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PYRROLYL COMPOUNDS OF THE MAIN-GROUP ELEMENTS. II. THE CRYSTAL --ETC(U)
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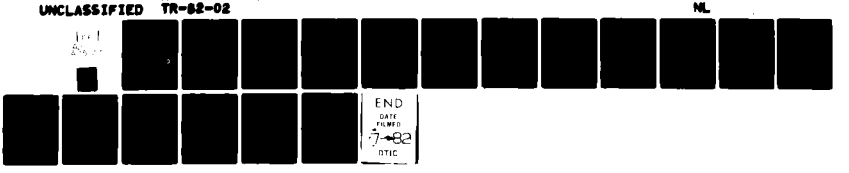

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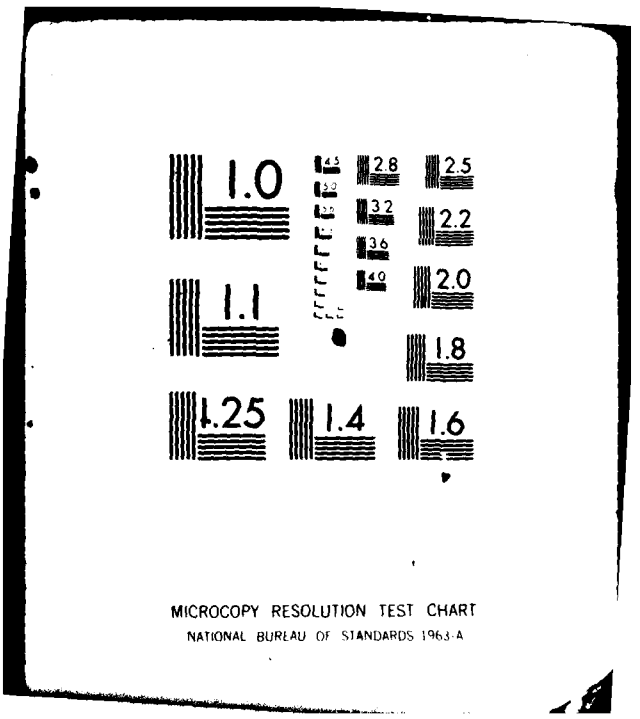
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$(\eta^1\text{-}C_4H_4N)_4Si$ (1) has been prepared by the reaction of C_4H_4NLi with $SiCl_4$ in diethyl ether solution. The molecular structure of 1 has been determined by single-crystal X-ray diffraction. Compound 1 crystallizes in the tetragonal system, space group $P\bar{4}2_1c$ with a = 10.924(6), b = 6.238(4) Å, and Z = 2.

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Each molecule of 1 resides on a $\bar{4}$ (S_4) symmetry site. Each pyrrolyl ligand is N-bonded with a silicon-N-ring centroid angle of 174.5°. The metric parameters for the pyrrolyl ligands in 1 are very similar to those of $(\eta^1\text{-}C_4H_5N)_3P$ and $(\eta^1\text{-}C_4H_4N)_3As$. The reaction of C_4H_4NLi with $SiHCl_3$ in diethyl ether solution results initially in $(\eta^1\text{-}C_4H_4N)_3SiH$ (?); however, in the presence of C_4H_4NLi, compound 2 is converted into compound 1. The structure of compound 2 was deduced from spectroscopic data. Unsuccessful attempts to prepare $(C_4H_4N)_4Sn$ are described.

Contribution from the Departments of Chemistry, University of Alabama, University, Alabama 35486 and The University of Texas at Austin, Austin, Texas 78712

PYRROLYL COMPOUNDS OF THE MAIN-GROUP ELEMENTS. II. THE CRYSTAL AND MOLECULAR STRUCTURE OF $(\eta^1\text{-}C_4H_4N)_4Si$ AND THE REACTION OF C_4H_4NLi WITH $SiHCl_3$

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INTRODUCTION

In a previous paper,[2] we discussed the syntheses, structures and reactivities of tris(pyrrolyl)phosphine and arsine. The present paper is concerned with pyrrolyl compounds of silicon. Interestingly, $(C_4H_4N)_4Si$ (1)[3] and $(C_4H_4N)_3SiH$

(2)[4] were prepared over seventy years ago by the reaction of C_4H_4NK with $SiCl_4$ and $SiHCl_3$, respectively. However, compound 1 has been mentioned only once[5] in the intervening years. We have investigated the reaction of C_4H_4NLi with $SiCl_4$ and $SiHCl_3$ and found that compound 1 is converted to compound 2 in the presence of C_4H_4NLi. A single-crystal X-ray diffraction study of 1 was undertaken to define the mode of bonding between silicon and the pyrrolyl ligands. Moreover, since the pyrrolyl ligand is potentially a two-fold rotor, considerable interest focussed on the stereochemistry of 1.[6]

Experimental Section

Materials and General Procedures. All solvents were dried by distillation from CaH_2 immediately prior to use. Trichlorosilane, $SiCl_4$, $SnCl_4$, and n-BuLi were procured commercially and used as received. Pyrrole was obtained commercially and purified by distillation at reduced pressure (bp 47-48°C at 30 torr).

Most of the materials described herein are moisture and/or oxygen sensitive. Therefore all operations were performed under an inert atmosphere of dry nitrogen.

Spectroscopic Measurements. The ^1H NMR data were obtained in the CW mode at 90 MHz on a Varian EM-390 spectrometer, while the $^{13}C\{^1H\}$ NMR data were recorded in the FT mode on a Varian FT-80 instrument operating at 20 MHz. Deuterochloroform (76.9 ppm relative to Me_4Si) was employed as the internal reference for the ^{13}C spectra. Mass spectra were measured on a CEC-491 spectrometer at an ionizing voltage of 70 eV. Infrared spectra were recorded on a Perkin-Elmer 1330 instrument.

Preparation of $(C_4H_4N)_4Si$ (1). Freshly distilled pyrrole (10.0 mL, 144 mmol) in 250 mL of Et_2O was lithiated at 0°C by dropwise

addition of 90 mL of a 1.6 M solution of n-BuLi in hexane (144 mmol). The formation of C_4H_4NLi was evidenced by the immediate formation of a white solid. The stirring slurry was allowed to warm slowly to room temperature to permit elimination of the butane which had formed. After re-cooling the C_4H_4NLi solution to 0°C, a solution of $SiCl_4$ (4.0 mL, 35 mmol) in 50 mL of Et_2O was added dropwise. After warming slowly to room temperature, the reaction mixture was refluxed for 3 h, allowed to cool to room temperature, and filtered through a medium porosity frit to remove lithium chloride. The LiCl was washed with Et_2O and the washings were combined with the yellow filtrate. (Upon standing, the filtrate turned dark green.) Prolonged chilling of the Et_2O solution at -10°C afforded colorless crystals of $(C_4H_4N)_4Si$ (1) which were collected by filtration. A second crop was obtained by reducing the volume of the mother liquor and re-chilling to -10°C. Also, additional product could be obtained from the LiCl residue by sublimation at 100°C at 10^{-2} torr. The overall yield of 1 was 3.5g (59% based on $SiCl_4$), mp 174°C (uncorr.) (lit: 173.4°). The IR spectrum (nujol) mull) was in good agreement with that reported in the literature.[5] The product appeared to be air stable for several hours at ambient temperature (monitored by IR spectroscopy). NMR data for 1: ^1H, α-H's δ 6.78(m), β-H's δ 6.38(m); $^{13}C\{^1H\}$ α-C's 124.2 ppm(s), β-C's 113.7 ppm(s). The mass spectrum of 1 exhibits a parent peak at m/e 292 (100%), and peaks at m/e 226 (51%), 160 (18%), and 94 (18%) which correspond to the loss of one, two, and three pyrrolyl groups, respectively. Mass spectral peaks at m/e 67 (18%) and 132 (12.5%) correspond to $[C_4H_4NH]^+$ and $[(C_4H_4N)_2]^+$, respectively.

PREPARATION OF $(C_4H_4N)_3SiH$ (2). Pyrrole (202 mmol) in 400 mL of Et_2O was lithiated at -78°C by the addition of 126 mL of 1.6 M n-BuLi solution in hexane as described above. The yellow-white slurry of C_4H_4NLi was treated dropwise with Cl_3SiH (6.6 mL, 65 mmol) in 50 mL of Et_2O. When the addition was

complete, the reaction mixture was allowed to warm to room temperature, then refluxed for 2 h. The precipitated LiCl was separated by filtration and the solvents were removed by distillation. During this time, the filtrate gradually assumed a green color and a light green solid was isolated by filtration and purified for vacuum sublimation (100°C at 10^{-2} torr). The white crystalline sublimate was shown to be pure $(C_4H_4N)_4Si$ (4.9g, 17 mmol) on the basis of NMR and mass spectroscopy. Vacuum distillation of the dark green filtrate (bp 76-79°C at 10^{-2} torr) afforded 5.0g (22 mmol) of clear, colorless liquid $(C_4H_4N)_3SiH$ (2). An additional quantity of $(C_4H_4N)_4Si$ (1.3g, 4.5 mmol) sublimed onto the walls of the distillation apparatus when the temperature was raised to approximately 100°C. The total yields of $(C_4H_4N)_4Si$ (1) and $(C_4H_4N)_3SiH$ (2) were 33 and 34% respectively based on the Cl_3SiH consumed. If the undistilled dark green liquid was allowed to stand for several hours under a nitrogen atmosphere at room temperature it solidified slowly, the only isolable product being $(C_4H_4N)_4Si$. NMR data for 2: ^1H, α-H's δ 6.70(m), β-H's δ 6.33(m), SiH δ 5.91(s); $^{13}C\{^1H\}$ α-C's 123.3 ppm(s), β-C's 113.2 ppm(s). The mass spectrum of 2 exhibits a parent peak at m/e 227 (100%) and peaks at m/e 161 (43%) and 95 (5%) corresponding to the loss of one and two pyrrolyl groups, respectively. Other important peaks occur at m/e 226 (68%), 160 (13%) and 94 (40%) and correspond to $[(C_4H_4N)_3Si]^+$, $[(C_4H_4N)_2Si]^+$, and $[(C_4H_4N)Si]^+$, respectively. Like 1, 2 exhibits peaks at m/e 67 (40%) and 132 (11%) which are assigned to $(C_4H_4NH)^+$ and $[(C_4H_4N)_2]^+$, respectively. The IR spectrum of 2 features a peak at 2230 cm^{-1} which corresponds to ν_{SiH}.

REACTION OF $(C_4H_4N)_3SiH$ (2) WITH C_4H_4NLi. A slurry of 1.6 mmol of C_4H_4NLi in 10 mL of Et_2O was prepared as described above and treated with 0.36g (1.6 mmol) of $(C_4H_4N)_3SiH$ at ambient temperature. The reaction mixture

assumed a green color over a period of several hours. Evacuation of the solvent followed by sublimation afforded 0.35g (1.2 mmol, 75% yield) of pure $(C_4H_4N)_4Si$ which was identified by IR and mass spectroscopy.

ATTEMPTED PREPARATION OF $(C_4H_4N)_4Sn$. (A) REACTION OF $SnCl_4$ WITH C_4H_4NLi. A slurry of 101 mmol of C_4H_4NLi in 100 mL of Et_2O was prepared as described above and treated dropwise at -30°C with a solution of $SnCl_4$ (2.8 mL, 24 mmol) in 50 mL of hexane. The reaction mixture became yellow during this addition. After warming to room temperature and overnight stirring, the reaction mixture became an intractable rubbery mass which adhered to the walls of the flask. Filtration of the supernatant yellow-green solution, followed by vacuum evaporation of the solvents, resulted in traces of an insoluble dark green residue which was not investigated further.

(B) REACTION OF $(Me_2N)_4Sn$ WITH C_4H_4NH. Pyrrole (3.5 mL, 50.6 mmol) was added slowly to $(Me_2N)_4Sn$[7] (3.67g, 12.4 mmol) at -78°C. The reaction mixture was stirred throughout the addition. An exothermic reaction occurred immediately, accompanied by the formation of a yellow solid and the evolution of Me_2NH. Upon warming to room temperature, the entire contents of the flask solidified. The solid was insoluble in pentane and Et_2O; however, yellow solutions could be obtained by treatment of the reaction mixture with $CHCl_3$ or CH_2Cl_2. Rapid evaporation of solvent from these solutions (which deposit polymeric material on standing) resulted in a yellow solid; however mass spectral assay of this material revealed that no $(C_4H_4N)_4Sn$ had been formed.

X-Ray Data Collection and Solution and Refinement of the Structure of $(\eta^1\text{-}C_4H_4N)_4Si$ (1). Clear, colorless crystals of (1) suitable for X-ray diffraction experiments were grown in a sealed evacuated glass tube of 12 mm OD using a thermal gradient sublimator. An appropriate single crystal was sealed under a nitrogen atmosphere

in a 0.5 mm O.D. Lindemann glass capillary. A summary of data collection parameters and crystal data is presented in Table I. The diffracted intensities were collected on an Enraf-Nonius CAD-4 diffractometer using Mo Kα radiation in a manner similar to that described previously.[8] The space group was determined to be $P\bar{4}2_1c$ on the basis of systematic absences. The existence of two molecules per unit cell was interpreted to mean that the silicon atom resided on a $\bar{4}$ site, and difference Fourier maps based on this interpretation permitted the location of all non-hydrogen atoms. Refinement with isotropic temperature factors led to a reliability index, $R = 0.094$. The hydrogen atoms of the pyrrolyl ring were placed at calculated positions of 1.00 Å from the bonded carbon atoms. The hydrogen atoms were given fixed isotropic thermal parameters of 6.33 Å and allowed to ride on the carbon atoms. The non-hydrogen atoms were treated anisotropically and additional cycles of refinement led to the final R and R_w values shown in Table I. The *SHELX*[9] *system was* used for all calculations. Neutral atom scattering factors stored within the SHELX program were used for Si, N, C, and H. Final values for positional parameters and bond lengths/angles are presented in Tables II and III, respectively.

Results and Discussion

Preparation of $(C_4H_4N)_4E$ Compounds. As pointed out in the Introduction, $(C_4H_4N)_4Si$ (1) was prepared over 70 years ago via the reaction of C_4H_4NK with $SiCl_4$.[3] The germanium analogue, $(C_4H_4N)_4Ge$, was prepared subsequently[10] using the same synthetic approach. We have found that 1 can be produced in good yields using the more easily prepared and handled lithium reagent, C_4H_4NLi. In an effort to extend the range of Group 4A pyrrolyl compounds, we attempted the preparation of $(C_4H_4N)_4Sn$ via two routes: (i) the reaction of C_4H_4NLi with

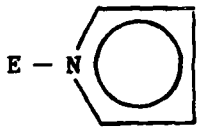
$SnCl_4$, and (ii) the reaction of pyrrole with $(Me_2N)_4Sn$. However, in each case only polymeric products could be isolated. A similar stability trend was evident in Group 5A, viz. $(C_4H_4N)_3P$ is stable, $(C_4H_4N)_3As$ is less so, and attempts to prepare the Sb and Bi analogues were unsuccessful.[2]

MOLECULAR STRUCTURE OF $(\eta^1\text{-}C_4H_4N)_4Si$ (1). The structure of 1 involves monohapto N-bonded attachment of pyrrolyl rings to silicon (Figure 1). Within experimental error the pyrrolyl rings are planar and the sum of bond angles at each nitrogen is 360°. The fact that molecules of 1 reside on S_4 symmetry sites is of some interest. As noted by Mislow and co-workers,[6a,b] the superimposition of four two-fold rotors onto a skeleton of T_d symmetry implies that the array will adopt a symmetry corresponding to one of the seven subgroups of D_{2d}. In the case of ϕ_4Si, X-ray crystallography,[11] gas phase electron diffraction,[12] and empirical force field calculations,[6b] all indicate that the ground state of this molecule possesses S_4 symmetry.[13] Interestingly, however, even though ϕ_4C adopts an S_4 structure in the solid state, empirical force field calculations indicate that the ground state is, in fact, of D_{2d} symmetry. The possibility that the ground state structure of 1 may not be S_4 should therefore be entertained. In view of the fact that the pyrrolyl and cyclopentadienyl ligands are isoelectronic, it would be of interest to compare the structures of 1 and $(\eta^1\text{-}C_5H_5)_4Si$. To the best of our knowledge, however, X-ray crystallographic data are available only for $(\eta^1\text{-}C_5H_5)_4Sn$.[14] The C_5H_5 rings are approximately planar; however, the molecular symmetry is C_1.

As with e.g. N-P bonds, N-Si bond lengths depend on the nature of the hybridization at nitrogen. Typical values for $Si\text{-}N_{sp2}$ bond lengths in silicon amides fall in the range 1.65 - 1.75 Å,[15] while the sum of covalent radii[16] for Si and N (i.e. the $Si\text{-}N_{sp3}$ bond length) is 1.87 Å.[17] The value of 1.723(3) Å

for the Si-N bond length in 1 is thus quite normal for silicon bound to a trigonal planar nitrogen.

Two extreme structures can be considered for a N-bonded pyrrolyl moiety.



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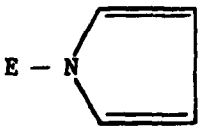

II

The differences in C-C bond lengths (~0.07 Å) and the E-N-ring centroid angle in 1 are very similar to those in $(C_4H_4N)_3P$ [2] and $(C_4H_4N)_3As$ [2] as well as in pyrrolyl complexes of the early transition metals.[18] The viewpoint is emerging, therefore, that the bonding mode of both main-group and early transition metal systems is better represented by I than II.

PREPARATION OF $(C_4H_4N)_3SiH$ (2) AND THE CONVERSION OF $(C_4H_4N)_3SiH$ TO $(C_4H_4N)_4Si$ (1). As part of his classical research in silicon chemistry, Reynolds[4] examined the reaction of $SiHCl_3$ with C_4H_4NK and noted that an approximately 10% yield of $(C_4H_4N)_4Si$ (1) was produced together with a dark green liquid. Reynolds was unable to distill the green liquid; however, he identified the material as $(C_4H_4N)_3SiH$ (2) on the basis of elemental analysis and a molecular weight determination. The formation of 1 was attributed to the presence of $SiCl_4$ in the $SiHCl_3$. We have found that the treatment of $SiHCl_3$ with C_4H_4NLi results in approximately equimolar amounts (33-34% yields) of 1 and 2. We also observed the green liquid described by Reynolds. However, we found that this liquid can, in fact, be distilled providing the pressure is low enough (bp 100°C at 10^{-2} torr). The clear, colorless liquid distillate was identified as pure 2 on the basis of IR, NMR, and mass spectroscopy (see Experimental Section for

data). Since our $SiHCl_3$ was pure and contained no $SiCl_4$, a reason had to be found for the presence of **1** in the products. To shed light on this question, we treated **2** with C_4H_4NLi in Et_2O solution and found that high yields of **1** (~75%) are produced. Clearly, the **1** observed by Reynolds and ourselves results from the reaction of **2** with C_4H_4NK or C_4H_4NLi. The mechanism of this reaction presumably involves the nucleophilic displacement of H^- by the pyrrolyl anion.

Finally, the similarity of the $^{13}C\{^1H\}$ and ^1H NMR data for **1** and **2** establishes that the latter also features a structure with N-bonded monohapto attachment of pyrrolyl rings to silicon.

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SUPPLEMENTARY MATERIAL AVAILABLE: Tables of anisotropic thermal parameters for nonhydrogen atoms in **1** and observed and calculated structure factors for **1** (4 pages). Ordering information is given on any current masthead page.

References and Notes

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TABLE I. Data Collection Parameters and Crystal Data for $(\eta^1\text{-}C_4H_4N)_4Si$ (1)

space group	$P\bar{4}2_1c$
cryst system	tetragonal
cell constants	
a, Å	10.924(6)
c, Å	6.238(4)
vol, Å^3	744.4
ρ, g cm^{-3}	1.30
μ, cm^{-1}	1.60
molecules/unit cell (Z)	2
2θ max, deg	50
measd reflctns	822
obsd reflctns	346
R	0.040
R_w	0.043
max cryst dimens, mm	0.25 x 0.30 x 0.70
mol wt	292.4

TABLE II. Positional Parameters in the $(\eta^1\text{-}C_4H_4N)_4Si$ (1) Molecule

Atom	x/a	y/b	z/c
Si	0.0000	0.0000	0.0000
N(1)	0.1287(3)	0.0046(4)	0.1595(6)
C(1)	0.2335(4)	-0.0669(4)	0.1395(9)
C(2)	0.3185(4)	-0.0270(4)	0.2782(9)
C(3)	0.2695(5)	0.0731(5)	0.3920(9)
C(4)	0.1546(4)	0.0897(4)	0.3165(8)
H(1)	0.2438(4)	-0.1369(4)	0.0378(9)
H(2)	0.4022(4)	-0.0622(4)	0.2975(9)
H(3)	0.3113(5)	0.1221(5)	0.5059(9)
H(4)	0.0968(4)	0.1545(4)	0.3671(8)

TABLE III. Bond Lengths (Å) and Bond Angles (Deg) in the $(\eta^1\text{-}C_4H_4N)_4Si$ (1) Molecule

Bond Lengths			
Si -N(1)	1.723(3)	N(1)-C(1)	1.391(5)
N(1)-C(4)	1.380(6)	C(1)-C(2)	1.342(6)
C(1)-H(1)	1.000	C(2)-C(3)	1.409(7)
C(2)-H(2)	1.000	C(3)-C(4)	1.352(6)
C(3)-H(3)	1.000	C(4)-H(4)	1.000
Bond Angles			
Si -N(1)-C(1)	127.1(3)	Si -N(1)-C(4)	126.6(3)
C(1)-N(1)-C(4)	105.9(4)	N(1)-C(1)-C(2)	109.2(4)
N(1)-C(1)-H(1)	125.4(3)	C(2)-C(1)-H(1)	125.4(3)
C(1)-C(2)-C(3)	108.3(4)	C(1)-C(2)-H(2)	125.9(3)
C(3)-C(2)-H(2)	125.8(3)	C(2)-C(3)-C(4)	106.3(4)
C(2)-C(3)-H(3)	126.8(3)	C(4)-C(3)-H(3)	126.9(3)
N(1)-C(4)-C(3)	110.3(4)	N(1)-C(4)-H(4)	124.8(2)
C(3)-C(4)-H(4)	124.8(3)	Si-N(1)-Cent$(\eta^1\text{-}C_4H_4N)$ [a]	174.5

[a] Cent $(\eta^1\text{-}C_4H_4N)$ is the centroid of the pyrrolyl group

FIGURE CAPTIONS

FIGURE 1. Molecular structure of $(\eta^1\text{-}C_4H_4N)_4Si$ (1) illustrating the atom numbering scheme.

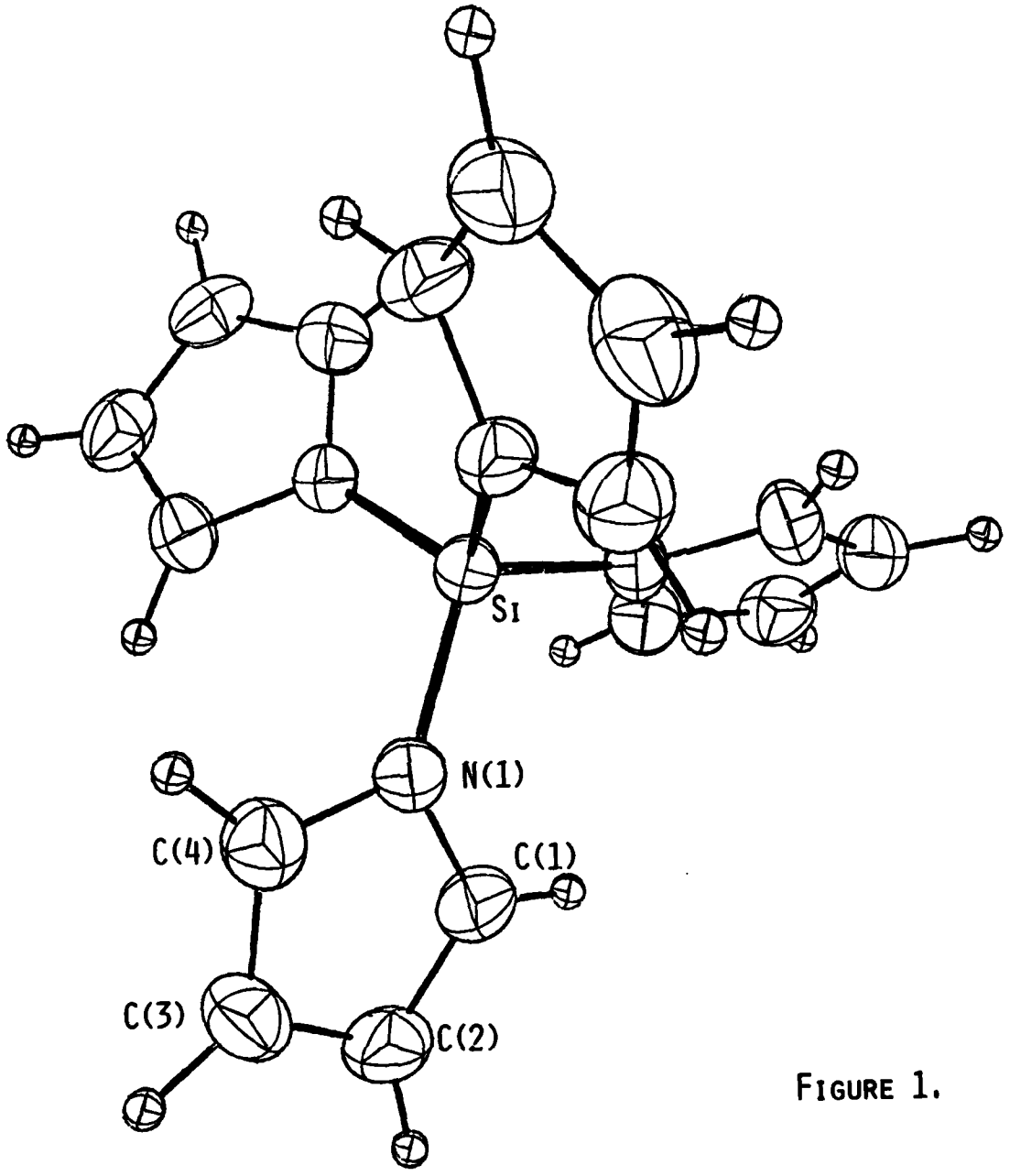

FIGURE 1.