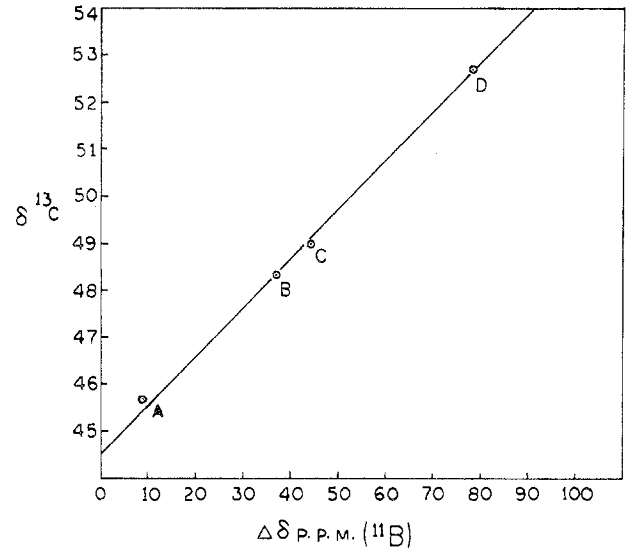

Figure 2. Plot of ^{13}C chemical shifts *vs.* $\Delta\delta(^{11}B)$ values for trimethylamine-borane adducts: A, $(CH_3)_3N{\cdot}BF_3$; B, $(CH_3)_3N{\cdot}BCl_3$; C, $(CH_3)_3N{\cdot}BBr_3$; D, $(CH_3)_3N{\cdot}BH_3$.

of such data especially when comparing adjacent members of the series, but there is nevertheless a clear suggestion the BH_3 adduct is significantly stronger than the fluoroborane adducts. Other workers[15,16] have accounted for the apparent weakening effect of substituting fluorine on borane Lewis acids by considering a competition between π lone pairs on fluorine and the σ-electron pair of the Lewis base for the boron acceptor orbital tending to weaken fluoroborane acceptors. If this effect is operative here, it might weaken the fluoroborane adducts relative to $(CH_3)_3N{\cdot}BH_3$, but alone, it does not account for the ordering of the entire series.

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Registry No. $(CH_3)_3N{\cdot}BH_3$, 75-22-9; $(CH_3)_3N{\cdot}BH_2F$, 35449-24-2; $(CH_3)_3N{\cdot}BHF_2$, 35237-72-0; $(CH_3)_3N{\cdot}BF_3$, 420-20-2; $(CH_3)_3N{\cdot}BCl_3$, 1516-55-8; $(CH_3)_3N{\cdot}BBr_3$, 1516-54-7; ^{13}C, 14762-74-4; ^{11}B, 14798-13-1.

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The New Compound Silylbis(trifluoromethyl)phosphine and Some Phosphorus–Silicon Bond Cleavage Reactions

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Compounds of the type $(CF_3)_2PSiR_3$ might be expected to serve as useful reagents for making other new $(CF_3)_2P$– and R_3Si– compounds, because the P–Si bond is relatively easy to cleave. The first work on this subject concerned $(CF_3)_2PSi(CH_3)_3$, which was found to react easily with HBr to make $(CF_3)_2PH$ and $(CH_3)_3SiBr$ or with CH_3I to make $(CF_3)_2PCH_3$ and $(CH_3)_3SiI$.[1]

We now have made $(CF_3)_2PSi(CH_3)_3$ and the new compound $(CF_3)_2PSiH_3$ by a relatively direct and highly efficient method:

$$(CF_3)_2PI + ISiH_3 + 2Hg \rightarrow (CF_3)_2PSiH_3 + Hg_2I_2 \qquad (1)$$

$$(CF_3)_2PI + ISi(CH_3)_3 + 2Hg \rightarrow (CF_3)_2PSi(CH_3)_3 + Hg_2I_2 \qquad (2)$$

However, our explorations of their P–Si bond cleavage reactions have been aimed at producing unusual and unstable new $(CF_3)_2PX$ compounds, so that the usual result was a variety of more stable products, often to be ascribed to decomposition of the desired cleavage products. Especially interesting and relatively well studied was a BF_3 cleavage, the first stage of which probably was

$$(CF_3)_2PSiH_3 + BF_3 \rightarrow SiH_3F + (CF_3)_2PBF_2 \qquad (3)$$

However, no $(CF_3)_2PBF_2$ could be isolated; indeed, this unknown compound was sought in vain some years ago in many experiments by our erstwhile colleague Dr. Andrew P. Lane, who concluded that its only possible synthesis would be at low temperatures, such that its decomposition might not be inevitable.

Instead of $(CF_3)_2PBF_2$, then, the reaction gave a 47% yield of the interesting ring compound $(CF_3PCF_2)_2$,[2] along with much H_2SiF_2, nonvolatiles, and traces of H_2, B_2H_6, $HSiF_3$, $(CF_3)_2PH$, and $P_2(CF_3)_4$. It is suggested that much bond energy will be gained if $(CF_3)_2PBF_2$ transfers one F from C to form BF_3, with dimerization of the resulting CF_3PCF_2 unit; indeed, something similar must have happened when $(CF_3PCF_2)_2$ was discovered in an attempt to make an $RZnP(CF_3)_2$ compound.[2] On this basis, BF_3 should act as a catalyst for converting $(CF_3)_2PSiH_3$ to $(CF_3PCF_2)_2$; and indeed it seems to do so.

Silylbis(trifluoromethyl)phosphine

Synthesis. Iodosilane was made from $C_6H_5SiH_3$ by action of hydrogen iodide[3] and shaken with mercury and $(CF_3)_2PI$ at 25°. The container was a vacuum-baked vertical stopcocked tube or tubulated bulb, with a side arm into which the volatiles could be condensed at −196°; then a fresh mercury surface could be restored by vacuum sublimation of the mercury iodides. The process was regarded as complete when the typical infrared pattern of $ISiH_3$ at 903 cm^{-1} (a doublet with side fine structure) had disappeared. A typical experiment employed 3 mmol of $ISiH_3$ with $(CF_3)_2PI$ added in 0.5-mmol portions up to a total of 7 mmol and yielded 2.85 mmol of $(CF_3)_2PSiH_3$ (95%). The surplus $(CF_3)_2PI$ was represented mostly by $P_2(CF_3)_4$, which implied no loss because its reconversion to $(CF_3)_2PI$ is very easy.

When $BrSiF_3$ was used instead of $ISiH_3$, it failed to react;

Table I. Reactions of $(CF_3)_2PSiH_3$

Reactant (mmol)	Conditions	Products (mmol where known)
BCl_3 (0.75)	20 hr at 25°	0.4 BF_3, 0.02 SiH_3Cl, 0.73 H_2SiCl_2, 0.13 $(CF_3)_2PH$, 0.125 $P_2(CF_3)_4$, trace $(CF_3)_2PH$; $(H_2PBF_2)_x$ empirical formula of residue by difference
$B(OCH_3)_3$ (0.15 used of 0.50)	24 hr at 25° nil, 24 hr at 50°	0.2 SiH_4, 0.24 $(CF_3)_2PH$, 0.26 $(CF_3)_2PCH_3$, nonvolatiles
CH_3OBCl_2 (0.45)	Fast at <25°	0.36 $(CF_3)_2PCH_3$, 0.3 SiH_3Cl, 0.2 H_2SiCl_2, <0.05 $(CF_3)_2PH$
$(CH_3)_2PCl$ (0.46)	Fast at <25°	Yellow ppt, 0.12 $(CH_3)_2P\text{-}P(CF_3)_2$,[a] nil SiH_3Cl, 0.45 H_2SiCl_2, 0.07 $(CF_3)_2PH$, 0.02 $P_2(CF_3)_4$
$(CF_3)_2PCN$ (0.5)	28 hr at 25°	0.22 $P_2(CF_3)_4$, 0.34 SiH_3CN, 0.17 $(CF_3)_2PH$
PF_3	1 hr at 105°	No reaction
PF_5	Fast at <25°	Obsd $(CF_3)_2PH$, $(CF_3)_2PF$, H_2SiF_2; minor unidentified products
CH_3CN (0.5)	3 hr at 65°	Obsd $P_2(CF_3)_4$, SiH_4
CH_3NC (0.45)	Fast at <25°	Brown solids, 0.1 SiH_4, 0.03 $(CF_3)_2PH$, 0.06 $P_2(CF_3)_4$
$BrSiF_3$ (0.07 used of 0.5)	16 hr at 50° nil, 24 hr at 120°	Total of $BrSiH_3$, $(CF_3)_2PH$, and SiF_4 amounts to 0.13

[a] Prior synthesis: L. R. Grant, Jr., and A. B. Burg, *J. Amer. Chem. Soc.*, 84, 1834 (1962).

the only result was synthesis of $P_2(CF_3)_4$. Thus it appears that our synthesis of $(CF_3)_2PSiH_3$ succeeded only because of the relatively weak and reactive character of the Si–I bond.

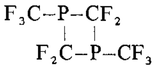
The product $(CF_3)_2PSiH_3$ was purified by high-vacuum fractional condensation in a Stock-type manifold: it passed a trap at −78° and condensed at −112°. Its equilibrium vapor pressures are represented by 3.0 mm at −63°, 44 mm at −23°, and 144 mm at 0° ($\log P = 7.767 - 1532/T$; calcd values 3.0, 43.9, and 144 mm). Its vapor-phase molecular weight was determined as 200.4 (calcd 200.1). It proved to be stable during 16 hr at 90° but was 30% decomposed during 5 hr at 175°.

Spectroscopic Characterization. The infrared spectrum of $(CF_3)_2PSiH_3$ vapor was recorded by the Beckman IR20A instrument, with a calibrated expanded scale for accuracy to 1 cm^{-1}. Its most characteristic frequencies (cm^{-1}, with relative intensities in parentheses) are listed as 2203 (6.3), 1192 (19), 1157 (21), 1118 (21), 930 (2.6), 896 (9), 745 (1.2), 595 (0.3), and 417 (2.5). Traces of moisture (as from the KBr windows) could lead to $(CF_3)_2PH$ and $(SiH_3)_2O$, the strongest bands of which superposed upon those of $(CF_3)_2PSiH_3$; thus such impurities could be detected better by nmr spectra.

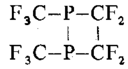
The proton nmr spectrum (Varian T-60 instrument) showed the expected doublet of septets, centered at 3.76 ppm downfield of TMS ($^2J = 19.5$ sec^{-1} and $^7J = 1.35$ sec^{-1}). The satellite doublet of doublets due to ^{29}Si gave J_{SiH} as 229 sec^{-1}. The ^{19}F spectrum (T-60) was a doublet of quartets as expected, centered at 46.7 ppm upfield of Cl_3CF ($^2J = 68.5$ sec^{-1} and $^4J = 1.35$ sec^{-1}). The ^{31}P spectrum (HA-100 instrument) showed the expected septet of quartets, centered at 42.5 ppm upfield of H_3PO_4 and confirming the coupling constants 68.5 and 19.5 sec^{-1}. All results agree with the formula $(CF_3)_2PSiH_3$.

Boron Fluoride Cleavage. For reaction 3 and its sequel, a mixture of 0.85 mmol each of $(CF_3)_2PSiH_3$ and BF_3, in a thick-walled nmr tube, was monitored by the ^1H and ^{19}F spectra. After 64 hr at 25° the ratio of SiH_3F to $(CF_3)_2PSiH_3$ was 1:8; it rose to 1:1 during 10 hr at 60°. After a total of 26 hr at 60°, the $(CF_3)_2PSiH_3$ was not detectable, and the SiH_3F had gone to form H_2SiF_2, $HSiF_3$, and nonvolatiles. The mixture was resolved into its major components by high-vacuum fractional condensation: 0.20 mmol of $(CF_3PCF_2)_2$ (47% yield), a trace of $P_2(CF_3)_4$, and a 1.37-mmol fraction consisting primarily of BF_3 and H_2SiF_2, with traces of B_2H_6 and $HSiF_3$.

The $(CF_3PCF_2)_2$ from all such experiments showed ^{19}F nmr spectra exactly matching those for the previously reported ring compound[2]



but always accompanied by about one-fifth as much of what appears to be the isomer



Its CF_3 groups appeared as a doublet of triplets of triplets centered at 47 ppm upfield of Cl_3CF ($^2J = 92$ sec^{-1}; $^3J = 12.2$ and 3.4 sec^{-1}), while the CF_2 groups showed a second-order spectrum centered at 81 ppm. This pattern was bounded by two major multiplets whose centers were 278 sec^{-1} apart.

Attempts to get a cleaner reaction photochemically did not succeed: with low-intensity mercury radiation, nothing happened during 14 hr at 25°, whereas a high-intensity quartz–mercury lamp (60 min, 25°) gave a mixture of HCF_3, SiH_3F, F_2SiH_2, $(CF_3)_2PH$, and even some $(CF_3)_3P$; more interesting products may have been destroyed.

According to the presumed overall process

$$(CF_3)_2PSiH_3 + BF_3 \rightarrow SiH_3F + (CF_3)_2PBF_2$$

$$2(CF_3)_2PBF_2 \rightarrow 2BF_3 + (CF_3PCF_2)_2 \qquad (4)$$

the formation of $(CF_3PCF_2)_2$ should be BF_3 catalyzed. In fact, 0.53 mmol of $(CF_3)_2PSiH_3$ and 0.12 mmol of BF_3 reacted during 40 hr at 60° to give 0.02 mmol of $P_2(CF_3)_4$, a 0.53-mmol fraction containing BF_3 with more H_2SiF_2, and 0.12 mmol of $(CF_3PCF_2)_2$, a 45% yield for which reaction 3 would require twice as much BF_3 as was actually employed. Thus the catalytic role of BF_3 was apparent despite side reactions. With 2 BF_3 per $(CF_3)_2PSiH_3$, the main difference was an increase of rate: after 20 hr at 60°, the yield of $(CF_3PCF_2)_2$ was 40%.

Other Reactions. Further explorations of the chemistry of $(CF_3)_2PSiH_3$ are summarized by Table I. Almost all of these experiments were aimed at simple cleavage of the P–Si bond and so were tried with equimolar mixtures. They were performed in small nmr tubes, for monitoring of reaction progress; then most tubes were opened to the vacuum line for resolution of the mixtures and identification of isolated products by their nmr or infrared spectra.

Few of these reactions show the initial cleavage products expected by analogy to hypothetical reaction 3; even the expected SiH_3X often is represented only by its decomposition products, including nonvolatiles containing most of the Si–H bonds. The BCl_3 reaction seems intrinsically different from that of BF_3; indeed, the trace of $(CF_3PCF_2)_2$ may be due only to the formation of BF_3. The formula $(H_2PBF_2)_x$ does not necessarily imply a clean polymer.

The methyl borate reaction is interesting because it evidently occurs by cleavage at the C–O bond; no trace of CH_3OSiH_3 could be found. A process like reaction 3 might be too much to expect if the driving force is partly an increase of π-bond energy, which is fairly high in methyl borate. The CH_3OBCl_2 reaction is far faster and gives more $(CF_3)_2PCH_3$.

The $(CF_3)_2PCN$ and $(CH_3)_2PCl$ reactions are relatively clean cleavages, giving the expected products. The experiment with PF_5 was intended for fluorination of the SiH_3 group, as succeeded for disiloxanes.[4]

Trimethylsilylbis(trifluoromethyl)phosphine

Synthesis. The action of mercury upon $(CF_3)_2PI$ and $ISi(CH_3)_3$ favored the formation of $P_2(CF_3)_4$ more than in the $ISiH_3$ case: the conversion to $(CF_3)_2PSi(CH_3)_3$ never was complete; even though small portions of $(CF_3)_2PI$ were added to bring the total to 5 times the calculated amount, the conversion did not exceed 78%. However, this still is an efficient and fairly convenient synthesis, from reactants not too difficult to obtain. Purification is possible by repeated slow high-vacuum fractional condensations: $(CF_3)_2PSi(CH_3)_3$ condenses in a U trap at −55°, through which $P_2(CF_3)_4$ and $ISi(CH_3)_3$ pass more easily.

Boron Fluoride Cleavage. With equimolar BF_3, the cleavage of $(CF_3)_2PSi(CH_3)_3$ was completed during 48 hr at 25°; or with $2BF_3$, 5 hr sufficed. The results are expressed in mmol as follows

$$\begin{array}{cccc} (CF_3)_2PSi(CH_3)_3 + & BF_3 \rightarrow & (CF_3PCF_2)_2 + & (CH_3)_3SiF \\ 0.45 & 0.500 & 0.09 & 0.45 \\ & \underline{-0.465} & & \\ & 0.035 & & \\ 0.47 & 0.97 & 0.12 & 0.47 \\ & \underline{-0.85} & & \\ & 0.12 & & \end{array}$$

with the first experiment yielding also 0.10 $P_2(CF_3)_4$ and 0.06 $(CF_3)_2PH$ and the second respectively 0.03 and 0.18 of these.

Here the silane product was not subject to loss by decomposition, so that the relation to reaction 3 is more apparent. However, the yields of $(CF_3PCF_2)_2$ were not better. Again, BF_3 played a catalytic role.

Acknowledgment. This research was supported by Grant No. GP-17472 from the National Science Foundation.

Registry No. $(CF_3)_2PSiH_3$, 53716-32-8; $(CF_3)_2PSi(CH_3)_3$, 21658-00-4; $ISiH_3$, 13598-42-0; $(CF_3)_2PI$, 359-64-8; $ISi(CH_3)_3$, 16029-98-4; BF_3, 7637-07-2; BCl_3, 10294-34-5; $B(OCH_3)_3$, 121-43-7; CH_3OBCl_2, 867-46-9; $(CH_3)_2PCl$, 811-62-1; $(CF_3)_2PCN$, 431-97-0; PF_5, 7647-19-0; CH_3CN, 75-05-8; CH_3NC, 593-75-9; $BrSiF_3$, 14049-39-9.

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Base Hydrolysis of Coordinated Benzonitrile. Reactions of Rhodium(III) and Iridium(III) Complexes

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Recent studies of the base hydrolyses of organonitriles coordinated to non-labile metal centers have included the acetonitrile–pentaammine complexes of Co(III),[2] Rh(III),[3] Ru(II),[3] and Ru(III)[3] ($M(NH_3)_5(CH_3CN)^{m+}$) and analogous benzonitrile complexes of Co(III)[4,5] and Ru(III).[3] Reported here are the base hydrolysis rates for benzonitrile complexes of rhodium(III) and iridium(III), thus allowing comparisons for a complete homologous series $M(NH_3)_5(C_6H_5CN)^{3+}$ where M is the tripositive metal ion of Co, Rh, or Ir. In an earlier paper we suggested that the nitrile hydrolysis rates are a reflection of the acidities of the respective metal centers and that such effects are functions of both electrostatic and electronic factors.[3] The pK_a values of the amide complexes provide one quantitative measure of the relative metal ion acidities and these are reported here for the benzamide[6] complexes of Rh(III) and Ir(III).

Experimental Section

Materials. Reagent grade chemicals and doubly distilled water were used throughout. $[Rh(NH_3)_5(C_6H_5CN)](ClO_4)_3$ was prepared according to Foust's procedure[7] (λ_{max} 235 nm, ϵ 2.04 × 10^4 M^{-1} cm^{-1}). $[Ir(NH_3)_5(C_6H_5CN)](ClO_4)_3$ was prepared similarly[8] by heating $[Ir(NH_3)_5OH_2](ClO_4)_3$ and benzonitrile together in dry dimethylacetamide and was recrystallized twice [λ_{max} 236 nm (ϵ 1.23 × 10^4 M^{-1} cm^{-1}), 283 nm (sh) (2.88 × 10^3), 255 nm (sh) (9.98 × 10^3); ν_{CN} 2280 (w) cm^{-1}, KBr disk].

Spectra of Benzamide Complexes. An accurately weighed sample of the appropriate benzonitrile complex was dissolved in ~10 ml of dilute NaOH. After hydrolysis was complete, the solution was diluted to a known volume at the desired pH and the uv spectrum was run on a Cary Model 14 spectrophotometer. $Rh(NH_3)_5(benzamide)^{3+}$: λ_{max} 235 nm (ϵ 1.29 × 10^4 M^{-1} cm^{-1}) (0.2 M $HClO_4$, 0.8 M $NaClO_4$). $Rh(NH_3)_5(benzamido)^{2+}$: 240 nm (sh) ($\epsilon$ 7.70 × 10^3) (pH 9, 1 M $NaClO_4$). $Ir(NH_3)_5(benzamide)^{3+}$: λ_{max} 229 nm (ϵ 8.70 × 10^3), 253 nm (sh) (ϵ 7.23 × 10^3) (1 M $HClO_4$). $Ir(NH_3)_5(benzamido)^{2+}$: λ_{max} 263 nm (ϵ 5.07 × 10^3), 218 nm (ϵ 8.78 × 10^3) (pH 7, 1 M $NaClO_4$).

pK_a Measurements. The benzamido complexes were generated in aqueous solution by base hydrolysis of the corresponding benzonitrile complex, and the solution was adjusted to pH ~6 with 1 M $HClO_4$. Solutions over a series of pH values were prepared[3] with $[Rh(III)] = 7.2 \times 10^{-5}$ M or $[Ir(III)] = 1.4 \times 10^{-4}$ M. The spectrum of each solution was recorded on a Cary Model 14 at 25°, and the pK_a values were determined by plotting absorbance against pH at 235 nm [Rh(III)] and 260 nm [Ir(III)]. The pK_a was calculated as the pH where the absorbance equaled $[\epsilon(B) + \epsilon(BH^+)][complex]/2$).

Rate Studies. A stock solution of the benzonitrile complex was prepared in 1.0 M $NaClO_4$. Stock NaOH solutions (from 8 × 10^{-4} to 0.01 M) were also prepared at μ 1.0 M. Equal volumes of reactants at 25.0° were mixed in cylindrical 2.0-cm cells and absorbance *vs.* time was recorded at 236 nm on a Cary Model 118 equipped with a thermostated (25.0°) cell compartment. Rates for the iridium(III) complex were examined at five hydroxide concentrations and for the rhodium(III) complex at four concentrations. Triplicate runs were carried out at most concentrations.

Results and Discussion

The base hydrolysis products were characterized by their uv spectra which displayed reversible acid–base behavior in a manner analogous to that for previously studied Co(III)[4] and Ru(III)[3] complexes. The spectra were neither those of the corresponding aquo or hydroxo complexes nor those of the starting material. The molar extinction coefficients and general spectral features in the uv region are consistent with the π–π^* bands expected from coordinated benzamide. The pK_a values for the reversible acid–base behavior were 2.2 ± 0.1 and 2.4 ± 0.1 for the rhodium(III) and iridium(III) benzamide products, respectively. The iridium(III) product was isolated from a reaction solution of 25 mg of nitrile complex dissolved in ~3 ml of dilute sodium hydroxide. After hydrolysis the solution was neutralized with $HClO_4$ and saturated $NaClO_4$ solution was added dropwise. The white precipitate which appeared upon cooling was isolated by filtration, washed with ethanol and ether, and dried under vacuum (yield 8.5 mg). The uv spectrum and acid–base behavior was the same as that