

**Figure 1.** Copper  $2p_{3/2}$  spectra for (a)  $\text{Cu}(o\text{-phen}(\text{bi})_2)_2^-$ , (b)  $\text{Cu}(o\text{-phen}(\text{bi})_2)^-$ , (c)  $\text{Cu}(\text{en}(\text{bi})_2)_2^-$ , and (d)  $\text{Cu}(o\text{-en}(\text{bi})_2)^-$ .

analogue  $\text{K}_2\text{Ni}(\text{bi})_2$  is isoelectronic with  $\text{KCu}(\text{bi})_2$  yet shows no satellite structure nor do other low-spin nickel(II) systems.<sup>1</sup> The qualitative theoretical arguments employed to explain the presence of satellites in copper appear to be valid for nickel(II) in  $\text{K}_2\text{Ni}(\text{bi})_2$ . It is hoped that this inconsistent behavior for isoelectronic nickel(II) and copper(III) will be successfully addressed by theoreticians thus improving the current model for satellite structure in transition-metal XPS.

### Experimental Section

The complexes were prepared following procedures reported in the literature.<sup>7</sup> Both potassium and tetra-*n*-butylammonium salts of the complexes were investigated. The ligands were prepared by the method of Birker et al.<sup>7a,14</sup> Chemicals were reagent grade. *o*-Phenylene-diamine was recrystallized immediately before use.

XPS measurements were carried out on a Varian V-IEE 15 high-resolution electron spectrometer equipped with a 620 on-line computer (8K). During the course of the measurements the sample was maintained at about 100 K by cooling with liquid nitrogen. The samples were run as finely ground powders dusted onto the backing of a one-sided adhesive Cellotape (Scotch Tape, 3M Co.) which was attached to the cylindrical sample holder. The spectra were standardized with the  $\text{C}_{1s}$  aliphatic hydrocarbon line for which a binding energy of 284.0 eV was assigned.<sup>15</sup>

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**Registry No.**  $\text{Cu}(o\text{-phen}(\text{bi})_2)_2^-$ , 71964-24-4;  $\text{Cu}(o\text{-phen}(\text{bi})_2)^-$ , 71964-25-5;  $\text{Cu}(\text{en}(\text{bi})_2)_2^-$ , 71964-26-6;  $\text{Cu}(\text{en}(\text{bi})_2)^-$ , 71964-27-7;  $\text{Cu}(\text{bi})_2^{2-}$ , 57674-47-2;  $\text{Cu}(\text{bi})_2^-$ , 71964-28-8.

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## Chemistry of Trifluoromethyl Compounds. 2. Synthesis and Characterization of Bis(pyridine)bis(trifluoromethyl)zinc

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There has been interest in the use of bis(trifluoromethyl)mercury as a source for trifluoromethyl groups by ligand-exchange reactions.<sup>1</sup> Exchange reactions involving  $\text{Hg}(\text{CF}_3)_2$  have concentrated on the main-group elements, although a report has appeared on the dimethylcadmium/bis(trifluoromethyl)mercury system.<sup>2</sup>

In a previous paper,<sup>3</sup> we reported  $^{19}\text{F}$  NMR evidence for bis(trifluoromethyl)zinc. It was found that facile methyl and trifluoromethyl exchange occurred between dimethylzinc and bis(trifluoromethyl)mercury in pyridine. We report here the isolation of bis(pyridine)bis(trifluoromethyl)zinc from the reaction of dimethylzinc and bis(trifluoromethyl)mercury in pyridine. Chemical and physical properties of this compound are presented. The stability of the resulting bis(trifluoromethyl)zinc compound is due to the pyridine, which as a result of its electron-withdrawing trifluoromethyl groups evidently satisfies the electronic properties of zinc.

### Experimental Section

**Preparation of  $\text{Zn}(\text{CF}_3)_2 \cdot 2\text{C}_5\text{H}_5\text{N}$ .**  $\text{Hg}(\text{CF}_3)_2$  (0.521 g, 1.54 mmol) was dissolved in 4.0 mL of dried pyridine. To this was added 0.071 g (0.74 mmol) of  $\text{Zn}(\text{CH}_3)_2$ . The solution was stirred for approximately 8 h at room temperature. During this time, the initially clear solution turned red-brown. The volatile materials were removed under vacuum over a 2-day period in order to remove all unreacted  $\text{Hg}(\text{CF}_3)_2$ . The volatile materials were identified as pyridine,  $\text{Hg}(\text{CF}_3)(\text{CH}_3)$ , and  $\text{Hg}(\text{CF}_3)_2$ . The light brown residue remaining was identified as bis(trifluoromethyl)zinc by its  $^{19}\text{F}$  NMR spectrum in pyridine solution.<sup>3</sup> No other trifluoromethyl-containing compounds were identified from both  $^{19}\text{F}$  and  $^1\text{H}$  NMR spectra. The purity of the compound was determined by mass spectral analysis. No mercury-containing compounds were found to be present. The sample was stored in evacuated containers and all subsequent transfers were made in an oxygen-free argon drybox to exclude exposure to moisture. Recrystallization was accomplished by dissolving the light brown residue in benzene. Filtration by Schlenk-type apparatus removed insoluble polymeric materials. Removal of the benzene by vacuum distillation resulted in a light brown residue of  $\text{Zn}(\text{CF}_3)_2 \cdot 2\text{py}$ . Purification by this method resulted in 0.22 g (82% yield) of material.

Anal. Calcd for  $\text{ZnC}_{12}\text{H}_{10}\text{F}_6\text{N}_2$ : C, 39.86; H, 2.79; F, 31.53; N, 7.75; Zn, 18.08. Found: C, 40.20; H, 3.02; F, 29.92; N, 7.68; Zn, 16.87.

**Infrared Spectrum.** The infrared spectrum of  $\text{Zn}(\text{CF}_3)_2 \cdot 2\text{py}$  in a KBr disk was recorded on a Perkin-Elmer 180 infrared grating spectrophotometer: 3120 (vw), 3080 (vw), 3060 (vw), 2920 (vw), 2885 (vw), 2820 (vw), 1650 (w), 1606 (s), 1572 (w), 1490 (m), 1449 (s), 1257 (w), 1220 (m), 1161 (sh), 1144 (s), 1130 (s), 1070 (m), 1042 (m), 1015 (m), 960 (s), 945 (s), 880 (w), 756 (s), 700 (s), 680 (w), 635 (m), 500 (w), 421 (w), 416 (w), 288 (w), 245 (w)  $\text{cm}^{-1}$ . (See Figure 1.)

**Nuclear Magnetic Resonance Spectra.**  $^{19}\text{F}$  and  $^1\text{H}$  NMR spectra were recorded on a Varian EM-390 NMR spectrometer operating at 84.56 and 90 MHz, respectively. Interfaced with the instrument was a Nicolet NMR-80 system employing a Bruker Correlation Program for signal averaging. Proton-decoupled  $^{13}\text{C}$  NMR spectra were recorded on a Varian XL-100 NMR spectrometer operating at 25.2 MHz interfaced with a Nova 1210 computer for Fourier

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transform operations. Spectra were recorded with 2.5 wt % of sample.  $Zn(CF_3)_2 \cdot 2py$  has a  $^{19}F$  NMR chemical shift of 39.7 ppm upfield from  $CFCl_3$  in  $CH_2Cl_2$ . (The chemical shift in pyridine is 38.0 ppm.)<sup>3</sup> The  $^1H$  NMR spectrum in  $CH_2Cl_2$  shows the ortho, meta, and para protons<sup>4</sup> on pyridine at  $\delta$  8.49, 7.40, and 7.81 ppm, respectively. Chemical shifts were calculated from internal  $CH_2Cl_2$  chemical shift with  $\delta(Me_4Si) = \delta(CH_2Cl_2) + 5.28$  ppm. (Free pyridine in  $CH_2Cl_2$  has ortho, meta, and para chemical shifts of 8.43, 7.18, and 7.56.) The  $^{13}C$  NMR spectrum in  $CD_2Cl_2$  shows ortho, meta, and para carbons on pyridine at 149.3, 125.9, and 139.9 ppm from  $Me_4Si$ . (Free pyridine in  $CD_2Cl_2$  has ortho, meta, and para chemical shifts of 150.1, 124.0, and 136.0 ppm from  $Me_4Si$ .) (See Figure 2.) Due to the inability to decouple the fluorines on the trifluoromethyl group, the trifluoromethyl carbon chemical shift was not observed.

**Thermal Stability.** Heating a sample of  $Zn(CF_3)_2 \cdot 2py$  in vacuo at 100 °C for 4 h yielded  $CF_3H$  and some pyridine as indicated by infrared and mass spectral analysis. In solution,  $Zn(CF_3)_2 \cdot 2py$  is stable for several weeks at room temperature. At 50 °C solutions of  $Zn(CF_3)_2 \cdot 2py$  showed slow decomposition, yielding  $CF_3H$ . At 80 °C a pyridine solution of  $Zn(CF_3)_2 \cdot 2py$  showed complete decomposition in 2 h. Pyridine is the major product identified in mass spectral decomposition of  $Zn(CF_3)_2 \cdot 2py$ .

**Hydrolysis.** Addition of water to samples of  $Zn(CF_3)_2 \cdot 2py$  yielded  $CF_3H$ . Hydrolysis was slow as trifluoromethane was formed over a period of several hours from the yellow solution which resulted from addition of 0.2 mL of  $H_2O$  to 22 mg of sample.

**Reaction with Chlorine Gas.** To 11.4 mg (0.032 mmol) of  $Zn(CF_3)_2 \cdot 2py$  in a glass bulb was added 11.4 mmol of  $Cl_2$ . The light brown material turned light tan. Isolated was 9.4 mg of  $ZnCl_2 \cdot 2py$  (theoretical, 9.35 mg).  $ZnCl_2 \cdot 2py$  was identified by comparison of its infrared spectrum to an authentic sample prepared by literature methods.<sup>5</sup>  $CF_3Cl$  was identified to be the only volatile gas after removal of excess chlorine gas by trap to trap vacuum distillation.

**Solubility.**  $Zn(CF_3)_2 \cdot 2py$  dissolves in various solvents such as pyridine, chloroform, methylene chloride, acetonitrile, tetrahydrofuran, benzene, and diethyl ether in respective ratios of 1:0.3:1:1:0.5:0.7:0.3 as determined by  $^{19}F$  NMR spectral integration on 5 mg of sample in 0.2 mL of solvent. The compound is insoluble in  $CS_2$  and  $CCl_4$ . After several weeks, no appreciable decomposition was noticed in any of the solvents, except for chloroform where  $CF_3H$  was the only  $^{19}F$  signal present. Solution stability appears very high in dry solvents.

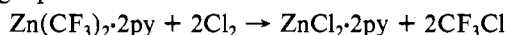
## Results and Discussion

Bis(pyridine)bis(trifluoromethyl)zinc has been isolated from the products of the reaction between dimethylzinc and bis(trifluoromethyl)mercury (1:2 mol ratio) in pyridine. Subsequently it has been found that strong coordinating solvents, such as pyridine, are not necessary. What has been important is the presence of strong donating ligands already present on the dimethylzinc, such as  $Zn(CH_3)_2 \cdot 2py$ .<sup>6</sup> Addition of  $Hg(CF_3)_2$  to  $Zn(CH_3)_2 \cdot 2py$  in methylene chloride results in exchange between the trifluoromethyl groups of  $Hg(CF_3)_2$  and the methyl groups of  $Zn(CH_3)_2 \cdot 2py$ . Final distribution of products is dependent on the mole ratios of the two starting materials. In the absence of pyridine no exchange occurs between  $Hg(CF_3)_2$  and  $Zn(CH_3)_2$  in methylene chloride, up to temperatures of 100 °C. Therefore in this example, the pyridine is necessary for the stability of bis(trifluoromethyl)zinc compounds.<sup>7</sup>

Rapid exchange occurs between  $Zn(CF_3)_2 \cdot 2py$  and  $Zn(C_6H_5)_2$  in pyridine, resulting in  $Zn(CF_3)(C_6H_5)$  at room temperature.  $Zn(CF_3)(C_6H_5)$  has a  $^{19}F$  NMR chemical shift 0.2 ppm less than  $Zn(CF_3)_2$ .<sup>3</sup> This is unusual in that corresponding compounds  $M(CF_3)_2$  and  $M(CF_3)(CH_3)$ ,  $M = Hg$

or Cd, have  $^{19}F$  chemical shifts such that  $M(CF_3)_2$  is of lower field than  $M(CF_3)(CH_3)$ .<sup>2,3</sup>

As expected, hydrolysis liberated fluorofrom,  $CF_3H$ . Reaction with chlorine gas gave chlorotrifluoromethane,  $CF_3Cl$ . The resulting residue was identified as  $ZnCl_2 \cdot 2py$ . The following equation describes the behavior.



Thermal decomposition of bis(pyridine)bis(trifluoromethyl)zinc gave, unexpectedly,  $CF_3H$ , as opposed to the expected  $c-C_3F_6$ .<sup>8</sup> The source of  $CF_3H$  is believed to result from proton abstraction from pyridine by the trifluoromethyl group. The resulting residue after thermal decomposition is dark brown and insoluble in pyridine. While  $CF_3H$  results as the major product from thermal decomposition, pyridine is the first occurring species on ionization in a mass spectrometer. No zinc-containing ions were detected in the mass spectrum.

The  $^1H$  and  $^{13}C$  NMR spectra indicate that pyridine is bound to the zinc. Noticeable shifts of the ortho, meta, and para protons and carbons on the pyridine ring in both the  $^1H$  and  $^{13}C$  NMR spectra indicate considerable electronic effects arising as a result of bonding to trifluoromethyl containing zinc. It is difficult to ascertain the true significance of the effects by examining the  $^1H$  and  $^{13}C$  NMR chemical shifts.<sup>9</sup> The differences in chemical shifts in the ortho, meta, and para positions in the  $^{13}C$  NMR spectra of free pyridine and  $Zn(CF_3)_2 \cdot 2py$  indicate the largest effect at the para position. (Differences in respective spectra are as follows: ortho, 0.8; meta, -1.8; para -3.9 ppm.) This effect is also seen in the  $^1H$  NMR spectra (ortho, 0.06; meta, -0.22; para, -0.25 ppm).<sup>10</sup> One would naturally have expected shifts to involve the pyridine residue on complexation, but the direction of the shifts and factors attributable to these shifts can be debated. However, the  $^1H$  and  $^{13}C$  chemical shifts resemble those of the pyridinium ion rather than diamagnetic transition metal-pyridine complexes.<sup>11</sup> This is expected since a  $d^{10}$  system is involved here. However, the magnitudes of the shift are not as large as in the pyridinium ion.

The structure of  $Zn(CF_3)_2 \cdot 2py$  is believed to be analogous to that of the tetrahedral  $ZnCl_2 \cdot 2py$ . Both the symmetric and asymmetric stretches of the trifluoromethyl group on  $Zn(CF_3)_2 \cdot 2py$  (1144, 1130, 960, 945  $cm^{-1}$ ) shift to lower wavenumbers compared to bis(trifluoromethyl)mercury.<sup>12</sup> The infrared spectrum of  $Zn(CF_3)_2 \cdot 2py$  has many similarities to other transition metal-pyridine complexes.<sup>13</sup> It is difficult to ascertain the significance for the changes as certain modes show large shifts while other modes remain virtually unchanged. In general, the shifts are to higher energy. The noticeable shift to lower wavenumbers results from the ring deformation mode at 700  $cm^{-1}$  (decrease in 2  $cm^{-1}$  from free pyridine). Attempts thus far at obtaining single crystals for

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(10) A  $^{15}N$  study would be informative to see the extent of interaction between the zinc and the pyridine nitrogen. Since the nitrogen is bonded directly to the zinc, one might expect a more dramatic effect on chemical shift of the  $^{15}N$  resonance. From this one could infer some information concerning the strength of the  $\sigma$  bond.

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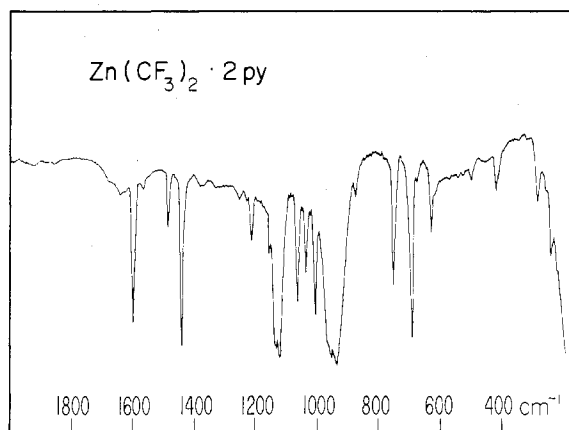


Figure 1. Infrared spectrum of  $\text{Zn}(\text{CF}_3)_2 \cdot 2\text{py}$ .

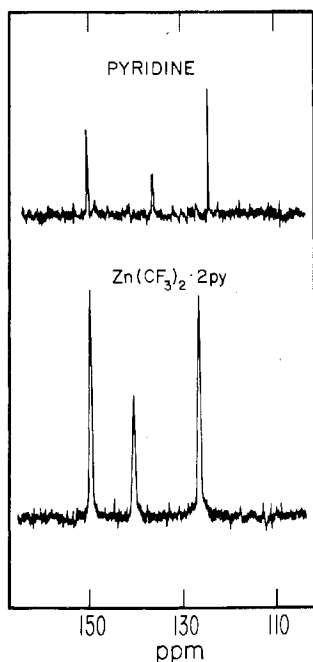


Figure 2. Carbon-13 NMR spectrum of pyridine and  $\text{Zn}(\text{CF}_3)_2 \cdot 2\text{py}$  in  $\text{CD}_2\text{Cl}_2$ .

X-ray structural determination have failed.

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**Registry No.**  $\text{Zn}(\text{CF}_3)_2 \cdot 2\text{py}$ , 71672-49-6;  $\text{Hg}(\text{CF}_3)_2$ , 371-76-6;  $\text{Zn}(\text{CH}_3)_2$ , 544-97-8.

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### Crystal and Molecular Structure of

### $\text{Cl}_2\text{AlN}(\text{C}_2\text{H}_5)\text{C}_2\text{H}_4\text{N}(\text{CH}_3)_2$ , a Neutral, Chelated Four-Coordinate Aluminum Compound, Which Contains Two Types of Al-N Bond

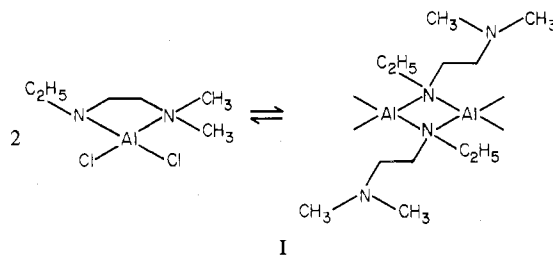
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The chemistry of chelated four-coordinate organoaluminum-nitrogen derivatives has been investigated only in

mol formula	$\text{Cl}_2\text{AlN}_2\text{C}_6\text{H}_{11}$	$2\theta$ range	4-50
mol wt	213.1	scan technique	$\omega$ - $2\theta$
cryst dimens,	$0.40 \times 0.50 \times 0.60$	scan rate,	$0.3 \rightarrow 20.0$
cryst system	monoclinic	deg $\text{min}^{-1}$	
space group	$P2_1/c$	scan range, deg	$0.8 + 0.2$
$a$ , Å	7.240 (4)	$\tan \theta$	
$b$ , Å	9.667 (5)	scan:bkgd time	2:1
$c$ , Å	16.074 (8)	stds	3 every 50
$\beta$ , deg	100.76 (5)	no. of data	1530
$V$ , Å <sup>3</sup>	1105	with $F_o^2 >$	
$Z$	4	$3\sigma(F_o^2)$	
$\rho_{\text{caled}}$ ,	1.28	no. of variables	159
$\text{g cm}^{-3}$		refined	
radiation	Mo K $\alpha$	final $R$	0.031
	( $\lambda = 0.71069$ Å)	final $R_w$	0.032
		abs coeff, $\text{cm}^{-1}$	6.16
		std dev of an	0.652
		observn of	
		unit wt	

recent years,<sup>1,2</sup> and  $(\text{CH}_3)_2\text{AlN}(\text{C}_2\text{H}_5)\text{C}_2\text{H}_4\text{N}(\text{CH}_3)_2$  was the first example of this class of compound. In aromatic solvents the compound has been characterized as a monomeric chelate whereas in aliphatic solvents there is an equilibrium between the monomeric chelate and a dimeric species (I). The



chemistry of this compound is still not fully understood, especially its behavior as a Lewis base, but the compound is a liquid at room temperature, stunting a structural investigation. To overcome this problem, we obtained the similar substance  $\text{Cl}_2\text{AlN}(\text{C}_2\text{H}_5)\text{C}_2\text{H}_4\text{N}(\text{CH}_3)_2$ , and herein we report the results of our structural investigation of this compound.

### Experimental Section

The title compound,  $\text{Cl}_2\text{AlN}(\text{C}_2\text{H}_5)\text{C}_2\text{H}_4\text{N}(\text{CH}_3)_2$ , was kindly supplied by Professor O. T. Beachley, having been prepared by his previously described method.<sup>1</sup> The crystal selected for data collection was sealed in a thin-walled 0.5-mm capillary tube under an inert atmosphere. The final lattice parameters, as determined from a least-squares refinement of the angular settings of 15 reflections accurately centered on an Enraf-Nonius CAD-4 diffractometer, are, along with other relevant crystallographic details, given in Table I. The systematic absences  $h0l, l = 2n + 1$ , and  $0k0, k = 2n + 1$ , uniquely defined the space group to be  $P2_1/c$ . Data were collected by the  $\omega$ - $2\theta$  scan technique with graphite-crystal-monochromated molybdenum radiation. One independent quadrant of data was measured to  $2\theta = 50^\circ$ , leading to 1530 observed [ $I > 3\sigma(I)$ ] reflections. The structure was solved by application of the direct methods program MULTAN.<sup>3</sup> Refinement of the structure so obtained was carried out by using the Busing and Levy full-matrix least-squares program ORFLS.<sup>4</sup> No corrections were made for extinction. Neutral atom scattering factors were taken from the compilations of Cromer and Waber<sup>5</sup> for Cl, Al,

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