Inorg. Chem. **1995,** *34,* 13−17 **13**

Articles

Per- and Polyfluoroaryl Mono- and Disiloxanes as Transfer Reagents in the Synthesis of Highly Fluorinated Mono- and Diethers

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Received July 12, 1994[⊗]

In the presence of fluoride ion, per- or polyfluoroaromatic siloxanes $C_6F_5OSi(CH_3)_3$ (**1**), $(CH_3)_3SiOC_6F_4OSi(CH_3)_3$ (**2**), 1-fluoro-2,3-bis((trimethylsilyl)oxy)benzene (**3**), and $(CH_3)_3SiOC(CF_3)_2C_6F_4C(CF_3)_2OSi(CH_3)_3$ (**4**) are useful transfer reagents for the synthesis of mono- and diethers. Siloxane **1** forms R_fOCF_2Br (**5**), $(R_fO)_3CF$ (**6**), $R_fOCH_2OR_f$ (**7**), $(R_fO)_2CHCH(OR_f)_2$ (**8**), $(R_fO)_2SO$ (**9**), $(R_fO)_2CO$ (**10**), $C_3N_3(OR_f)_3$ (**11**), $R_fOC(O)(CF_2)_3C(O)OR_f$ (**12**), $CF_3SO_2OR_f$ (**13**), $\overline{CF_2C(OR_f)=C(OR_f)CF_2}$ (**14**), and $\overline{CF_2C(OR_f)=C(F)CF_2}$ (**15**) ($R_f = C_6F_5$) with CF_2Br_2, $CFBr_3$, CH_2Br_2, $Br_2CHCHBr_2$, SOF_2, COF_2, $(CNF)_3$, $ClC(O)(CF_2)_3C(O)Cl$, CF_3SO_2F, 1,2-dichlorotetrafluorocyclobutene, and perfluorocyclobutene, respectively. Compound **5** is readily converted to $C_6F_5OCF_2Si(CH_3)_3$ (**16**) with hexaethylphosphorous triamide and $(CH_3)_3SiCl$ in benzonitrile. With C_6F_5CN, $CF_3C_6F_5$, C_5F_5N, CH_3I, perfluorocyclobutene, $CF_3C(O)Cl$ and $(CNF)_3$, **2** forms diethers *p*-$CNC_6F_4OC_6F_4OC_6F_4CN$-*p* (**17**), *p*-$CF_3C_6F_4OC_6F_4OC_6F_4CF_3$-*p* (**18**), $NC_5F_4OC_6F_4OC_5F_4N$ (**19**), $CH_3OC_6F_4OCH_3$ (**20**), and $C_2F_5C(O)OC_6F_4OC(O)C_2F_5$ (**21**), respectively. Reaction of **3** with 1,2-diiodotetrafluorobenzene in diglyme gives 1,4,8-trifluoro-2,3-diiodophenozine (**22**). Disiloxane **4** with $C_6H_5CH_2Br$, CH_3I, $C_6F_5CH_2Br$, and COF_2 results in $C_6H_5CH_2OC(CF_3)_2C_6F_4C(CF_3)_2OCH_2C_6H_5$ (**23**), $CH_3OC(CF_3)_2C_6F_4C(CF_3)_2OCH_3$ (**24**), $C_6F_5CH_2OC(CF_3)_2C_6F_4C(CF_3)_2OCH_2C_6F_5$ (**25**), and $\overline{C(O)OC(CF_3)_2C_6F_4C(CF_3)_2OC(O)OC(CF_3)_2C_6F_4C(CF_3)_2O}$ (**26**), respectively. These materials are thermally and hydrolytically stable and are formed in high yields.

Introduction

The utilization of silyl ethers as transfer reagents in the syntheses of polyfluorinated compounds has provided an exciting new route to a variety of heretofore difficultly accessible or unknown compounds.[1−7] Reactions that involve cleavage of the silicon−oxygen bond are not as common as the large number of reactions which require breaking of other silicon−element bonds.[9] Although in some cases extreme reaction conditions[10] or highly reactive reagents[11] are required, very often the silicon−oxygen bond is broken under mild conditions, e.g., with compounds containing P−F,[12] S−F,[13] M−F,[14−16] and C−F[7] bonds. In these cases advantage is taken of the formation of the very strong silicon−fluorine bond when R_3SiF is the nonether product that forms. The formation of a silicon−fluorine bond (~142 kcal/mol)[9] in the volatile byproducts (trimethylsilyl fluoride) of these reactions is the driving force for the high-yield formation of ethers under mild reaction conditions.

Earlier we reported the synthesis of acyclic mono- and disiloxane derivatives of various alcohols and their reactions with a number of halogenated compounds to prepare new cyclic and acyclic polyfluoro- and perfluoroethers.[3] In this work, we report the reactions of three previously synthesized perfluoroaromatic mono- and disiloxanes plus a new polyfluorodisiloxane. Very little reaction chemistry has been reported of the parent alcohols. If studied, it is usually through the use of the alkali salts. Although this method is widely used, these metal alkoxides are only moderately stable and hydrolytically unstable which limits their use. The siloxanes are very stable and can be stored at ambient temperature for many months without decomposition. The reactions are accomplished in the presence of a fluoride ion catalyst with a variety of per- and polyhalogenated compounds to form the respective ethers in high yields.

[⊗] Abstract published in *Advance ACS Abstracts,* December 1, 1994.

(1) Farnham, W. B. In *Synthetic Fluorine Chemistry*; Olah, G. A., Chambers, R. D., Prakash, G. K. S., Eds.; John Wiley & Sons, Inc.: New York, 1992; Chapter 11 and references therein.

(2) Zhang, Y.-F.; Kirchmeier, R. L.; Shreeve, J. M. *J. Fluorine Chem.* **1994,** *68,* 267.

(3) Patel, N. R.; Chen, J.; Zhang, Y.-F.; Kirchmeier, R. L.; Shreeve, J. M. *Inorg. Chem.* **1994,** *33,* 5463.

(4) Elias, A. J.; Kirchmeier, R. L.; Shreeve, J. M. *Inorg. Chem.* **1994,** *33,* 2727.

(5) Elias, A. J.; Hope, H.; Kirchmeier, R. L.; Shreeve, J. M. *Inorg. Chem.* **1994,** *33,* 415 and references therein.

(6) Guo, C.-Y.; Elias, A. J.; Kirchmeier, R. L.; Shreeve, J. M. Manuscript in preparation.

(7) Farnham, W. M.; Roe, D. C.; Dixon, D. A.; Calabrase, J. C. *J. Am. Chem. Soc.* **1990,** *112,* 7707.

(8) Fujita, M.; Hiyama, T. *J. Am. Chem. Soc.* **1985,** *107,* 4085. Fujita, M.; Obayashi, J.; Hiyama, T. *Tetrahedron* **1988,** *44,* 4135.

(9) Weber, W. P. *Silicon Reagents in Organic Synthesis;* Springer-Verlag: New York, 1983.

(10) Mascony, J. J.; MacDiarmid, A. G. *Chem. Commun.* **1965,** 307.

(11) Crans, C. D.; Felty, R. A.; Anderson, O. P.; Miller, M. M. *Inorg. Chem.* **1993,** *32,* 247.

(12) (a) Peake, S. C.; Schmutzler, R. *Chem. Commun.* **1968,** 665. (b) Peake, S. C.; Field, M.; Hewson, M. J. C.; Schmutzler, R. *Inorg. Chem.* **1971,** *10,* 2372. (c) Koop, H.; Schmutzler, R. J. *Fluorine Chem.* **1971/72,** *1,* 252. (d) Riess, J. G.; Robert, D. U. *Bull. Soc. Chim. Fr.* **1975,** *3,* 425. (e) Poulin, D. D.; Demay, C.; Riess, J. G. *Inorg. Chem.* **1977,** *16,* 2278. (f) Jeanneaux, F.; Riess, J. G. *Tetrahedron Lett.* **1978,** *48,* 4845.

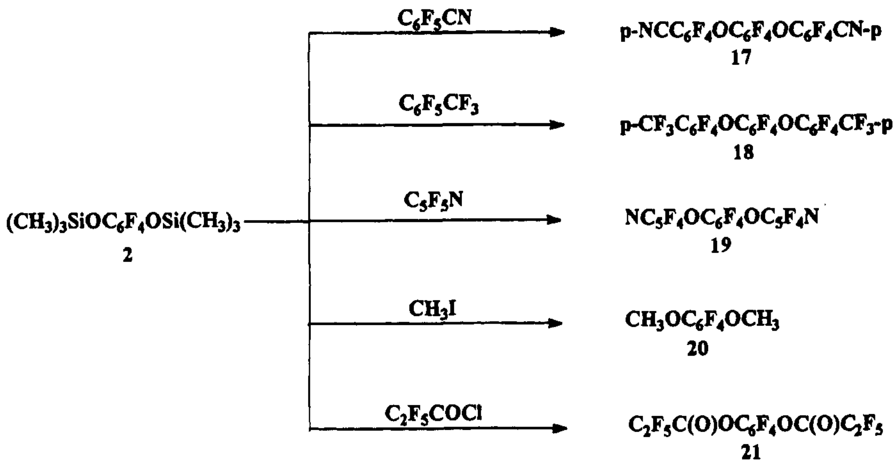
(13) Ykman, P.; Hall, H. K. *J. Organomet. Chem.* **1976,** *116,* 153.

(14) Walker, D. W.; Winfield, J. M. *J. Fluorine Chem.* **1971/72,** *1,* 493.

(15) Handy, L. B. *J. Fluorine Chem.* **1976,** *6,* 641.

(16) Singer, R. J.; Eisenhut, M.; Schmutzler, R. *J. Fluorine Chem.* **1971/72,** *1,* 193.

Scheme 1

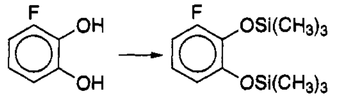


Results and Discussion

The siloxanes $C_6F_5OSi(CH_3)_3$ (**1**),[17] $(CH_3)_3SiOC_6F_4OSi(CH_3)_3$ (**2**),[18] and $FC_6H_3[OSi(CH_3)_3]_2$ (**3**) are prepared by refluxing the alcohols pentafluorophenol, 2,3,5,6-tetrafluorophenol, and 3-fluorocatechol with hexamethyldisilizane and a small amount ofsaccharin[2,5,13,18] at 80 °C.

$$C_6F_5OH \longrightarrow \underset{\mathbf{1}}{C_6F_5OSi(CH_3)_3}$$

$$HOC_6F_4OH + [(CH_3)_3Si]_2NH \longrightarrow \underset{\mathbf{2}}{(CH_3)SiOC_6F_4OSi(CH_3)_3}$$



3

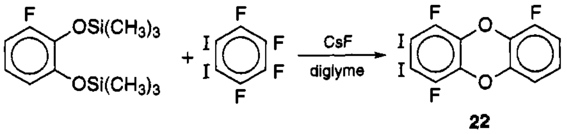
Small amounts of CsF catalyze the reactions of **1** with CF_2Br_2, $CFBr_3$, CH_2Br_2, $Br_2CHCHBr_2$, SOF_2, COF_2, $(CNF)_3$, $ClC(O)(CF_2)_3COCl$, and CF_3SO_2F to form the respective per- and polyfluorinated ethers $C_6F_5OCF_2Br$ (**5**), $(C_6F_5O)_3CF$ (**6**), $C_6F_5OCH_2OC_6F_5$(**7**), $(C_6F_5O)_2CHCH(OC_6F_5)_2$ (**8**), $(C_6F_5O)_2SO$ (**9**), $(C_6F_5O)_2CO$ (**10**),[19] $C_3N_3(OC_6F_5)_3$ (**11**), $C_6F_5OC(O)(CF_2)_3C(O)OC_6F_5$ (**12**), and $CF_3SO_2OC_6F_5$ (**13**).[20] The earlier synthesis of **10** and **13** was realized by reactions of KOC_6F_5 with COF_2 and CF_3SO_2F, the advantage of our method being the ease of handling the precursor siloxane **1**. All of the products are stable liquids and are formed in 50–75% yield. It is interesting to compare the relative nucleophilicities of **1** and $CF_3CH_2OSi(CH_3)_3$ in reaction with $CFBr_3$, where under identical conditions $(C_6F_5O)_3CF$ and $C(OCH_2CF_3)_4$, respectively, form.[3] It is assumed that **1** is slightly less nucleophilic, but steric considerations cannot be ruled out entirely. Both siloxanes produce analogous products with each of the other electrophiles.

With 1,2-dichlorotetrafluorocyclobutene in the presence of excess CsF, **1** reacts to form $\overline{CF_2C(OC_6F_5){=}C(OC_6F_5)CF_2}$ (**14**) and $\overline{CF_2C(OC_6F_5){=}C(F)CF_2}$ (**15**). When perfluorocyclobutene is mixed with **1** in a ratio of 1:2 in the presence of a catalytic amount of CsF, only a single product **14** is obtained. In every case, $(CH_3)_3SiF$ is found as a volatile product.

Reaction of $C_6F_5OCF_2Br$ (**5**) with hexaethylphosphorous triamide and chlorotrimethylsilane in benzonitrile as solvent gives the stable silane $C_6F_5OCF_2Si(CH_3)_3$ (**16**).[21] Studies on the reaction chemistry of this new volatile material are underway.

Disiloxane $(CH_3)_3SiOC_6F_4OSi(CH_3)_3$ (**2**) can be reacted smoothly with C_6F_5CN, $C_6F_5CF_3$, C_5F_5N, CH_3I, and $C_2F_5C(O)Cl$ in acetonitrile, THF, or diglyme in the presence of a trace of CsF to form a family of solid perfluoroaromatic diethers **17**-**21** (Scheme 1). Each of these materials melts cleanly without decomposition.

When disiloxane **3** is treated with 1,2-diiodotetrafluorobenzene at 70 °C, a partially fluorinated tricyclic diether results. This white crystalline material is extracted in high yield from the reaction mixture with diethyl ether.



22

The synthesis of $(CH_3)_3SiOC(CF_3)_2C_6F_4C(CF_3)_2OSi(CH_3)_3$ (**4**)[22] is accomplished by first lithiating tetrafluorobenzene with *n*-butyllithium and then by reaction of the dilithium salt with hexafluoroacetone and chlorotrimethylsilane,

$$LiC_6F_4Li + CF_3C(O)CF_3 + (CH_3)_3SiCl \xrightarrow{-78\text{ to }25\ ^\circ C} \underset{\mathbf{4}}{(CH_3)_3SiOC(CF_3)_2C_6F_4C(CF_3)_2OSi(CH_3)_3}$$

Reactions of **4** with several electrophiles in the presence of CsF as catalyst and diglyme as solvent give very high yields of diether derivatives of 1,4-bis(hexafluoroisopropyl)tetrafluorobenzene, *viz.*

$$\mathbf{4} + R_fX \xrightarrow{70\ ^\circ C} R_fOC(CF_3)_2C_6F_4C(CF_3)_2OR_f$$

$$R_f = C_6H_5CH_2\ (\mathbf{23}),\ CH_3\ (\mathbf{24}),\ C_6F_5CH_2\ (\mathbf{25});\ X = Br \text{ or } I$$

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(17) Oliver, A. J.; Graham, W. A. G. *J. Organomet. Chem.* **1969**, *19*, 17.

(18) Bruynes, C. A.; Jurriens, T. K. *J. Org. Chem.* **1982**, *47*, 3966.

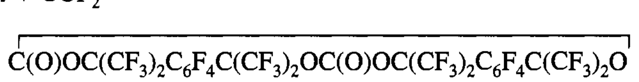
(19) DesMarteau, D. D.; Falardeau, E. R. *J. Fluorine Chem.* **1976**, *7*, 409.

(20) DesMarteau, D. D.; Falardeau, E. R. *J. Chem. Eng. Data* **1976**, *21*, 386.

(21) Ruppert, I.; Schlich, K.; Volbach, W. *Tetrahedron Lett.* **1984**, *25*, 2195.

(22) Tamborski, C.; Burton, W. H.; Breed, L. W. *J. Org. Chem.* **1966**, *31*, 4229.

$$\mathbf{4} + COF_2 \xrightarrow{25\ °C}$$



$$\overline{C(O)OC(CF_3)_2C_6F_4C(CF_3)_2OC(O)OC(CF_3)_2C_6F_4C(CF_3)_2O}$$

26

Compounds **23-25** are purified by recrystallization from heptane and are stable to temperatures above their melting points. While **26** is also stable well beyond its melting point, it is impossible to identify any solvent in which it is soluble. To date attempts to derivatize **26** have resulted in complete decomposition of the molecule. Mass spectral and elemental analysis support the proposed structure which is similar to those reported earlier.[1,23] Our work continues toward tailoring the structures of these mono- and diethers in order to ensure appropriate properties for a variety of applications where high temperature and hydrolytic stability and long liquid ranges are required.

Experimental Section

Materials. C_6F_5OH, HOC_6F_4OH, $CF_3C(O)CF_3$, C_6F_5CN, $CF_3C_6F_5$, C_5F_5N, C_2F_5COCl, $C_6F_5CH_2Br$, CF_2Br_2, COF_2, $ClCO(CF_2)_3COCl$, 1,2-dichlorotetrafluorocyclobutene and perfluorocyclobutene (PCR), *n*-BuLi, 3-fluorocatechol, CH_3I, $C_6H_5CH_2Br$, $CFBr_3$, CsF, $Br_2CHCHBr_2$, hexamethyldisilazane, hexaethylphosphorous triamide, chlorotrimethylsilane, benzonitrile (Aldrich), CH_2Br_2 (Eastman), CF_3SO_2F (gift from 3M Co.), and $(CNF)_3$ (Alfa) are used as received. SOF_2[24] and $C_6F_5(CF_3)_2COH$[22] are prepared by the literature methods.

General Procedures. A conventional vacuum system, comprised of a Pyrex glass vacuum line equipped with Heise Bourdon tube and Televac thermocouple gauges, is used to handle gases and volatile liquids. Reactions are performed in thick-walled 100-mL round-bottomed flasks with Teflon stopcocks. Products are separated and purified by distillation or extraction with ether followed by recrystallization. Infrared spectra are recorded on a Perkin-Elmer 1710 FTIR spectrometer equipped with an IBM PS-2 data station by using KBr plates for neat samples or with a 10 cm gas cell equipped with KBr disks. ^1H and ^{19}F NMR spectra are obtained with a Bruker AC200 Fourier transform NMR spectrometer by using $CDCl_3$ or CD_3CN as solvent and Me_4Si and CCl_3F as references. Mass spectra are obtained with a VG 7070HS GC/MS by using electron impact or chemical ionization techniques. Elemental analyses are performed by Beller Mikroanalytisches Laboratorium, Göttingen, Germany.

Preparation of Siloxanes 1-3. Siloxanes **1-3** are prepared by reaction of C_6F_5OH, HOC_6F_4OH, or 3-fluorocatechol (25 mmol) with $[(CH_3)_3Si]_2NH$ (30−60 mmol) in a 100-mL round-bottomed flask fitted with a reflux condenser. A small amount of sodium saccharin is added as catalyst to drive the reaction to completion.[18] After the initial evolution of ammonia subsides, the mixture is heated to 80 °C and maintained at that temperature for 8−10 h. Upon vacuum distillation, the siloxanes are obtained as colorless air stable liquids.

Properties of $C_6F_5OSi(CH_3)_3$ (1).[17] Infrared and proton NMR spectral data compared well with the literature. NMR: ^{19}F, δ −158.7 (*o*-F, 2F, m), −165.1 (*m*-F, 2F, m), −167.5 (*p*-F, 1F, m).

Properties of $(CH_3)_3SiOC_6F_4OSi(CH_3)_3$ (2).[18] Spectral data are as follows. IR (neat): 2957 s, 2899 m, 1510 s, 1412 w, 1315 m, 1256 s, 1182 s, 1056 s, 988 s, 935 s, 885 m, 846 s, 757 m, 687 m, 619 m cm^{-1}. NMR: ^{19}F, δ −160.3 (4F, s); ^1H, δ 0.26 (18H, s). CI MS [*m/e* (species) intensity]: 327 ($M^+ + 1$) 3.7; 326 (M^+) 12.6; 257 ($M^+ - CF - 2F$) 5; 236 ($M^+ - OSi(CH_3)_3 - H$) 1.2; 219 ($M^+ - OSi(CH_3)_3 - F + 1$) 3.7; 184 ($M^+ - Si(CH_3)_3 - CF - 2F$) 6.5; 164 ($C_6F_4O^+$) 7.5; 149 ($C_6F_4^+ + 1$) 20.5; 148 ($C_6F_4^+$) 100. Anal. Calcd for $C_{12}H_{18}F_4O_2Si_2$: C, 44.17; F, 23.31; H, 5.52. Found: C, 44.43; F, 23.5; H, 5.7.

Properties of 1-Fluoro-2,3-bis((trimethylsilyl)oxy)benzene (3). Spectral data are as follows. IR (neat): 2962 s, 2903 m, 1606 s, 1583 s, 1505 vs, 1471 m, 1412 m, 1306 s, 1254 vs, 1234 s, 1156 m, 1065 s, 1035 vs, 916 s, 844 vs, 800 m, 780 s, 760 s, 729 s, 716 m cm^{-1}. NMR: ^{19}F, δ −130.4 (CF, s); ^1H, δ 6.6 (aromatic CH, m), 0.24 (CH_3, s). CI MS [*m/e* (species) intensity]: 274 ($M^+ + 2$) 2.7; 273 ($M^+ + 1$) 12.4; 272 (M^+) 44.8; 257 ($M^+ - CH_3$) 7.1; 241 ($M^+ - CF$) 5.3; 183 ($M^+ - OSi(CH_3)_3$) 2.6; 179 ($M^+ - Si(CH_3)_3) - HF$) 2.5; 165 ($M^+ - Si(CH_3)_3) - F - CH_3$) 32.4; 151 ($M^+ - OSi(CH_3)_3) - F - CH$) 5; 137 ($M^+ - OSi(CH_3)_3) - CF - CH_3$) 2.8; 73 ($Si(CH_3)_3^+$) 100.

General Procedure for the Preparation of Ethers 5-17. In a typical reaction, 5 mL of acetonitrile, a catalytic amount of CsF, and siloxane **1-3** (6−10 mmol) are combined in a 100-mL round-bottomed Pyrex flask equipped with a Teflon stopcock. The mixture is cooled to −196 °C, and the vessel is evacuated. From 2−5 mmol of substrate (CF_2Br_2, $CFBr_3$, CH_2Br_2, $Br_2CHCHBr_2$, SOF_2, COF_2, $(CNF)_3$, $ClCO(CF_2)_3COCl$, CF_3SO_2F, 1,2-dichlorocyclobutene, or perfluorocyclobutene) is then condensed into the flask. The flask is warmed slowly to 25 °C, and the mixture is stirred for 10−12 h at 60−70 °C. The products are separated by low-temperature distillation or by extracting the residue with diethyl ether. Yields of the products **5-17** range from 50 to 75%.

Properties of $C_6F_5OCF_2Br$ (5). This compound is retained in a trap at −30 °C. Spectral data are as follows. IR (neat): 1528 s, 1218 m, 1142 s, 1081 m, 1049 m, 1003 m, 720 m cm^{-1}. NMR: ^{19}F, δ −19.2 (CF_2, t, $^5J_{F-F}$ = 9.8 Hz), −149.6 (*o*-F, 2F, m), −154.3 (*p*-F, 1F, m), −160.8 (*m*-F, 2F, m). CI MS [*m/e* (species) intensity]: 314/312 (M^+) 43.3/42.2; 295/293 ($M^+ - F$) 30.8/33.5; 233 ($M^+ - Br$) 100. Anal. Calcd for C_7F_7BrO: C, 26.92; F, 42.62. Found: C, 25.9; F, 43.2.

Properties of $(C_6F_5O)_3CF$ (6). This compound melts at 98 °C. Spectral data are as follows. IR (KBr disk): 1649 m, 1521 s, 1321 m, 1240 w, 1123 s, 1039 s, 998 s, 833 w, 726 m, 655 m cm^{-1}. NMR: ^{19}F, δ −15.1 (CF, t, $^5J_{F-F}$ = 15.0 Hz), −150.6 (*o*-F, 6F, m), −155.9 (*p*-F, 3F, m), −162.3 (*m*-F, 6F, m). CI MS [*m/e* (species) intensity]: 561 ($M^+ - F$) 3.9; 456 ($M^+ - C_4F_4$) 2.7; 397 ($M^+ - OC_6F_5$) 60.6; 378 ($M^+ - OC_6F_5 - F$) 3.6; 359 ($M^+ - OC_6F_5 - 2F$) 3.5; 331 ($C_9F_{10}O_2^+ + 1$) 1.9; 312 ($C_9F_9O_2^+ + 1$) 1; 293 ($C_9F_8O_2^+ + 1$) 52.4; 277 ($C_8F_7O_3^+$) 1.9; 262 ($C_8F_7O_2^+ + 1$) 0.5; 230 ($C_7F_6O_2^+$) 1.4; 214 ($C_7F_6O^+$) 1.5; 184 ($C_6F_5O^+ + 1$) 100. Anal. Calcd for $C_{19}F_{16}O_3$: C, 39.31; F, 52.4. Found: C, 40.00; F, 52.6.

Properties of $C_6F_5OCH_2OC_6F_5$ (7). This compound melts at 48 °C. Spectral data are as follows. IR (KBr disk): 2961 m, 1517 s, 1412 m, 1277 s, 1178 s, 1160 s, 1122 s, 1051 vs, 997 vs, 932 vs, 785 w, 745 w cm^{-1}. NMR: ^{19}F, δ −156.2 (*o*-F, 4F, m), −160.8 (*p*-F, 2F, m), −162.5 (*m*-F, 4F, m); ^1H, δ 5.76 (CH_2, s). CI MS [*m/e* (species) intensity]: 380 (M^+) 8.3; 197 ($M^+ - OC_6F_5$) 100. Anal. Calcd for $C_{13}H_2F_{10}O_2$: C, 41.05; F, 50.0. Found: C, 40.9; F, 50.5.

Properties of $(C_6F_5O)_2CHCH(OC_6F_5)_2$ (8). This compound melts at 120 °C. Spectral data are as follows. IR (KBr disk): 1526 vs, 1501 vs, 1468 m, 1365 s, 1289 s, 1244 m, 1149 s, 1134 s, 998 s, 978 vs, 714 w, 608 w cm^{-1}. NMR: ^{19}F, δ −164.7 (*o*-F, 8F, m), −168.4 (*m*-F, 8F, m), −178.2 (*p*-F, 4F, m); ^1H, δ 4.8 (CH, s). CI MS [*m/e* (species) intensity]: 738 ($M^+ - HF$) 3.5; 630 ($M^+ - 2HF - 2F - CF_2$) 1.8; 603 ($M^+ - C_5F_5$) 1.4; 575 ($M^+ - OC_6F_5$) 1.4; 572 ($M^+ - C_6F_5 - F$) 1; 553 ($M^+ - C_6F_5 - 2F$) 2.4; 541 ($M^+ - C_6F_5 - CF_2$) 1.4; 489 ($M^+ - C_6F_5 - 2HF - 2CF$) 2.6; 451 ($M^+ - OC_6F_5 - C_4F_4$) 5.2; 450 ($M^+ - C_6F_5OH - C_4F_4$) 50.1; 440 ($M^+ - C_6F_5 - 3CF - HF - 2F$) 13.9; 436 ($M^+ - C_6F_5 - C_5F_5$) 2.9; 420 ($M^+ - OC_6F_5 - C_5F_5$) 76.0; 413 ($M^+ - OC_6F_5 - C_4F_4 - 2F$) 6; 378 ($C_{13}F_{10}O_2^+$) 1.1; 329 ($C_{12}F_8O_2^+ + 1$) 3.4; 298 ($C_{11}F_7O_2^+ + 1$) 12.8; 290 ($C_{12}F_6O_2^+$) 11; 285 ($C_{10}F_7O_2^+$) 34.6; 225 ($C_8F_5O_2H_2^+$) 3.9; 216 ($C_9F_4O_2^+$) 4; 209 ($C_8F_5H_2O^+ + 1$) 2.6; 207 ($C_8F_5O^+$) 2.6; 197 ($C_7F_5H_2O^+$) 4.6; 195 ($C_7F_5O^+$) 2.3; 183 ($C_6F_5O^+$) 100. Anal. Calcd for $C_{26}H_2F_{20}O_4$: C, 41.17; F, 50.13. Found: C, 41.50; F, 50.78.

Properties of $(C_6F_5O)_2SO$ (9). This compound melts with decomposition at 95 °C. Spectral data obtained are as follows. IR (KBr disk): 1520 vs, 1480 s, 1385 m, 1315 m, 1190 m, 1029 s, 999 s, 834 m, 739 m, 690 m cm^{-1}. NMR: ^{19}F, δ −157.5 (*o*-F, 4F, m), −163.2 (*m*- F, 4F, m), −165.0 (*p*-F, 2F, m). CI MS [*m/e* (species) intensity]: 414 (M^+) 1.8; 395 ($M^+ - F$) 1.2; 358 ($M^+ - 3F + 1$) 2.7; 296 ($M^+ - C_2F_5 + 1$) 1.2; 291 ($M^+ - C_4F_4 + 1$) 1.2; 272 ($M^+ - C_4F_5 + 1$) 2.7; 253 ($C_8F_4SO_3^+ + 1$) 3.5; 241 ($C_7F_4SO_3^+ + 1$) 2.8; 231 ($M^+ - OC_6F_5$) 1.2; 224 ($C_7F_4SO_2^+$) 1.2; 217 ($C_5F_4SO_3^+ + 1$) 1.2; 200 ($C_5F_4SO_2^+$) 3.7; 197 ($C_6F_4SO^+ + 1$) 3.5; 196 ($C_6F_4SO^+$) 1.9; 184 ($C_6F_5O^+$

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(23) Guo, C.-Y.; Kirchmeier, R. L.; Shreeve, J. M. *J. Am. Chem. Soc.* **1991**, *113*, 9000.

(24) Tullock, C. W.; Coffman, D. D. *J. Org. Chem.* **1960**, *25*, 2016.

+ 1) 100. Anal. Calcd for $C_{12}F_{10}O_3S$: C, 34.78; F, 45.89. Found: C, 35.23; F, 46.16.

Properties of $(C_6F_5O)_2CO$ (10).[19] This compound melts at 50 °C. Infrared and mass spectral data compare favorably with the literature. Previously unreported spectral data are as follows. NMR: ^{19}F, −166.7 (*o*-F, 4F, m), −169.9 (*m*-F, 4F, m), −183.7 (*p*-F, 2F, m). CI MS [*m/e* (species) intensity]: 394 (M^+) 7; 375 ($M^+ - F$) 5.5.

Properties of $C_3N_3(OC_6F_5)_3$ (11). Spectral data are as follows. IR (KBr disk): 1612 s, 1576 m, 1520 s, 1480 m, 1441 w, 1392 s, 1305 s, 1176 s, 1160 s, 1096 s, 998 s, 960 m, 810 m cm^{-1}. NMR: ^{19}F, δ −153.6 (*o*-F, 6F, m), −157.7 (*p*-F, 3F, m), −163.0 (*m*-F, 6F, m). CI MS [*m/e* (species) intensity]: 628 ($M^+ + 1$) 19.4; 478 ($M^+ - C_3F_6 + 1$) 4.4; 465 ($M^+ - C_4F_6$) 4.4; 445 ($M^+ - OC_6F_5 +1$) 7.1; 444 ($M^+ - OC_6F_5$) 31.6; 418 ($M^+ - OC_6F_5 - CN$) 3.5; 374 ($C_{13}F_9NO_2^+ + 1$) 5.1; 311 ($C_{11}F_7NO_2^+$) 7.6; 295 ($C_{10}F_6N_2O_2^+ + 1$) 24.9; 281 ($C_{10}F_6NO_2^+ + 1$) 3.7; 280 ($C_{10}F_6NO_2^+$) 36; 252 ($C_8F_5N_2O_2^+ + 1$) 2.2; 235 ($C_8F_5N_2O^+$) 10.3; 223 ($C_7F_5N_2O^+$) 5.4; 210 ($C_7F_5NO^+ + 1$) 10.4; 209 ($C_6F_5OCN^+$) 55.2; 195 ($C_6F_5OC^+$) 1.2; 184 ($C_6F_5O^+ + 1$) 100.

Properties of $C_6F_5OC(O)(CF_2)_3C(O)OC_6F_5$ (12). This compound melts at 92−94 °C. Spectral data are as follows. IR (KBr disk): 1718 s, 1684 s, 1527 s, 1501 s, 1469 s, 1387 m, 1283 m, 1166 vs, 1073 m, 996 vs, 884 m, 813 m, 748 w, 714 w cm^{-1}. NMR: ^{19}F, δ −123.7 (CF_2, 4F, s), −128.6 (CF_2, 2F, s), −168.9 (*o*-F, 4F, m), −172.5 (*m*-F, 4F, m), −182.5 (*p*-F, 2F, m). CI MS [*m/e* (species) intensity]: 389 ($M^+ - OC_6F_5$) 10.5; 239 ($M^+ - 2C_6F_5 + 1$) 60.2; 222 ($M^+ - OC_6F_5 - C_6F_5$) 12.7; 195 ($C_4F_6O_2^+ + 1$) 8.5; 184 ($C_6F_5O^+ + 1$) 100.

Properties of $CF_3SO_2OC_6F_5$ (13).[20] Infrared and NMR spectral data compare favorably with the literature data. CI MS: shows M^+ and $M^+ + 15$.

Properties of $\overline{CF_2C(OC_6F_5)=C(OC_6F_5)CF_2}$ (14). This compound melts at 74 °C. Spectral data are as follows. IR (KBr disk): 1751 s, 1521 s, 1477 s, 1376 s, 1359 s, 1318 s, 1256 w, 1125 s, 1028 s, 1001 s, 946 m, 883 m, 601 w cm^{-1}. NMR: ^{19}F, δ -116.7 (CF_2, 4F, s), −154.4 (*o*-F, 4F, m), −155.4 (*p*-F, 2F, m), −160.4 (*m*-F, 4F, m). CI MS [*m/e* (species) intensity]: 491 ($M^+ + 1$) 12.3; 490 (M^+) 73; 471 ($M^+ - F$) 45.9; 421 ($M^+ - CF_3$) 1.4; 307 ($M^+ - OC_6F_5$) 8.6; 279 ($C_{11}F_6O_2^+ + 1$) 3.8; 273 ($M^+ - C_6F_5 - CF_2$) 5; 257 ($M^+ - OC_6F_5$ $-CF_2$) 40.1; 229 ($C_{10}F_4O_2^+ + 1$) 43.7; 195 ($C_6F_5OC^+$) 10.9; 184 ($C_6F_5O^+ + 1$) 33.5; 183 ($C_6F_5O^+$) 26.4; 167 ($C_6F_5^+$) 19.8; 155 ($C_5F_5^+$) 15.6; 149 ($C_6F_4^+ + 1$) 100. Anal. Calcd for $C_{16}F_{14}O_2$: C, 39.18; F, 54.29. Found: C, 38.89; F, 55.01.

Properties of $\overline{CF_2C(OC_6F_5)=C(F)CF_2}$ (15). Spectral data are as follows. IR (neat): 1770 s, 1527 vs, 1445 m, 1376 vs, 1316 m, 1268 w, 1157 s, 1137 s, 1026 vs, 1015 s, 1004 s, 983 m, 941 m, 919 m, 874 w, 750 w cm^{-1}. NMR: ^{19}F, δ −117.4 (CF_2, 2F, m), −118.9 (CF_2, 2F, m), −132.3 (CF, 1F, m), −154.2 (*o*-F, 2F, m), −155.0 (*p*-F, 1F, m), −160.0 (*m*-CF, 2F, m). CI MS [*m/e* (species) intensity]: 326 (M^+) 14.8; 307 ($M^+ - F$) 6.9; 231 ($C_{10}F_5O^+$) 4.1; 219 ($C_9F_5O^+$) 3; 184 ($C_6F_5O^+ + 1$) 100.

Preparation and Properties of $C_6F_5OCF_2Si(CH_3)_3$ (16). In a 100-mL round-bottomed flask equipped with a Teflon stopcock are combined $C_6F_5OCF_2Br$ (5 mmol), chlorotrimethylsilane (5 mmol), hexaethylphosphorous triamide (5 mmol), and 5 mL of benzonitrile. The flask is kept at −196 °C, and the air is evacuated. It is allowed to warm slowly to 25 °C and the mixture is stirred for 12−14 h. The product **16** is subsequently isolated in about 55% yield in a trap cooled at −30 °C via low temperature distillation. Spectral data are as follows. IR (neat): 2970 m, 1520 vs, 1258 m, 1206 s, 1041 m, 1009 s, 975 m, 927 w, 854 s, 759 s cm^{-1}. NMR: ^{19}F, δ −75.93 (CF_2, t, $^5J_{F-F} = 9.97$ Hz), −151.4 (*o*-F, 2F, m), −158.4 (*p*-F, 1F, m), −163.1 (*m*-F, 2F, m); ^{1}H, δ 0.29 (CH_3Si, s). CI MS [*m/e* (species) intensity]: 287 ($M^+ - F$) 4.2; 256 ($M^+ - CF_2$) 13.2; 234 ($M^+ - (CH_3)_3Si + 1$) 1.2; 215 ($M^+ - (CH_3)_3SiF + 1$) 1.0; 184 ($C_6F_5O^+ + 1$) 2.8; 167 ($C_6F_5^+$) 4.7; 148 ($C_6F_4^+$) 1.1; 123 ($M^+ - OC_6F_5$) 5.4; 118 ($C_5F_3^+ + 1$) 18.3; 117 ($C_5F_3^+$) 2; 105 ($C_4F_3^+$) 7.6; 104 ($C_4H_9SiF^+$) 88.2; 103 ($C_4H_8SiF^+$) 100.

Preparation of Ethers 17-21. Siloxane **2** (8−10 mmol), 5 mL of actonitrile or THF or diglyme, and a catalytic amount of CsF are combined in a 100-mL round-bottomed Pyrex flask equipped with a Teflon stopcock. The mixture is cooled at −196 °C, and the vessel is evacuated. A 5−10 mmol amount of substrate (CNC_6F_5, $CF_3C_6F_5$, C_5F_5N, CH_3I, perfluorocyclobutene, or C_2F_5COCl) is then added to the flask. The flask is allowed to warm to 25 °C, and the mixture is stirred for 10−12 h at 60−70 °C. The products are separated by trap-to-trap distillation or by extracting the residue with diethyl ether. Product yields range from 50 to 70%.

Properties of *p*-$CNC_6F_4OC_6F_4OC_6F_4CN$-*p* (17). This compound melts at 154−156 °C. Spectral data are as follows. IR (KBr disk): 2250 m, 1652 m, 1504 vs, 1439 m, 1322 m, 1304 m, 1127 s, 994 vs, 921 m, 652 m cm^{-1}. NMR: ^{19}F, δ −133.16 (*o*-F to CN, 4F, m), −154.3 (*m*-F to CN, 4F, m), −155.9 (OC_6F_4O, 4F, m). CI MS [*m/e* (species) intensity]: 557 ($M^+ + 29$) 5.1; 543 ($M^+ + 15$) 20.7; 530 ($M^+ + 2$) 20.6; 529 ($M^+ + 1$) 100; 528 (M^+) 94.9.

Properties of *p*-$CF_3C_6F_4OC_6F_4OC_6F_4CF_3$-*p* (18). This compound melts at 110 °C. Spectral data are as follows. IR (KBr disk): 1660 m, 1515 vs, 1499 vs, 1427 m, 1402 w, 1351 s, 1228 s, 1192 s, 1163 s, 1112 m, 1087 m, 1017 s, 986 s, 969 s, 877 m, 720 m cm^{-1}. NMR: ^{19}F, δ −55.8 (CF_3, 6F, m), −141.6 (*o*-F to CF_3, 4F, m), −155.6 (*m*-F to CF_3, 4F, m), −164.2 (OC_6F_4O, 4F, m). EI MS [*m/e* (species) intensity]: 614 (M^+) 100; 595 ($M^+ - F$) 22.5; 545 ($M^+ - CF_3$) 1.7; 397 ($M^+ - C_6F_4CF_3$) 44.9; 381 ($M^+ - OC_6F_4CF_3$) 3.4.

Properties of $NC_5F_4OC_6F_4OC_5F_4N$ (19). This compound melts at 130 °C. Spectral data are as follows. IR (KBr disk): 1646 s, 1505 vs, 1414 m, 1308 s, 1274 m, 1117 m, 1079 vs, 1005 s, 976 s, 954 m, 734 w, 697 m, 645 m, 618 m cm^{-1}. NMR: ^{19}F, -89.0 (*o*-F to N, 4F, m), −154.2 (*m*-F to N, 4F, m), −156.5 (OC_6F_4O, 4F, m). EI MS [*m/e* (species) intensity]: 480 (M^+) 100; 461 ($M^+ - F$) 3.5; 330 ($M^+ - NC_5F_4$) 51.7; 314 ($M^+ - OC_5F_4N$) 3.0. Anal. Calcd for $C_{16}F_{12}N_2O_2$: C, 40.0; F, 47.5. Found: C, 40.8; F, 48.1.

Properties of $CH_3OC_6F_4OCH_3$ (20). This compound melts at 52 °C. Spectral data are as follows. IR (KBr disk): 2956 m, 2844 w, 1507 vs, 1435 s, 1303 s, 1194 m, 1062 vs, 996 s, 906 s, 705 w cm^{-1}. NMR: ^{19}F, δ −159.1 (4F, s); ^{1}H, δ 3.97 (6H, s). CI MS [*m/e* (species) intensity]: 211 ($M^+ + 1$) 5.3; 210 (M^+) 66.5; 195 ($M^+ - CH_3$) 100.

Properties of $C_2F_5C(O)OC_6F_4OC(O)C_2F_5$ (21). This compound melts at 162 °C. Spectral data are as follows. IR (KBr disk): 1772 s, 1525 s, 1512 s, 1455 w, 1304 m, 1222 vs, 1161 vs, 1030 s, 999 m, 967 m, 847 w, 783 w, 707 m cm^{-1}. NMR: ^{19}F, δ −83.4 (CF_3, 6F, s), −122.2 (CF_2, 4F, s), −165.0 (C_6F_4, 4F, s). CI MS [*m/e* (species) intensity]: 474 (M^+) 2.8; 355 ($M^+ - C_2F_5$) 5.8; 311 ($M^+ - C_2F_5CO_2$) 3.1; 236 ($M^+ - C_4F_{10}$) 8.1; 211 ($C_7F_5O_2^+$) 1.9; 192 ($C_6F_4CO_2^+$) 4.1; 183 ($C_6F_5O^+$) 100. Anal. Calcd for $C_{12}F_{14}O_4$: C, 30.37; F, 56.11. Found: C, 31.1; F, 55.8.

Preparation and Properties of 1,4,8-Trifluoro-2,3-diiodophenozine (22). In a typical reaction, 5 mL of triglyme, a catalytic amount of CsF, siloxane **3** (5 mmol), and 1,2-diiodotetrafluorobenzene (5 mmol) are combined in a 100-mL round-bottomed flask equipped with Teflon stopcock. The mixture is cooled at −196 °C, and the vessel is evacuated. The flask is warmed to 25 °C, and the mixture is stirred for 10−12 h at 70−75 °C. After the reaction all the volatile compounds are removed and the residue on extraction with ether gives compound **22** in high yield. Spectral data are as follows. IR (KBr): 3093 m, 2980 w, 1623 m, 1583 m, 1500 s, 1469 vs, 1440 s, 1323 m, 1287 m, 1253 m, 1195 s, 1157 s, 1106 m, 1064 s, 1039 s, 869 w, 848 m, 833 s, 811 w, 777 w, 689 m cm^{-1}. NMR: ^{19}F, δ −106.5 (2F, ortho to iodo, m), −135.3 (1F, CF, m); ^{1}H, δ 6.8 (aromatic protons, m). CI MS [*m/e* (species) intensity]: 491 ($M^+ + 1$) 9.5; 490 (M^+) 100.

Preparation of $(CH_3)_3SiOC(CF_3)_2C_6F_4C(CF_3)_2OSi(CH_3)_3$ (4). Into a 100-mL three-necked flask equipped with magnetic stirrer, reflux condenser, and a nitrogen inlet is added 20 mmol of 2,3,5,6-tetrafluorobenzene and 15 mL of dry THF. The solution is cooled to −60 °C, and 20 mL of BuLi in hexane is added dropwise under a nitrogen atmosphere. The reaction mixture is stirred at this temperature for 2 h, and 40 mmol of hexafluoroacetone is condensed into the solution. Stirring is continued while the reaction temperature is allowed to raise gradually to 25 °C. The resulting lithium salt is trapped by chlorotrimethylsilane to give the disiloxane in 33% yield, mp 66−68 °C (heptane). Spectral data are as follows. IR (KBr pellet): 2966, w, 1612, m, 1495, m, 1219, s, 1126, s, 1046, s, 983, s, 931, s, 852, s, 764, s, 714, s cm^{-1}. NMR: ^{19}F, δ −74.3 (12F, s, CF_3), −132.5 (4F, m, Ar-F); ^{1}H δ 0.18 ($SiCH_3$) ppm. CI MS [*m/e* (species) intensity]: 611 ($M^+ - CH_3$) 5.0, 515 ($M^+ - (CH_3)_3SiF - F$) 4.2, 487 ($M^+ - (CH_3)_3SiO - CF_2$) 4.3, 404 ($C_{12}F_{12}O_2^+$) 4.2, 149 ($C_6F_4^+ + 1$) 11.0, 85 (SiF_3^+) 15.8, 81 ($CH_3SiF_2^+$) 31.1, 77 $(CH_3)_2SiF^+$) 100.

General Procedure for the Preparation of Compounds 23-25. In a typical procedure, 4.5 mmol of CsF, 3 mmol of **4**, 3 mL of diglyme, and 1.5−3 mmol of substrate ($C_6H_5CH_2Br$, CH_3I, $C_6F_5CH_2Br$) are added under dry nitrogen atmosphere to a oven-dried 50-mL Pyrex round-bottomed flask equipped with a magnetic stirrer. The mixture is stirred at 25 °C for 8 h. The product is extracted with 3 × 20 mL of ether. The extract is washed with water several times and dried over $MgSO_4$. Evaporation of the solvent gives the crude products, which are recrystallized from heptane to yield white crystals.

Properties of $C_6H_5CH_2OC(CF_3)_2C_6F_4C(CF_3)_2OCH_2C_6H_5$ (23). This compound which melts at 132−134 °C is recrystallized from heptane (96.2% yield). Spectral data are as follows. IR (film): 3079 w, 3072 w, 2966 m, 2908 m, 1473 s, 1428 s, 1397 s, 1290 s , 1236 s, 1169 s, 1133 s, 1023 s, 993 s, 958 s, 781 s, 747 s, 737 s, 725 m cm^{-1}. NMR: ^{19}F, δ −71.3 (12F, s, CF_3), −132.2 (4F, m); ^1H, δ 7.37 (10H, m, C_6H_5), 4.68 (4H, s, CH_2). CI MS [*m/e* (species) intensity]: 663 ($M^+ + 1$) 0.47; 662 (M^+) 1.9; 536 ($M^+ - C_6H_5CH_2O - F$) 2.0; 448 ($M^+ - 2C_6H_5CH_2O$) 0.8; 256 ($C_6H_5CHOC(CF_3)_2{}^+$) 1.5; 187 ($C_6H_5CHOC(CF_3))^+$) 1.5; 107 ($C_6H_5CH_2O^+$) 49.8; 106 ($C_6H_5CHO^+$) 32.5; 105 ($C_6H_5CO^+$) 17.4; 91 ($C_6H_5CH_2+$) 100.

Properties of $CH_3OC(CF_3)_2C_6F_4C(CF_3)_2OCH_3$ (24). This compound which melts at 110−112 °C is recrystallized from heptane (94.1% yield). Spectral data are as follows. IR (film): 3079 w, 3072 w, 2966 m, 2908 m, 1473 s, 1428 s, 1397 s, 1290 s, 1236 s, 1169 s, 1133 s, 1023 s, 993 s, 958 s, 781 s, 747 s, 737 s, 725 m cm^{-1}. NMR: ^{19}F, δ −72.0 (12F, s, CF_3), −133.2 (4F, m); ^1H, δ 3.55 (6H, s, CH_3). CI MS [*m/e* (species) intensity]: 511 ($M^+ + 1$) 3.0; 510 (M^+) 24.4; 491 ($M^+ - F$) 14.9; 441 ($M^+ - CF_3$) 100.

Properties of $C_6F_5CH_2OC(CF_3)_2C_6F_4C(CF_3)_2OCH_2C_6F_5$ (25). This compound which melts at 158−160 °C is recrystallized from heptane (93.4% yield). Spectral data are as follows. IR (film): 3079 w, 3072 w, 2966 m, 2908 m, 1473 s, 1428 s, 1397 s, 1290 s, 1236 s, 1169 s, 1133 s, 1023 s, 993 s, 958 s, 781 s, 747 s, 737 s, 725 m cm^{-1}. NMR: ^{19}F, δ −71.4 (12F, s, CF_3), −131.7 (4F, m), −142.1 (4F, m, *o*-F), −151.0 (2F, m, *p*-F), −161.1 (4F, m, *m*-F); ^1H, δ 4.81 (4H, s, CH_2). CI MS [*m/e* (species) intensity]: 843 ($M^+ + 1$) 4.3; 842 (M^+) 14.4; 662 ($M^+ - C_6F_5CH_2 + 1$) 1.4; 646 ($M^+ - C_6F_5CH_2O + 1$) 5.9; 645 ($M^+ - C_6F_5CH_2O$) 16.9; 450 ($M^+ - 2C_6F_5CH_2 0 + 2$) 16.9; 197 ($C_6F_5CH_2O^+$) 49.6; 196 ($C_6F_5CHO^+$) 23.6; 182 ($C_6F_5CH_2{}^+ + 1$) 56.4; 181 ($C_6F_5CH_2{}^+$) 100.

Preparation and Properties of $C(O)OC(CF_3)_2C_6F_4C(CF_3)_2OC(O)OC(CF_3)_2C_6F_4C(CF_3)_2O$ (26). This compound is prepared by the same procedure as described above except that substrate COF_2 is introduced into the reaction vessel under vacuum. After the reaction, the mixture is poured into water and the white solid which precipitates is washed several times with distilled water and dried (100% yield). This solid which melts from 200−210 °C does not dissolve in any solvent tried. Spectral data are as follows. IR (film): 1867 w, 1813 s, 1489 s, 1251 s , 1062 s, 1045 s, 1000 s, 962 s, 772 s, 724 s cm^{-1}. CI MS [*m/e* (species) intensity]: 1017 ($M^+ + 1$) 1.7; 1016 (M^+) 0.3; 998 ($M^+ - F + 1$) 2.1; 997 ($M^+ - F$) 8.4; 973 ($M^+ - CO_2 + 1$) 23.2; 953 ($M^+ - CO_2 - F$) 27.1; 509 ($^1/_2M^+ + 1$) 12.2; 489 ($^1/_2M^+ - F$) 22.7; 465 ($^1/_2M^+ - CO_2 + 1$) 100. Anal. Calcd for $C_{26}F_{32}O_6$: C, 30.73; F, 59.8. Found: C, 30.51; F, 59.1.

Acknowledgment. We are grateful to the National Science Foundation (Grants CHE-9310389 and OSR-9350539 (R.L.K. and J.C.)) and the Air Force Office of Scientific Research (Grant 91-0189) for support of this research. We also thank Dr. Gary Knerr for obtaining the mass spectral data.

IC940817R