of methyl[tris(trimethylsilylmethyl)]stannane (V), b.p. 78°C/1 mm Hg, n_D^{20} 1.4810. (Found: C, 39.10; H, 9.19; Si, 21.28; Sn, 29.97. $C_{13}H_{36}Si_3Sn$ calcd.: C, 39.49; H, 9.18; Si, 21.32; Sn, 30.00%.) In addition, hexakis(trimethylsilylmethyl)distannane (0.42 g, 80.0%) was isolated, b.p. 155—157°C/1 mmHg, m.p. (mixed) 107—109°C (in agreement with ref. 22).

A residue (0.45 g), insoluble in hexane and containing praseodymium was not identified.

Hydrolysis of I

To 2.13 g (1.5 mmol) of I in 20 ml of hexane was added 1 ml (excess) of oxygen-free water. The reaction mixture was heated with stirring at 50°C for 10 h. The organic layer was decanted from the precipitate in which 0.30 g (100%) of $Pr(OH)_3$ was identified. The IR spectrum of the isolated $Pr(OH)_3$ is identical with that of the pure sample. In the IR spectrum there are absorption bands at 1100 and 3400 cm^{-1}, characteristic of the Pr—O bond and OH group respectively. By distillation of the organic layer in vacuo, 1.05 g (87.5%) of compound V was obtained, b.p. 78—79°C/1 mmHg, n_D^{20} 1.4820. In addition, 0.54 g (90.0%) of hexakis(trimethylsilylmethyl)distannoxane was isolated, m.p. (mixed) 60—61°C (in agreement with ref. 23).

Hydrolysis of III

Following the above procedure, hydrolysis of 2.40 g (1.7 mmol) of III in 20 ml of hexane with excess of oxygen-free water (1 ml) gave 1.35 g (97.8%) of V, b.p. 78°C/1 mmHg, n_D^{20} 1.4815, and 0.45 g (68.2%) of $[(Me_3SiCH_2)_3Sn]_2O$, m.p. (mixed) 60°C. The usual work-up of the residue afforded 0.36 g (100%) of $Nd(OH)_3$. It was identified by IR spectroscopy.

Reaction of I with hydrogen chloride

(a). A molar ratio 1 : 4. A mixture of 2.40 g (1.7 mmol) of I and 0.26 g (7.1 mmol) of dry HCl in 30 ml of THF was shaken at room temperature for 1 h; 39 ml (49.8%) of hydrogen was evolved. The residue formed was filtered off, washed with THF and dried when 0.39 g (92.9%) of $PrCl_3$ was obtained and

converted to $Pr(OH)_3$, which was identified by IR spectroscopy. Vacuum distillation of the organic layer gave 1.20 g (86.9%) of compound V, b.p. 77—78°C/1 mmHg, n_D^{20} 1.4820, and 0.60 g (83.3%) of tris(trimethylsilylmethyl)tin chloride, b.p. 105—110°C/1 mmHg, m.p. (mixed) 94—95°C (lit. [23] b.p. 106—114°C/1.5 mmHg, m.p. 94—96°C).

(b). A molar ratio 1 : 6. A mixture of 2.40 g (1.7 mmol) of I and 0.38 g (10.4 mmol) of dry HCl in 40 ml of THF was shaken at room temperature for 1 h. A gas mixture (145 ml) containing 75 ml (95.7%) of hydrogen and 70 ml (89.3%) of methane was evolved (GLC). The usual work-up afforded 2.04 g (94.4%) of $(Me_3SiCH_2)_3SnCl$, m.p. (mixed) 93—95°C and 0.39 g (100%) of $PrCl_3$.

(c). Reaction of compound V with HCl. 0.11 g (3.0 mmol) of dry HCl gas was added to a solution of V (1.10 g, 2.8 mmol) in 15 ml of THF. The reaction was complete in 3—5 min at 20°C, after which 60 ml (92.3%) of methane had evolved (GLC). The reaction mixture on usual work-up gave 1.00 g (87.0%) of $(Me_3SiCH_2)_3SnCl$, m.p. (mixed) 94—95°C.

Reaction of I with 1,2-dibromoethane

A mixture of 1.60 g (1.2 mmol) of I and 2.20 g (12.0 mmol) of 1,2-dibromoethane in 10 ml of benzene was heated at 120°C for 5 h; 35 ml (89.3%) of ethylene was evolved. The organic layer was decanted from the residue of $PrBr_3$ (0.30 g, 100%) and distilled. $(Me_3SiCH_2)_6Sn_2$ (1.10 g, 84.6%) was obtained, b.p. 155—156°C/1 mmHg, m.p. (mixed) 106—108°C.

Reaction of I with benzoyl peroxide

2.20 g (1.6 mmol) of I in 5 ml of benzene was added to a solution of 0.80 g (3.3 mmol) of benzoyl peroxide in 10 ml of benzene. The reaction mixture was shaken at 50°C for 12 h. A precipitate of $(Me_3SiCH_2)_3SnPr(OCOPh)_2$ (1.25 g, 98.4%) was formed, dec. 156—160°C. (Found: C, 40.42; H, 5.33; Pr, 19.02. $C_{26}H_{43}O_4PrSi_3Sn$ calcd.: C, 40.90; H, 5.67; Pr, 18.46%.) In the IR spectrum of the compound obtained there are absorption bands at 1645 and 1420 cm^{-1}, characteristic of the C=O group, and absorption bands characteristic of Me_3SiCH_2Sn fragments. Fractionation of the organic layer afforded 1.20 g (75%) of $(Me_3SiCH_2)_3SnOCOPh$, b.p. 140—141°C/0.5 mmHg, n_D^{20} 1.5140 (lit. [24] b.p. 141—142°C/0.5 mmHg, n_D^{20} 1.5138).

Reaction of III with benzoyl peroxide

Following the above procedure, a solution of 0.65 g (2.7 mmol) of benzoyl peroxide in 10 ml of benzene on treatment with 1.70 g (1.3 mmol) of III in 5 ml of benzene gave 0.85 g of $(Me_3SiCH_2)_3SnOCOPh$, b.p. 141—142°C/0.5 mmHg, n_D^{20} 1.5135 and 0.90 g (95.7%) of lilac solid $(Me_3SiCH_2)_3SnNd(OCOPh)_2$, dec. 163—165°C. (Found: C, 42.75; H, 5.49; Nd, 18.53. $C_{26}H_{43}NdO_4Si_3Sn$ calcd.: C, 42.50; H, 5.90; Nd, 18.81%.) The IR spectrum of the compound is identical with that of $(Me_3SiCH_2)_3SnPr(OCOPh)_2$.

Reaction of I with silver trifluoroacetate

A solution of 2.44 g (11.0 mmol) of CF_3COOAg in 25 ml of THF was added in small portions to 2.36 g (1.7 mmol) of I in 15 ml of THF. After 3—5 min at ca. 20°C the mixture was completely discoloured. The organic layer was decanted from the metallic silver (0.90 g, 80.4%) and hexane was added in the usual manner. $(CF_3COO)_3Pr \cdot 2$ THF (0.80 g, 99.0%) was precipitated. (Found: C, 26.36; H, 2.85; Pr, 22.39. $C_{14}H_{16}F_9O_8Pr$ calcd.: C, 26.94; H, 2.58; Pr, 22.57%.) In the IR spectrum of the compound obtained there is an absorption band at 1660 cm^{-1} corresponding to the carbonyl group vibrations. Two bands at 1200 and 1150 cm^{-1} can be assigned to the vibrations of the CF_3 groups [25]. The presence of THF in $(CF_3COO)_3Pr \cdot 2$ THF is confirmed by absorptions at 1080 and 920 cm^{-1} in the IR spectrum of this compound [25]. Vacuum distillation of the organic layer gave 2.04 g (80.3%) of $(Me_3SiCH_2)_3SnOCOCF_3$, b.p. 96—98°C/1 mmHg, n_D^{20} 1.4590. (Found: C, 34.44; H, 6.98. $C_{14}H_{33}F_3O_2Si_3Sn$ calcd.: C, 34.08; H, 6.74%.) In addition, GLC analysis of the volatile products showed the presence of 0.16 g of DME which corresponds to one mole of coordinately linked DME in the starting compound I.

Reaction of III with silver trifluoroacetate

To 1.70 g (1.2 mmol) of III in 10 ml of THF was added in small portions a solution of 1.60 g (7.2 mmol) of CF_3COOAg in 20 ml of THF. After standing at 20°C for 3—5 min the colour disappeared. The reaction mixture was worked up as in the above experiment to give 0.82 g (100%) of the metallic silver and 0.64 g (100%) of $(CF_3COO)_3Nd \cdot 2$ THF. (Found: C, 26.24; H, 2.85; Nd, 23.28. $C_{14}H_{16}F_9NdO_8$ calcd.: C, 26.79; H, 2.57; Nd, 23.05%.) The IR spectrum of this compound is identical with that given for $(CF_3COO)_3Pr \cdot 2$ THF in the above experiment. By distillation of the organic layer in vacuo 1.70 g (93.4%) of $(Me_3SiCH_2)_3SnOCOCF_3$ was obtained, b.p. 97—98°C/1 mmHg, n_D^{20} 1.4600. GLC analysis of the volatile products gave 0.11 g of DME, which corresponds to one mole of coordinately linked DME in the starting compound III.

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