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(50) In the ammonium compounds ν_s(FHF) is shifted to higher energy and δ(FHF) to lower energy relative to potassium trihydrogen tetrafluoride (Table II), which is indicative of weaker F–H···F hydrogen bonds.

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Fluorinated Four-Membered Sila or Stanna Heterocycles with Nitrogen and Elements of Group 4, 5, or 6

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The dilithium salts $(CH_3)_2Si[NLiC(CF_3)_2CH_3]_2$ and $(CH_3)_2Sn[NLiC(CF_3)_2CH_3]_2$ are ready precursors to cyclic compounds. They can be reacted with $(CH_3)_2SiCl_2$, $(CH_3)_2SnCl_2$, $(CH_3)_2GeCl_2$, $(C_5H_5)_2TiCl_2$, $(C_5H_5)_2ZrCl_2$, $(C_6H_5)_2PbCl_2$, $C_2H_5PCl_2$, and $SeCl_4$ to form the corresponding four-membered heterocycles and the spiro compound.

The current literature reflects the increasing interest in cyclodisilazanes and cyclodistannazanes[1–4] as well as in the mixed four-membered heterocycles[4,5] and spiro compounds with silicon on tin or phosphorus and nitrogen.[4–9] However, with the exception of cyclodisilazanes with pentafluorophenyl groups bonded to the nitrogen members of the rings,[10,11] the nitrogen substituents on these compounds are typically hydrogenated.

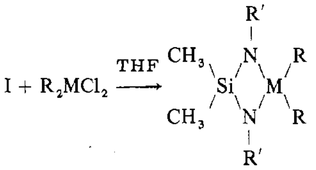
We now have been able to prepare a variety of four-membered heterocycles with nitrogen and silicon or tin and germanium or titanium or phosphorus or zirconium or lead as well as a spiro compound which contains selenium. The substituents on nitrogen are fluorinated alkyl groups. The compounds $(CH_3)_2M[NLiC(CF_3)_2CH_3]_2$ (M = Si, Sn) are useful nucleophiles in the preparation of these new cyclic molecules.

Discussion

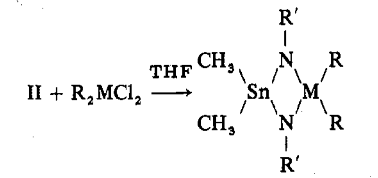
Polar addition of methyllithium to the >C=N– bond of the hexafluoroisopropylidenimino group in $(CH_3)_2M[N{=}C(CF_3)_2]_2$ (M = Si, Sn) gives rise to a dilithium salt which is useful in the preparation of heterocycles.

$$(CH_3)_2M[N{=}C(CF_3)_2]_2 + 2CH_3Li \xrightarrow{Et_2O} \underset{\substack{\text{I, M = Si} \\ \text{II, M = Sn}}}{(CH_3)_2M[NLiC(CF_3)_2CH_3]_2}{}^{12}$$

These dilithium salts are soluble in tetrahydrofuran. While it is possible to prepare a cyclodisilazane by reacting I with dimethyl(dichloro)silane, the analogous cyclodistannazane did not result when II was reacted with dimethyl(dichloro)stannane. However, several mixed four-membered heterocycles were synthesized in this manner:

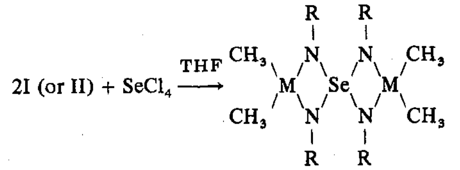


M = Si,[12] Sn,[12] Ge, P, Ti, Zr, Pb; R = CH_3 except R = C_5H_5 when M = Ti, Zr, R = C_2H_5 when M = P, and R = C_6H_5 when M = Pb; R′ = $C(CF_3)_2CH_3$



M = Ge, Ti, Zr, Pb; R = CH_3, C_5H_5, C_6H_5; R' = $C(CF_3)_2CH_3$

Although a variety of reactions were studied in an attempt to prepare spiro compounds, we found that only $SeCl_4$ was a useful precursor to such a molecule.



M = Si, Sn; R = $C(CF_3)_2CH_3$

We were unable to form the mixed Si–Se–Sn compound.

All of these compounds are low-melting stable solids (82–123 °C) with the exception of the spiro ones which melt at 161 °C (Si) and 185 °C (Sn). A molecular ion is observed in the mass spectrum of each of the new compounds.

Experimental Section

Materials. $(CH_3)_2SiCl_2$ (Matheson Coleman and Bell), $(CH_3)_2SnCl_2$, $(C_6H_5)_2ZrCl_2$, and $(C_6H_5)_2PbCl_2$ (PCR), $(CH_3)_2GeCl_2$, $(C_5H_5)_2TiCl_2$, and $SeCl_4$ (Alfa Inorganics), and $CH_3CH_2PCl_2$ (Ethyl Corp.) were used as received without further purification. Literature method preparations were used for $(CH_3)_2Si[N{=}C(CF_3)_2]_2$ and $(CH_3)_2Sn[N{=}C(CF_3)_2]_2$,[13] $HN{=}C(CF_3)_2$,[14] $LiN{=}C(CF_3)_2$,[15] and $(CH_3)_2M[NLiC(CF_3)_2CH_3]$ [I (M = Si), II (M = Sn)].[12]

General Procedures. Most gases and volatile liquids were handled in a conventional Pyrex vacuum apparatus equipped with a Heise-Bourdon tube gauge. Products were purified by sublimation or recrystallization and handled as solids. Infrared spectra were recorded on a Perkin-Elmer 457 spectrometer. The ^{19}F NMR spectra were obtained on a Varian HA-100 spectrometer by using Freon-11 (CCl_3F) as an internal standard. The ^1H NMR spectra were obtained on a Varian EM-360 spectrometer by using TMS as an external standard. Mass spectra were obtained with a Hitachi Perkin-Elmer RMU-6E spectrometer at an ionization potential of 17 or 70 eV.

Preparation of Heterocycles. **$CH_3(CF_3)_2CN$-$Si(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Si(CH_3)_2$.**[12] Freshly distilled THF (15 mL) was added to the dry dilithium salt $(CH_3)_2Si[NLiC(CF_3)_2CH_3]_2$ (6 mmol). $(CH_3)_2SiCl_2$ (6 mmol) in THF (5 mL) was added dropwise to the solution over a period of 30 min via syringe through the side arm under an atmosphere of nitrogen. The reaction vessel was immersed in an ice bath, and after being stirred for 1 h at that temperature, the reaction mixture was allowed to warm to ambient temperature. The mixture was stirred for 24 h after which the solvent was removed. The product was isolated by sublimation (41% yield). It was identified from its previously reported spectral data.[12]

$CH_3(CF_3)_2CN$-$Si(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Sn(CH_3)_2$. The dilithium salt (I, 6 mmol) and $(CH_3)_2SnCl_2$ (6 mmol) in dry THF were used as in the preceding reaction and worked up similarly. The product was isolated by sublimation (38% yield); mp 113.5–115 °C. The compound was identified from spectral data reported previously.[12]

$CH_3(CF_3)_2CN$-$Si(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Ge(CH_3)_2$. The dilithium salt (I, 5 mmol) and $(CH_3)_2GeCl_2$ (5 mmol) in freshly THF were reacted in the same manner. The product was isolated by recrystallization from ethanol (31% yield); mp 79–81 °C. The ^1H NMR spectrum has peaks at τ 8.40, 8.80, and 9.96 and the ^{19}F spectrum a peak at ϕ 76.1. The infrared spectrum is as follows: 2970 (w), 1460 (m), 1388 (m), 1285 (s), 1262 (ms), 1210 (vs), 1190 (s), 1120 (vs), 1078 (s), 969 (m), 830 (ms), 700 (ms), 532 cm^{-1} (w).

Anal. Calcd for $C_{12}H_{18}N_2SiGeF_{12}$: C, 27.72; H, 3.49; N, 5.39. Found: C, 28.41; H, 3.82; N, 5.62.

$CH_3(CF_3)_2CN$-$Si(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Ti(C_5H_5)_2$. Equimolar amounts (5 mmol) of the dilithium salt I and bis(cyclopentadienyl)titanium dichloride were used as in the reaction above and worked up similarly. The product, which was dark red, was recrystallized from ethanol (18% yield); mp 90–91 °C. The ^1H NMR spectrum has peaks at τ 4.00, 8.52, and 9.85 and the ^{19}F spectrum a peak at ϕ 77.2. The infrared spectrum is as follows: 2950 (w), 1440 (ms), 1368 (m), 1290 (s), 1240 (s), 1212 (s), 1180 (s), 1124 (s), 1090 (ms), 1018 (ms), 962 (m), 940 (m), 830 (w), 760 (m), 731 (m), 575 (m), 490 cm^{-1} (w).

Anal. Calcd for $C_{20}H_{22}N_2SiTiF_{12}$: C, 40.35; H, 3.73; N, 4.71. Found: C, 40.91; H, 3.53; N, 4.81.

$CH_3(CF_3)_2CN$-$P(CH_2CH_3)$-$N[C(CF_3)_2CH_3]$-$Si(CH_3)_2$. In an analogous manner, a solution of $CH_3CH_2PCl_2$ (5 mmol) in dry THF was added dropwise slowly into the solution of the dilithium salt I (5 mmol) at 0 °C. After warming to ambient temperature, the reaction mixture was stirred for 24 h and the solvent was removed. The pale brown solid was recrystallized from benzene to give the product (43% yield); mp 103 °C. The ^1H NMR spectrum has four signals at τ 7.74 ($J_{H\text{-}P}$ = 20 Hz), 8.13, 8.70 ($J_{H\text{-}P}$ = 11 Hz), and 9.94, and the ^{19}F spectrum has a peak at ϕ 77.9. The infrared spectrum is as follows: 2955 (w), 1455 (m), 1378 (m), 1285 (s), 1210 (s), 1185 (s), 1160–1078 (vs), 871 (m), 831 (w), 793 (w), 703 (w), 653 (w), 554 (m), 418 cm^{-1} (w).

Anal. Calcd for $C_{12}H_{17}N_2SiPF_{12}$: C, 30.26; H, 3.60; N, 5.88. Found: C, 29.81; H, 3.65; N, 5.58.

$CH_3(CF_3)_2CN$-$Si(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Zr(C_5H_5)_2$. Equimolar amounts (5 mmol) of the dilithium salt I and bis(cyclopentadienyl)zirconium dichloride were used as in the preceding reaction and worked up similarly. The product was recrystallized from ethanol (36% yield); mp 121–123 °C. The ^1H NMR spectrum has peaks at τ 3.87, 8.61, and 9.91 and the ^{19}F spectrum a peak at ϕ 76.1. The infrared spectrum is as follows: 2951 (w), 1451 (m), 1372 (m), 1288 (s), 1240 (s), 1208 (s), 1121 (s), 1088 (ms), 1010 (m), 981 (ms), 765 (m), 732 (m), 570 (m), 490 cm^{-1} (w).

Anal. Calcd for $C_{20}H_{20}N_2SiZrF_{12}$: C, 37.61; H, 3.47; N, 4.39. Found: C, 37.42; H, 3.58; N, 4.12.

$CH_3(CF_3)_2CN$-$Si(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Pb(C_6H_5)_2$. In a similar manner, a solution of diphenyllead dichloride (5 mmol) in dry THF was added dropwise to the solution of the dilithium salt I (5 mmol) at 0 °C and worked up similarly. The product was recrystallized from ethanol (31% yield); mp 102–104 °C. The ^1H NMR spectrum has peaks at τ 2.10, 8.71, and 9.86 and the ^{19}F a peak at ϕ 76.1. The infrared spectrum is as follows: 3004 (w), 2960 (w), 1602 (m), 1542 (w), 1498 (m), 1465 (m), 1387 (m), 1290 (s), 1242 (s), 1211 (s), 1130 (s), 1076 (m), 992 (m), 761 (w), 501 cm^{-1} (w).

Anal. Calcd for $C_{22}H_{22}N_2SiPbF_{12}$: C, 33.94; H, 2.85; N, 3.60. Found: C, 33.69; H, 2.91; N, 3.60.

$CH_3(CF_3)_2CN$-$Sn(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Ge(CH_3)_2$. To a solution of $(CH_3)_2Sn[N{=}C(CF_3)_2]_2$ (5 mmol) and dry THF (15 mL), which was cooled in an ice bath, methyllithium (10 mmol) in Et_2O was added slowly under an atmosphere of nitrogen. When the addition was completed, the reaction mixture was stirred at 0 °C for 1 h. $(CH_3)_2GeCl_2$ (5 mmol) in THF (5 mL) was added to the vessel via syringe through the side arm. The reaction mixture was allowed to warm to ambient temperature and then stirred for 24 h. The product was recrystallized from ethanol (35% yield); mp 112–113 °C. The ^1H NMR spectrum has peaks at τ 8.20, 8.75, and 9.90, and the ^{19}F spectrum a peak at ϕ 78.1. The infrared spectrum is as follows: 2955 (w), 1468 (m), 1390 (m), 1283 (s), 1260 (s), 1162 (s), 1125 (s), 1075 (s), 1028 (ms), 880 (ms), 857 (m), 792 (m), 556 (w), 437 (ms), 382 cm^{-1} (w).

Anal. Calcd for $C_{12}H_{18}N_2SnGeF_{12}$: C, 23.61; H, 2.97; N, 4.59. Found: C, 23.73; H, 3.01; N, 4.72.

$CH_3(CF_3)_2CN$-$Sn(CH_3)_2$-$N[C(CF_3)_2CH_3]$-$Ti(C_5H_5)_2$. Equimolar amounts (5 mmol) of $(CH_3)_2Sn[N{=}C(CF_3)_2]_2$ and $(C_5H_5)_2TiCl_2$ and 10 mmol of CH_3Li were used as in the above preparation and worked up similarly. The product, which was dark red, was recrystallized from ethanol to give the product (5% yield); mp 121–123 °C. The ^1H NMR spectrum has peaks at τ 3.91, 8.40, and 9.86, and the ^{19}F spectrum a peak at ϕ 77.5. The infrared spectrum is as follows: 2957 (w), 1442 (ms), 1376 (m), 1292 (s), 1246 (s), 1184 (s), 1086 (ms), 1016 (ms), 951 (m), 876 (w), 831 (w), 750 (m), 556 (m), 401 cm^{-1} (w).

Anal. Calcd for $C_{20}H_{22}N_2SnTiF_{12}$: C, 35.02; H, 3.23; N, 4.08. Found: C, 35.13; H, 3.31; N, 4.25.

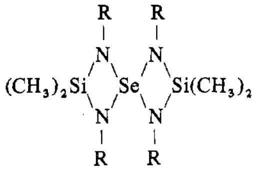
$CH_3(CF_3)_2C\overline{N-Sn(CH_3)_2-N[C(CF_3)_2CH_3]-Zr}(C_5H_5)_2$. In an analogous manner, the dilithium salt II (5 mmol) and $(C_5H_5)_2ZrCl_2$ (5 mmol) in dry THF were used as in the above reaction and worked up similarly. The product was recrystallized from ethanol (11% yield); mp 136–138 °C. The ^1H NMR spectrum has peaks at τ 3.85, 8.42, and 9.90 and the ^{19}F spectrum a peak at ϕ 76.5. The infrared spectrum is as follows: 2950 (w), 1460 (m), 1380 (m), 1291 (ms), 1245 (s), 1201 (s), 1116 (s), 1075 (m), 1005 (m), 978 (ms), 751 (m), 726 (m), 495 cm^{-1} (w).

Anal. Calcd for $C_{20}H_{22}N_2SnZrF_{12}$: C, 32.94; H, 3.04; N, 3.84. Found: C, 32.90; H, 3.16; N, 3.92.

$CH_3(CF_3)_2C\overline{N-Sn(CH_3)_2-N[C(CF_3)_2CH_3]-Pb}(C_6H_5)_2$. The dilithium salt II (5 mmol) and $(C_6H_5)_2PbCl_2$ (5 mmol) in dry THF were used in the same manner. The product was recrystallized from ethanol (16% yield); mp 131–133 °C. The ^1H NMR spectrum has peaks at τ 2.15, 8.81, and 9.90 and the ^{19}F spectrum a peak at ϕ 76.6. The infrared spectrum is as follows: 3002 (w), 2960 (w), 1600 (m), 1546 (m), 1486 (m), 1383 (m), 1281 (s), 1221 (s), 1198 (s), 1081 (m), 991 (m), 781 (w), 506 cm^{-1} (w).

Anal. Calcd for $C_{22}H_{22}N_2SnPbF_{12}$: C, 30.40; H, 2.55; N, 3.22. Found: C, 30.27; H, 2.61; N, 3.19.

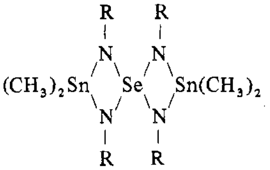
Compound III (R = $C(CF_3)_2CH_3$). To a mixture of the dilithium salt I (5 mmol) and dry THF (15 mL) selenium tetrachloride (2.5 mmol) was added under an atmosphere of nitrogen. The mixture was stirred for 24 h. The pale yellow product was recrystallized from THF (29% yield); mp 161–164 °C. The ^1H NMR spectrum has peaks at τ 8.48 and 9.98, and the ^{19}F spectrum a peak at ϕ 77.9. The infrared spectrum is as follows: 2961 (w), 1451 (m), 1390 (m), 1291 (s), 1256–1221 (vs), 1191 (s), 1081 (ms), 1021 (m), 956 (m), 871 (w), 748 (m), 545 (m), 408 (w), 381 cm^{-1} (w).



III

Anal. Calcd for $C_{20}H_{24}N_4Si_2SeF_{24}$: C, 26.30; H, 2.65; N, 6.13. Found: C, 26.12; H, 2.81; N, 6.23.

Compound IV (R = $C(CF_3)_2CH_3$). Five millimoles of the dilithium salt II and 2.5 mmol of $SeCl_4$ were reacted as above and worked up similarly. The pale yellow product was recrystallized from ethanol (15% yield); mp 185–186 °C. The ^1H NMR spectrum has peaks at τ 8.51 and 9.92 and the ^{19}F spectrum a peak at 77.9. The infrared spectrum is as follows: 2958 (w), 1449 (m), 1389 (m), 1293 (s), 1251 (s), 1239 (s), 1186 (s), 1078 (ms), 1020 (m), 958 (m), 868 (w), 751 (m), 551 (m), 406 cm^{-1} (w).



IV

Anal. Calcd for $C_{20}H_{24}N_4Sn_2SeF_{24}$: C, 21.94; H, 2.21; N, 5.12. Found: C, 21.56; H, 2.15; N, 5.31.

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Registry No. I, 57821-12-2; II, 63089-38-3; III, 63089-39-4; IV, 63089-40-7; $CH_3(CF_3)_2C\overline{N-Si(CH_3)_2-N[C(CF_3)_2CH_3]-Si}(CH_3)_2$, 57821-13-3; $CH_3(CF_3)_2C\overline{N-Si(CH_3)_2N[C(CF_3)_2CH_3]-Sn}(CH_3)_2$, 57821-14-4; $CH_3(CF_2)_2C\overline{N-Si(CH_3)_2-N[C(CF_3)_2CH_3]-Ge}(CH_3)_2$, 63089-41-8; $CH_3(CF_3)_2C\overline{N-Si(CH_3)_2-N[C(CF_3)_2CH_3]-Ti}(C_5H_5)_2$, 63105-39-5; $CH_3(CF_3)_2C\overline{N-P(CH_2CH_3)-N[C(CF_3)_2CH_3]-Si}(CH_3)_2$, 63089-42-9; $CH_3(CF_3)_2C\overline{N-Si(CH_3)_2-N[C(CF_3)_2CH_3]-Zr}(C_5H_5)_2$, 63105-38-4; $CH_3(CF_3)_2C\overline{N-Si(CH_3)_2-N[C(CF_3)_2CH_3]-Pb}(C_6H_5)_2$, 63089-43-0; $CH_3(CF_3)_2C\overline{N-Sn(CH_3)_2-N[C(CF_3)_2CH_3]-Ge}(CH_3)_2$, 63089-44-1; $CH_3(CF_3)_2C\overline{N-Sn(CH_3)_2-N[C(CF_3)_2CH_3]-Ti}(C_5H_5)_2$, 63105-37-3; $CH_3(CF_3)_2C\overline{N-Sn(CH_3)_2-N[C(CF_3)_2CH_3]-Zr}(C_5H_5)_2$, 63105-36-2; $CH_3(CF_3)_2C\overline{N-Sn(CH_3)_2-N[C(CF_3)_2CH_3]-Pb}(C_6H_5)_2$, 63122-44-1; $(CH_3)_2SiCl_2$, 75-78-5; $(CH_3)_2SnCl_2$, 753-73-1; $(CH_3)_2GeCl_2$, 1529-48-2; $(C_5H_5)_2TiCl_2$, 1271-19-8; $(C_5H_5)_2ZrCl_2$, 1291-32-3; $CH_3CH_2PCl_2$, 1498-40-4; $(C_6H_5)_2PbCl_2$, 2117-69-3; $(CH_3)_2Sn[N{=}C(CF_3)_2]_2$, 40168-62-5; $SeCl_4$, 10026-03-6.

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