which had a molecular weight of 1000 and $[\alpha]_D$ −8.4° (*c* 0.0083, $CHCl_3$). ^{1}H NMR ($CDCl_3$): 5.3, 1.9, 1.6, 0.9 ppm (br). The ^{1}H NMR spectrum was found to match with that reported previously.[30]

Catalytic Polymerization of Phenylcyclopropane. Phenylcyclopropane (0.80 mL, 0.756 g, 6.4 mmol) was added to a solution of 0.050 g of **1** (0.093 mmol in Eu(III)) in 1.0 mL of CH_3NO_2. The solution was allowed to stir for 5 days after which time an oily white layer appeared above the catalyst solution. $CHCl_3$ (40 mL) was added, and the resulting mixture was chromatographed through a silica gel column. Removal of the solvent under vacuum yielded 0.71 g (95%) of a viscous oil of molecular weight 600. ^{1}H NMR ($CDCl_3$): 7.17 (4 H, br), 3.63 (1 H, br), 1.96 (2 H, br), 0.84 ppm (3 H, br). $^{13}C\{^1H\}$ NMR ($CDCl_3$): 145.3, 142.7, 128.8–125.9, 52.9, 52.5, 28.9, 28.7, 12.8 ppm. Selected ^{13}C NMR (^{1}H-coupled): 52.9 (d, J = 125 Hz, CH), 52.5 (d, J = 125 Hz, CH), 28.9 (t, J = 125 Hz, CH_2), 28.7 (t, J = 125 Hz, CH_2), 12.8 ppm (q, J = 125 Hz, CH_3).

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Registry No. 1, 102233-99-8; Eu, 7440-53-1; $(CH_3)_2C{=}C(CH_3)_2$, 563-79-1; $CH_2{=}C(CH_3)CH(CH_3)_2$, 563-78-0; 1-PhCH-CH_2-$)_n$, 9003-53-6; $PhCH{=}CH_2$, 100-42-5; $PhC(CH_3){=}CH_2$, 98-83-9; 1-PhC-$(CH_3)CH_2$-$)_n$, 25014-31-7; $Ph_2C{=}CH_2$, 530-48-3; 1,3-cyclohexadiene, 592-57-4; poly(1,3-cyclohexadiene) (homopolymer), 27986-50-1; poly-(1,3-cyclohexadiene) (SRU), 102233-96-5; norbornadiene, 121-46-0; 1,3-dimethyl-1,3-diphenyl-2,3-dihydroindene, 102233-98-7; 1,3-dimethyl-1-(2-methyl-2-phenylpropyl)-3-phenyl-2,3-dihydroindene, 41906-71-2; 1-methyl-1,3,3-triphenyl-2,3-dihydroindene, 19303-32-3; 6,6′-dimethyl-2-methylenebicyclo[3.1.1]heptane, 127-91-3; poly(6,6′-dimethyl-2-methylenebicyclo[3.1.1]heptane) (homopolymer), 25719-60-2; poly(6,6′-dimethyl-2-methylenebicyclo[3.1.1]heptane) (SRU), 40022-48-8; cyclopropylbenzene, 873-49-4; polycyclopropylbenzene (homopolymer), 102233-95-4; polycyclopropylbenzene (SRU), 102233-97-6; tetracyclo[$3.2.0^{2,7}.0^{4,6}$]heptane, 278-06-8.

Group IIB (12[†]) Metal Alkyls: The Synthesis of Trifluorosilyl and Trifluoromethyl Alkyls of Cadmium and Zinc

M. A. Guerra, T. R. Bierschenk, and R. J. Lagow*

Contribution from the Department of Chemistry, University of Texas, Austin, Texas 78712.
Received October 9, 1985

Abstract: Metal vapors of cadmium and zinc have been reacted with trifluorosilyl and trifluoromethyl radicals to produce several new metal alkyls. A low temperature radio frequency glow discharge or plasma of hexafluoroethane and hexafluorodisilane was used to generate the trifluoromethyl and trifluorosilyl radicals, respectively. The compounds bis(trifluorosilyl)cadmium, bis(trifluorosilyl)zinc, bis(trifluoromethyl)cadmium, and bis(trifluoromethyl)zinc were isolated at low temperatures. All compounds were unstable at room temperature with the exception of bis(trifluorosilyl)cadmium which was marginally stable at 20 °C. Addition of glyme (glyme = dimethoxyethane) to the cadmium compounds gave stable alkyl–cadmium–glyme complexes while the addition of pyridine to the zinc compounds yielded stable complexes. The synthesis and physical properties are reported.

Over the past 6 years, a novel synthesis for fully substituted metal alkyls has been developed in our laboratories. The technique involves the reaction of metal vapors with radicals that are produced in a radio frequency generated glow discharge or plasma. Although radicals can be produced by using a variety of techniques, we have found low pressure plasmas to be very clean and convenient source of radicals if plasma gases with very simple and symmetrical structures are used. For example, ethane, hexafluoroethane, and hexafluorodisilane have been used as a source for methyl, trifluoromethyl and trifluorosilyl radicals, respectively.[1]

In spite of the extensive study of fluorocarbon-substituted metal compounds in recent years, very few compounds exist in which the SiF_3 ligand is bonded to an element other than carbon. A general synthesis for trifluorosilyl organometallic compounds has been absent from the scene, and it is particularly significant that no organolithium reagent such as $LiSiF_3$ exists, for this potential reagent is unstable at temperatures above −80 °C.[2] The first metal trifluorosilyl complex, $F_3SiCo(CO)_4$, was prepared by using a method analogous to that used by Chalk and Harrod for preparing $R_3SiCo(CO)_4$ complexes (R = organic groups).[3] The reaction of trifluorosilane with the binuclear metal carbonyl $Co_2(CO)_8$ gave $F_3SiCo(CO)_4$ in 84% yield.[4] Similar reactions at 160 °C with $Mn_2(CO)_{10}$, $Re_2(CO)_{10}$, and $[Fe(\pi\text{-}C_5H_5)(CO)_2]_2$ yielded $F_3SiMn(CO)_5$, $F_3SiRe(CO)_5$, and $F_3SiFe(\pi C_5H_5)(CO)_2$.[4] It was later noticed that the compounds could be made equally well with iodotrifluorosilane in place of the trifluorosilane, but the products were more difficult to separate.[5] The scarcity of known trifluorosilyl compounds is not too surprising when one considers the forcing reaction conditions necessary (160 °C) with older synthetic methods. Recently Sharp has reported the synthesis of P $(SiF_3)_3$ using a novel approach.[6] The compound was prepared from ·SiF_3 radicals generated by mercury-sensitized photolysis of Si_2F_6 in the presence of PF_3.

In an earlier publication, we reported the successful preparation of the first fully substituted trifluorosilyl complexes.[1c] For example, $Hg(SiF_3)_2$, $Bi(SiF_3)_3$, $Te(SiF_3)_2$, and $Sb(SiF_3)_3$ can be prepared by using the metal vapor/plasma chemistry.

Experimental Section

Starting Materials. Cadmium shot was obtained from PCR and zinc mesh was purchased from MCB. Reagent grade pyridine and glyme were used after drying over 4Å molecular sieves. Ether, methylene

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[†] In this paper the periodic group notation in parentheses is in accord with recent actions by IUPAC and ACS nomenclature committees. A and B notation is eliminated because of wide confusion. Groups IA and IIA become groups 1 and 2. The d-transition elements comprise groups 3 through 12, and the p-block elements comprise groups 13 through 18. (Note that the former Roman number designation is preserved in the last digit of the new numbering: e.g., III → 3 and 13.)

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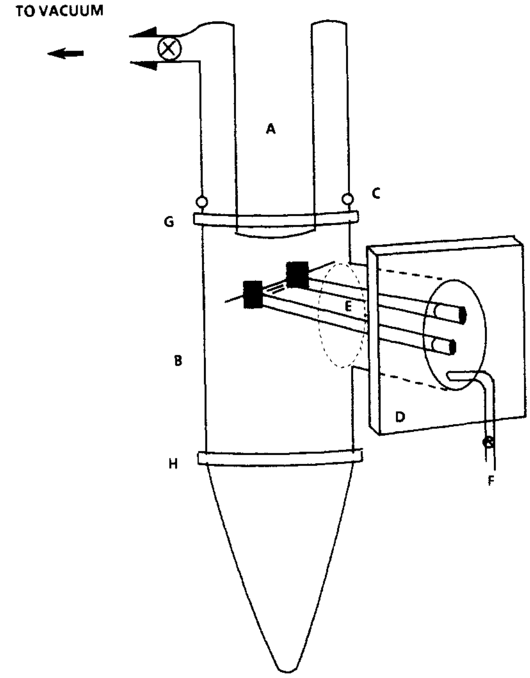

Figure 1. Pyrex reactor used in the experiments.

chloride, and hexane were degassed and dried over CaH_2. Calcium silicide (Petrarch Systems, Inc.) and liquified chlorine (Amerigas) were used without further purification. Antimony halides were obtained from Alfa Products. The hexafluorodisilane was prepared by using a slight modification of the method reported by Booth and Sullivan.[7] Hexachlorodisilane was fluorinated with SbF_3 in a closed system with $SbCl_5$ used as a catalyst. Calcium silicide was chlorinated at 150 °C to prepare hexachlorodisilane. Hexafluoroethane was dielectric grade Du Pont Freon 116. Purification of the metal alkyl complexes was done by recrystallization from hexane. Fluorine and hydrogen NMR spectra were recorded on a Varian EM 390 spectrometer operating at 84.6 and 90 MHz, respectively. Low-temperature ^{19}F NMR spectra were recorded on a Bruker WH-90 spectrometer with deuterated methylene chloride being used for the lock signal. IR spectra were taken on a Beckman IR-20A. The mass spectra were taken on a Bell and Howell Model 21-490 low-resolution mass spectrometer operating at 70 eV with a source temperature of 240 ° C.

Apparatus. The metal vapor/plasma reacotr used is shown in Figure 1. The top protion of the reactor consisted of a 60-mm Pyrex cold finger (A) that was sealed to a Pyrex reactor base (B) with use of a 100-mm O-ring connector (C). The metal was vaporized from a tungsten basket which was resistively heated by power supplied by two copper leads (E). The copper leads were water-cooled and sealed to a $^1/_2$ in. Plexiglas plate (D) which was attached to the reactor bottom by a 75-mm O-ring. Hexafluorodisilane and hexafluoroethane entered the reactor through an inlet (F). The solvents and reagents were also introduced through inlet F. A Tegal Corporation, 100-W, 13.56-MHz radio-frequency generator equipped with a matching network was used to generate the radio-frequency power. The plasma gas was capacitively coupled with the radio-frequency generator with two metal bands (G and H).

General Procedure. The metal is preweighed and loaded on a tungsten basket or in a quartz crucible which is resistively heated. The cold finger is filled with liquid nitrogen and the plasma gas, either hexafluoroethane or hexafluorodisilane, is metered into the reactor at a rate of approximately 60 mm/h. The radio-frequency power is turned on and, if necessary, the plasma is initiated with a Tesla coil. Following the reaction, the unreacted plasma gas is pumped from the reactor leaving behind the compound of interest. All of the products are initially treated as being extremely air sensitive and are handled only in an argon drybox. In the case of the unsolvated $Cd(CF_3)_2$ and $Zn(CF_3)_2$ compounds, the reactor and glassware used for subsequent handling and storage of the compounds are never warmed to a temperature greater than −45 °C until the thermal stability of the complexes is first checked by ^{19}F NMR. Typically, a stainless steel cannula is used to quickly transfer the compound from the reactor into a glass bulb with a NMR tube fused to the bottom. The carrier solvent is vacuum distilled from the bulb, leaving behind the majority of the compound in the bottom of the NMR tube. An appropriate NMR solvent is then added and the tube is sealed in vacuo.

Preparation of Compunds. (a) $Cd(SiF_3)_2$. Cadmium vapor (100 mg) was vaporized and condensed with SiF_3 radicals on a liquid nitrogen cooled surface. Following the 2-h reaction, the reactor was warmed to −40 °C to pump off the excess Si_2F_6. Methylene chloride (10 mL) was condensed into the reactor at −196 °C. The reactor was warmed to 0 °C and the crude product mixture was removed via a cannula. Evaporation of the methylene chloride gave the marginally stable compound Cd $(SiF_3)_2$. The compound has a half-life of approximately 24 h at +25 °C. ^{19}F NMR (84.87 MHz, Cd_2Cl_2) δ 102.3 (s, $J(Cd^{113/111}\text{–}F)$ = 601/564 Hz); IR (KBr pellet) 1250 (m), 1070 (vs), 1005 (s), and 785 (m) cm^{-1}; mass spectrum, $Cd(SiF_3)_2^+$ (1.4%), $Cd(SiF_3)(SiF_2)^+$ (2%), $Cd(SiF_3)^+$ (75%), Cd^+ (58%), and SiF_3^+ (100%).

(b) $(SiF_3)_2Cd{\cdot}glyme$. The reaction of $Cd(SiF_3)_2$ (99.4 mg) with glyme gave $(SiF_3)_2Cd{\cdot}glyme$. A reaction identical with that described above was carried out. After vacuum distillation of the unreacted Si_2F_6 from the reactor at −40 °C, 10 mL of ether and 1 mL of dimethoxyethane were condensed into the reactor. The reactor contents were warmed to room temperature and the volatile products were pumped from the reactor. The nonvolatile residue was removed from the reactor in a drybox and was extracted with methylene chloride to give a crude product. Crystallization of the impure product with hexane gave $(SiF_3)_2Cd{\cdot}glyme$ in high purity (36 mg, 11% yield based on metal vaporized). Bis(trifluorosilyl)cadmium·glyme is a light yellow solid which is stable in air for short periods of time and is stable indefinitely when stored in a nitrogen atmosphere. ^{19}F NMR (84.87 MHz, CD_2Cl_2) δ 98.7 (J-$(Cd^{113/111}\text{–}F)$ = 581/556 Hz); ^1H NMR (90 MHz, CD_2Cl_2) δ +3.7 (S, CH_2), +3.5 (S, CH_3); IR (KBr pellet) 2920 (br m), 1400 (m), 1250 (m), 1120 (w), 1070 (vs), 1005 (s), and 785 (m) cm^{-1}; mass spectrum, Cd-$(SiF_3)^+$ (9%), Cd·glyme$^+$ (1%), Cd^+ (15%), and SiF_3^+ (100%).

(c) $(SiF_3)_2Zn{\cdot}2pyridine$ and Zn $(SiF_3)_2$. Zinc metal (214 mg) was vaporized and cocondensed on a −196 °C surface with plasma-generated $\cdot SiF_3$ radicals. Following the 2-h reaction, the reactor was warmed to −40 ° C to remove the unreacted Si_2F_6. Ether (10 mL) and pyridine (1 mL) were condensed into the reactor bottom which was cooled with liquid nitrogen. Upon warming the reactor to room temperature, bis(trifluorosilyl)zinc·2pyridine formed. The excess pyridine and other volatile products were distilled from the reactor, leaving behind a nonvolatile residue which was extracted with methylene chloride in an inert atmosphere box and recrystallized with hexane to give $(SiF_3)_2Zn{\cdot}2pyridine$ (128 mg, 10% yield). The compound is a white tacky solid which does not appear to have a stable vapor phase. ^{19}F NMR (84.87 MHz, CD_2Cl_2) δ 99.3; ^1H NMR (90 MHz, CD_2Cl_2) δ 8.3 (ortho), 7.2 (meta), 7.6 (para).

The addition of 10 mL of methylene chloride to the reactor instead of the ether and pyridine as described above gave a solution of the unstable compound $Zn(SiF_3)_2$ which was shown to decompose at approximately −40 ° C. The methylene chloride solution was transferred from the reactor at −78 °C via a cannula into a chilled NMR tube. ^{19}F NMR (84.87 MHz, CD_2Cl_2) δ 122.9.

(d) $Zn(CF_3)_2$ and $Zn(CF_3)_2{\cdot}2pyridine$. Zinc metal (200 mg) was vaporized and condensed with trifluoromethyl radicals generated in a hexafluoroethane plasma in a 3-h reaction. Following the reaction, the cold finger was warmed allowing the matrix to drop into the base of the reactor which was held at −78 °C. While maintaining the −78 °C reactor temperature, the unreacted hexafluoroethane was vacuum-distilled from the reactor, leaving behind a residue which is believed to be unsolvated $Zn(CF_3)_2$. The addition of 10 mL of CH_2Cl_2 (−78 °C) to the reactor yielded a solution which was transferred via a cannula into a −78 °C NMR tube. The methylene chloride was pumped from the NMR tube at −78 °C and replaced with a small amount of CD_2Cl_2. ^{19}F NMR spectra suggest that $Zn(CF_3)_2$ begins to decompose at −40 °C. ^{19}F NMR (84.87 MHz, CD_2Cl_2) δ 40.5.

The addition of 10 mL of ether and 1 mL of pyridine to the reactor prior to warming to room temperature gave the known stable complex $(CF_3)_2Zn{\cdot}2pyridine$. The complex, obtained in 8% yield (88 mg), was purified by extraction of the nonvolatile residue with methylene chloride followed by recrystallization with hexane. ^{19}F NMR (84.87 MHz, CD_2Cl_2) δ 39.7; ^1H NMR (90 MHz, CD_2Cl_2) δ 8.5 (ortho), 7.4 (meta), 7.8 (para). IR agrees with the published spectrum.[8]

(e) $Cd(CF_3)_2$ and $(CF_3)_2Cd{\cdot}glyme$. Cadmium metal (200 mg) was placed in a quartz crucible and vaporized slowly over a 3-h period by resistively heating with a tungsten crucible heater. The metal was condensed with $\cdot CF_3$ radicals on a cryogenic surface. Following the reaction, the frozen matrix was dropped quickly from the cold finger (by warming with air) into the bottom of the reactor which was cooled with a dry ice/acetone slush (−78 °C). The unreacted C_2F_6 was vacuum-distilled from the reactor, leaving behind the unstable compound, $Cd(CF_3)_2$, along with unreacted cadmium metal as well as smaller amounts of fluoro-

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carbon catenation products. The $Cd(CF_3)_2$ was selectively dissolved in cold methylene chloride and transferred from the reactor into a chilled glass container (−45 °C) with a NMR tube attached to the bottom. The methylene chloride was vacuum-distilled from the container (−45 °C), leaving behind $Cd(CF_3)_2$ which was subsequently dissolved in CD_2Cl_2 (NMR solvent). The compound, which begins to decompose slowly at 0 °C, is obtained with a purity exceeding 99% (based on ^{19}F NMR). ^{19}F NMR (84.87 MHz, CD_2Cl_2) δ 38.5 (s, $J(Cd^{113/111}–F) = 543/508$ Hz).

In a separate experiment, glyme was condensed onto the cold finger following the reaction and prior to warming the cold finger. Upon warming, the $Cd(CF_3)_2$ complexed with the glyme, giving $(CF_3)_2Cd\cdot$glyme. The compound was isolated by first pumping the volatiles from the reactor followed by extraction of the nonvolatile residue with diethyl ether in an inert atmosphere. The complex was obtained in 11% yield (66 mg) based on metal vaporized and gave an IR and ^{19}F NMR identical with that obtained for an authentic sample prepared by a method described by Morrison.[9]

Discussion

Although the yields obtained were generally low, the merits of the metal vapor/plasma technique should be evident. Very unstable compounds such as $Cd(CF_3)_2$ and $Zn(CF_3)_2$ can be prepared, isolated, and studied with use of this technique. Equally important, complexes such as $Cd(SiF_3)_2\cdot$glyme and $Zn(SiF_3)_2\cdot$2pyridine can be uniquely prepared by using a metal vapor reactor. Although many other trifluorosilyl–metal complexes will eventually be made by using this technique, the cadmium and zinc complexes will probably provide an alternate route to these complexes by serving as trifluorosilyl transfer reagents in much the same way as $Cd(CF_3)_2\cdot$glyme and $Hg(CF_3)_2$ have been used to prepare trifluoromethyl-substituted compounds.[9,10]

Acknowledgment. We are grateful for support of this work by the Air Force Office of Scientific Research (AFOSR-82-0197), the National Science Foundation (CHE-8210708), and the Robert A. Welch Foundation (F-700).

Registry No. Cd, 7440-43-9; Zn, 7440-66-6; C_2F_6, 76-16-4; Si_2F_6, 13830-68-7; $Cd(SiF_3)_2$, 101834-99-5; $(SiF_3)_2Zn\cdot$2pyridine, 101835-02-3; $(SiF_3)_2Cd\cdot$glyme, 101835-01-2; $Zn(SiF_3)_2$, 101835-00-1; $Zn(CF_3)_2$, 70331-87-2; $Zn(CF_3)_2\cdot$2pyridine, 71672-49-6; $Cd(CF_3)_2$, 33327-66-1; $(CF_3)_2Cd\cdot$glyme, 76256-47-8.

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Reductive Alkylation of Aceheptylene—A Simple Route to a Series of Novel π-Perimeters

Gerd Neumann and Klaus Müllen*

Contribution from the Department of Organic Chemistry, University of Mainz, D.-6500 Mainz, J.J.Becher-Weg 18-20, FRG.

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Abstract: ^{1}H and ^{13}C NMR spectroscopic studies point out that the dianion of aceheptylene (**9**), in accordance with theoretical predictions, tends to localize the greatest portion of the excess charge at the inner angular carbon C-10b. The kinetically controlled addition of an electrophile allows one to attack this quaternary center. Thus, methylation readily affords the quench products **10**, **11**, **12a**, and **12b** in good yields. Deprotonation of **12a** and **12b** gives rise to strongly diatropic monocyclic ions, **13a** and **13b**, respectively, the first 14π-homologues of the cyclopentadienide ion. The monoanion **13a** can be submitted to a hydroxymethylation reaction followed by a solvolytic ring enlargement of the corresponding tosylate to yield the new annulene **15**. The dimethyl adducts **10** and **11**, which constitute linear 12π-systems, undergo a remarkable 12 + 2 cycloaddition with dimethyl acetylenedicarboxylate. The resulting diesters **19a** and **20a** transform into the ethanediylidene[14]annulenes **7** and **14**. Easily accessible quench products thus allow for a most straightforward synthesis of the novel perimeter systems **7**, **13**, **14**, and **15**. The spectroscopic characterization of these compounds sheds light on the subtle interdependence of steric and electronic effects in π-perimeters.

Toward an understanding of cyclic π-conjugation the annulenes have served as useful model compounds. Their structural elucidation however is severely complicated by the occurrence of configurational and conformational interconversions[1,2] as well as by steric interactions of hydrogens inside the ring.[3,4] A logical extension therefore was the construction of bridged annulenes[5–7] since they give rise to essentially planar rigid perimeters with well-defined ring configurations. A fascinating example is the family of [14]annulenes which comprises, e.g., Sondheimer's parent compound **1**,[8] Schröder[9] and Staab's[10] derivatives of **1**, Vogel's acene perimeters **2** and **3**,[11–17] Boekelheide and Mitchell's pyrene

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