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SYNTHESIS OF -1-SILA-2-AZACYCLOPENTANE RING COMPOUNDS

TSU-TZU TSAI

TECHNICAL REPORT AFML-TR-70-116

SEPTEMBER 1970

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SYNTHESIS OF -1-SILA-2-AZACYCLOPENTANE RING COMPOUNDS

TSU-TZU TSAI

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FOREWORD

This report was prepared in the Polymer Branch, Nonmetallic Materials Division. The work was initiated under Project 7342, "Fundamental Research on Macromolecular Materials and Lubrication Phenomena," Task 734201, "Basic Factors in the Synthesis of Macromolecular Materials." The work was administered under the direction of Dr. Tsu-Tzu Tsai, project engineer, for the Air Force Materials Laboratory.

This report covers work conducted during the period November 1968 to March 1970.

The manuscript was released by the author in May 1970 for publication.

The technical report has been reviewed and is approved.



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ABSTRACT

New ring-compounds: 1,1-diethoxy-2-(triphenylsilyl)-1-sila-2-azacyclopentane, 1,1-diethoxy-2-(ethoxydimethylsilyl)-1-sila-2-azacyclopentane, and tri-(1-ethoxy-1-sila-2-azacyclopentane) were prepared and isolated. The third compound reacts with ethyl alcohol to form 3-aminopropyltriethoxysilane; and with phenyllithium and ethyllithium to form tri-(1-phenyl-1-sila-2-azacyclopentane) and tri-(1-ethyl-1-sila-2-azacyclopentane), respectively.

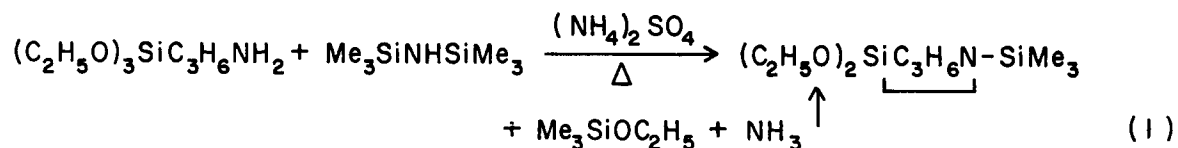
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SECTION I

INTRODUCTION

In our previous report (Reference 1), we reported the synthesis of 1,1-diethoxy-2-(trimethylsilyl)-1-sila-2-azacyclopentane. The ring structure in this compound had shown remarkable stability toward cleavage on standing as well as in the Grignard reactions in which the two ethoxy groups are replaced. Compounds with such ring structures are therefore unlike their oxygen analogs (References 2 through 5), which cleaved on standing and could be developed further for practical use. In this report we had synthesized more such ring-compounds by using hexaphenyl-disilazane and hexamethylcyclotrisilazane instead of the hexamethyl-disilazane in the following reaction:



SECTION II

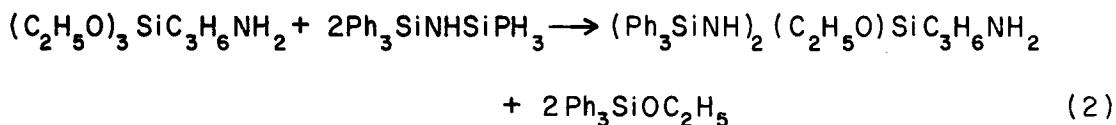
RESULTS AND DISCUSSION

Most products in this study had high boiling points at which ammonium sulfate decomposed to form an obnoxious mercaptan-like odor. We therefore replaced it with its chloride or bromide and found no apparent difference resulted from the change. These halides sublimed instead of decomposing during distillation and could be removed from the products by conventional purification methods.

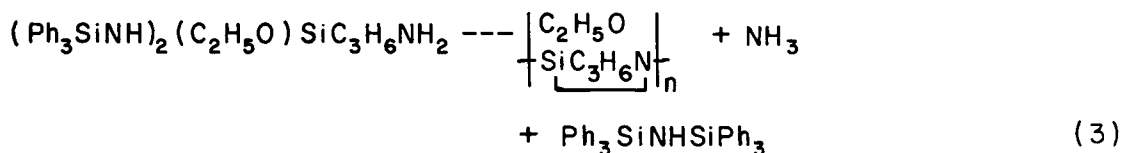
Some hexaphenyldisilazane and 3-aminopropyltriethoxysilane reacted as indicated by Equation 1. The reaction gave off a 50% theoretical amount of ammonia in four days and yielded a small amount of 1,1-diethoxy-2-(triphenylsilyl)-1-sila-2-azacyclopentane (Compound A). The remaining silazane was found to have been mostly converted into ethoxytriphenylsilane during the reaction. Since hexaphenyldisilazane, in every respect, is more stable than its methyl analog, it would be logical to assume that the low yield of A did not result from its own decomposition. Steric effects (References 6 through 8) from bulky group substituents on nitrogen has been claimed as a main factor in preventing the amine exchange reactions. It seems that the three bulky phenyl groups on silicon have had the same effect on these reactions even though two phenyl groups on silicon did not show any effect at all. Besides these products, there was evidence, obtained from mass spectrometric analysis, of two other products with molecular weights of 387 [tri-(1-ethoxy-1-sila-2-azacyclopentane) abbr. trimer D] and 516 [tetra-(1-ethoxy-1-sila-2-azacyclopentane), abbr. tetramer E] respectively. Their formation

probably came from the following mechanism (A):

1st: Formation of $(\text{Ph}_3\text{SiNH})_2 (\text{C}_2\text{H}_5\text{O})\text{SiC}_3\text{H}_6\text{NH}_2$



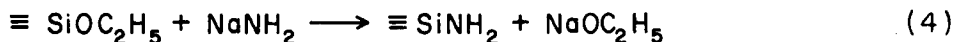
2nd: Condensation of $(\text{Ph}_3\text{SiNH})_2 (\text{C}_2\text{H}_5\text{O})\text{SiC}_3\text{H}_6\text{NH}_2$



where $n \geq 3$

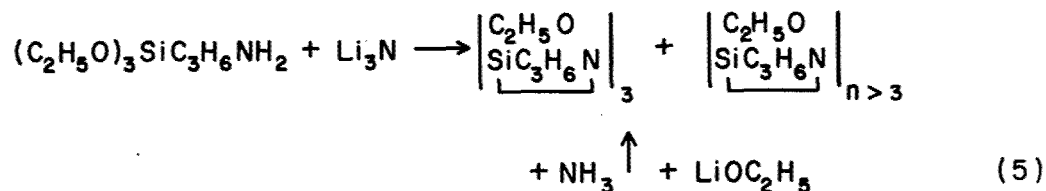
Examples of the 1st and 2nd reactions are known in the literature (References 9 and 10, respectively).

Separation of trimer D or tetramer E from the reaction mixture containing ethoxytriphenylsilane was not successful. In view of the fact that Wannagat and Schreiner's reaction (Reference 9 and Equation 4, below) show that a salt, sodium ethoxide was the side



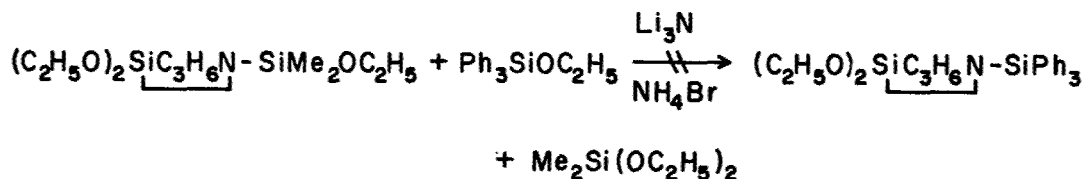
product which could be easily removed from the organosilylanimes, an inorganic amide was therefore used. However, for the following reasons we used lithium nitride for the amide: (1) lithium nitride is more stable, thus easier to handle than sodium amide, and (2) lithium nitride is a dark red powder, while lithium amide and oxide are white; thus, its purity could be ensured by its appearance.

The reaction of 3-aminopropyltriethoxysilane and lithium nitride (Equation 5) took place readily with the generation of heat and ammonia. At the end of the reaction, lithium ethoxide was precipitated out by the addition of xylene and removed



by filtration under nitrogen. The yield of trimer D was 30%. An attempt to isolate the tetramer E was not successful.

An attempt to synthesize A by the following reaction was not successful:



The reaction of hexamethylcyclotrisilazane, 3-aminopropyltriethoxysilane, and a catalytic amount of ammonium bromide yielded 1,1-diethoxy-2-(ethoxydimethylsilyl)-1-sila-2-azacyclopentane (Compound B) (49%) as the main product. The following observations,

1. ammonia evolution was fast and stoichiometric,
2. the yield of B was high and only a small amount of diethoxydimethylsilane was formed, and
3. the formation of high molecular weight substances containing ethoxydimethylsilyl end groups (See Table I),

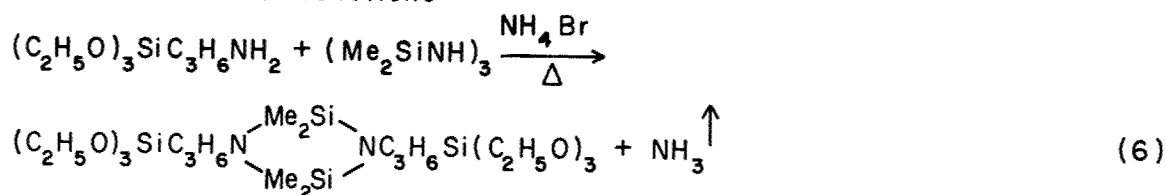
TABLE I
COMPOUNDS CONTAINING
ETHOXYDIMETHYLSILYL END GROUPS

m/e	possible structures
350	$(C_2H_5O)_2 \text{Si} \underline{C_3H_6N} - Me_2Si - NH - Me_2SiOC_2H_5$ or $(C_2H_5O)_2 \text{Si} \underline{C_3H_6N} - (C_2H_5O)_2 \text{Si} \underline{C_3H_6N} H_2$
351*	$(C_2H_5O)_2 \text{Si} \underline{C_3H_6N} - Me_2SiO - Me_2SiOC_2H_5$
387*	$\left[\begin{array}{c} OC_2H_5 \\ \\ Si \underline{C_3H_6N} \end{array} \right]_3$ (Trimer D)
406*	$(C_2H_5O)_2 \text{Si} \underline{C_3H_6N} - \overset{OC_2H_5}{\text{Si} - \underline{C_3H_6N}} - Me_2SiOC_2H_5$
516	$\left[\begin{array}{c} OC_2H_5 \\ \\ Si \underline{C_3H_6N} \end{array} \right]_4$ (Tetramer E)
535	$(C_2H_5O)_2 \text{Si} \underline{C_3H_6N} - \overset{OC_2H_5}{\text{Si} \underline{C_3H_6N}} - \overset{OC_2H_5}{\text{Si} \underline{C_3H_6N}} - \overset{Me_2}{\text{Si} \underline{C_3H_6N}} - SiOC_2H_5$
others	Assignment unsuccessful

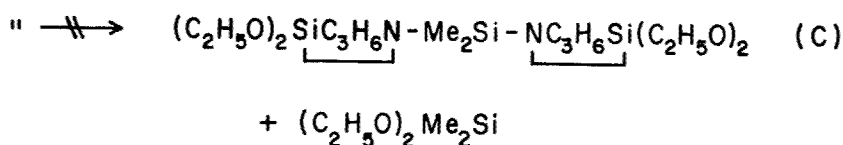
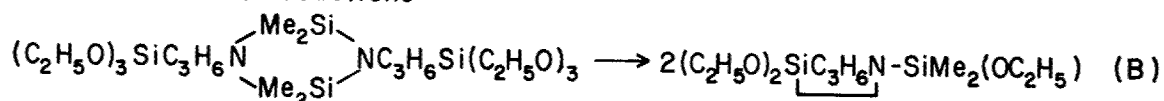
* isolated and proved by mol wt and elementary analysis

were different from the hexaphenyldisilazane reaction and suggested a different mechanism (B):

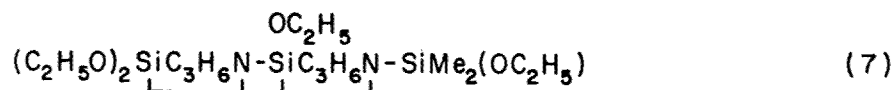
1st. Transamination reactions



2nd. Condensation reactions



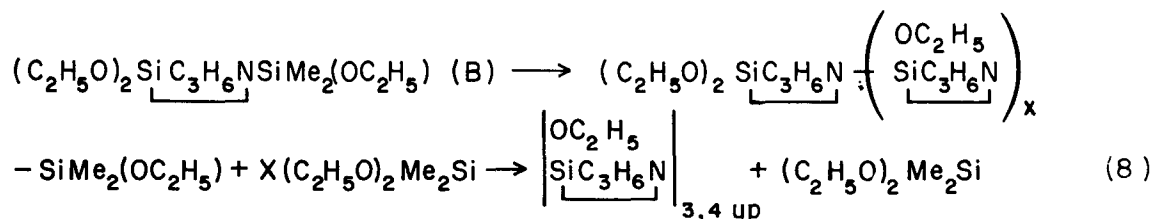
It was surprising to learn that C, which should be formed, was not isolated. A small amount of liquid, isolated by gas liquid partition chromatography (glpc), had a molecular weight and elementary analysis equivalent to that required for C. However, it had three different ethoxy groups in its nmr spectrum, suggesting the structure



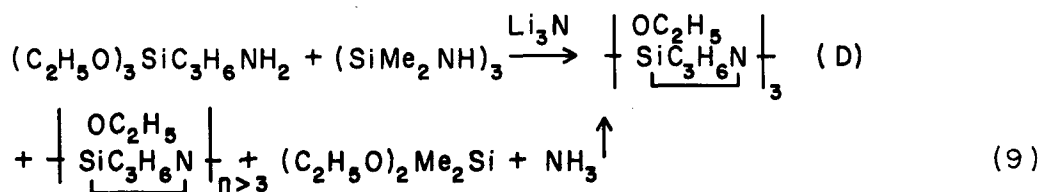
In separate similar experiments, the high boiling substance was analyzed by mass spectrometric techniques. Among numerous m/e peaks, there are several peaks (Table I) to which we could assign structures.

Trimer D and tetramer E were again among the products formed. Although their formation could have arisen from mechanical A, the

presence of other substances (mol wt 406 and 535) had suggested a possible reaction known in the literature (Reference 11). Condensation reaction of B as shown below had occurred along with other reactions.



Although we were not successful in demonstrating the condensation of B with ammonium bromide, the condensation did take place in the presence of a trace of lithium nitride to form diethoxydimethylsilane and a polymeric material in which trimer D was present. We had prepared trimer D in 26% yield by refluxing, under N_2 , the compound 3-amino-propyltriethoxysilane and hexamethylcyclotrisilazane with a trace of lithium nitride until the final result, as shown in Equation 9, was reached.



Although trimer D, obtained by either Equation 5 or 9, can be vacuum distilled repeatedly, a pure sample for analysis was obtained by glpc. The compound, like any other alkoxydimethyl amines (References 12 and 13), is not stable in air, and forms a white solid of unknown nature.

The trimer D isolated was believed to be a mixture of isomers. It had slightly splitting, ethoxy groups in its nmr spectrum. Similar results were reported (Reference 6) for those of unsymmetrically substituted cyclotrisiloxanes and cyclotrisilazanes. In addition to the molecular weight, elementary analysis, and evidence of the right number of hydrogen numbers in its nmr spectrum, the existence of trimer D was further proved by the following chemical reactions:

1. Alcoholysis of Trimer D

Trimer D was found to react slightly with anhydrous ethyl alcohol. Complete alcoholysis could be obtained only if a catalytic amount of lithium ethoxide was present. A sample purified by distillation gave a 79% yield of 3-aminopropyltriethoxysilane.

2. Alkylation and Arylation of Trimer D

Trimer D reacted readily with ethyllithium and phenyllithium prepared from ethyl bromide and phenylbromide with lithium to form tri-(1-ethyl-1-sila-2-azacyclopentane) (yield 45%) and tri-(1-phenyl-1-sila-2-azacyclopentane) (64%).

SECTION III

EXPERIMENTAL PROCEDURES

Reagent-grade 3-aminopropyltriethoxysilane was purchased from Union Carbide Corp.; hexamethylcyclotrisilazane was purchased from Peninsular ChemResearch Inc., Gainesville, Florida; lithium nitride was purchased from Foote Mineral Co., Exton, Pa. All were used without further purification.

Hexaphenyldisilazane was made in accordance with the method reported in our technical report AFML-TR-66-108.

Analytical glpc's were run on an F&M Model 500 Chromatograph using 0.25 in x 6 ft stainless steel column packed with 10% SE-30 on 60-80 Chromosorb W.

Elemental analyses, mass spectrometry, and nmr's were performed by the Analytical Branch, Air Force Materials Laboratory, WPAFB. Boiling points are uncorrected.

1. Preparation of 1,1-Diethoxy-2-(Triphenylsilyl)-1-Sila-2-Azacyclopentane

A mixture of 3-aminopropyltriethoxysilane (9.4 gm, 0.043 mol), hexaphenyldisilazane (17.0 gm, 0.032 mol) and ammonium chloride (trace) was heated under N_2 for 4 days. The NH_3 gas generated from the reaction was periodically neutralized by standard HCl solution (2.17 N) to the methyl red end point and a total of 0.016 mol of gas was collected. After the reaction mixture cooled to room temperature, hexane (30 ml) was added.

The insoluble hexaphenyldisilazane (3.0 gm) was filtered off and identified by infrared and mp. The filtrate was concentrated and vacuum distilled. The first fraction (15.6 gm) bp 110-165°/0.06 mm was identified by infrared as ethoxytriphenylsilane. One crystallization from hexane gave mp 58-63° (Reference 14, mp 65°). The second fraction (bp 168-222°/0.06 mm) was a yellow liquid which was taken-up in hexane and which gave 0.61 gm of the expected compound upon cooling. One crystallization from hexane gave a mp 99-101°.

Anal. Calcd for $(C_2H_5O)_2SiC_3H_6N-SiPh_3$

C, 69.28; H, 7.16; N, 3.20; Si, 12.92; mol wt 433.

Found: C, 68.82, 69.16; H, 7.21, 7.02; N, 3.31, 3.15; Si, 12.82, 12.67; mol wt 449, 452 (VP osmometry), 433 (mass spectrometry).

The residue from vacuum distillation was analyzed by mass spectrometry and found to contain compounds with m/e values of 304 ($Ph_3SiOC_2H_5$); 433 [$(C_2H_5O)_2SiC_3H_6N-SiPh_3$]; 387 (trimer D), 516 (tetramer E), and other values (assignment unsuccessful).

2. Attempt to Synthesize 1,1-Diethoxy-2-(Triphenylsilyl)-1-sila-2-Azacyclopentane.

A mixture of 1,1-diethoxy-2-(ethoxydimethylsilyl)-1-sila-2-azacyclopentane (11.1 gm, 0.04 mol) ethoxytriphenylsilane (12.2 gm, 0.04 mol) and ammonium bromide (trace) was heated to reflux for 24 hrs. No apparent reaction was observed. The mixture, after cooling, was heated again to reflux with a trace amount of lithium nitride. The distillate was periodically drained off by means of a Claissen head

takeoff. The reaction mixture was allowed to reflux overnight, a total of 4.7 gm of diethoxydimethylsilane was collected, yield 80%.

From the residue, only ethoxytriphenylsilane and hexaphenyldisiloxane were isolated.

3. Preparation of 1,1-Diethoxy-2-(Ethoxydimethylsilyl)-1-Sila-2-Azacyclopentane

A mixture of 3-aminopropyltriethoxysilane (96.5 gm, 0.44 mol), hexamethylcyclotrisilazane (32.0 gm, 0.15 mol), and ammonium bromide (0.062 gm) was heated to reflux for 3 days. The ammonia generated from the reaction was neutralized periodically with standard HCl solution (2.02 N). In the first 4 hrs, 0.40 mol of ammonia was collected and a total of 0.43 mol (100%) was obtained at the end of the reaction. The reaction mixture was distilled first at atmospheric pressure to obtain 10.0 gm of diethoxydimethylsilane, bp 110° (Reference 14, bp 113-114°). It was identified further by infrared. Vacuum distillation of the remaining substance gave first, the expected compound (60.0 gm) bp 70-73°/5 mm yield 49%. Glpc indicated that it was a pure sample.

Anal. Calcd for $(C_2H_5O)_2Si(C_3H_6N)SiMe_2(OC_2H_5)$

C, 47.65; H, 9.74; N, 5.05; Si, 20.22; mol wt 277.

Found: C, 48.01, 47.96; H, 9.89, 9.86; N, 4.92, 4.76; Si, 19.59, 19.97; mol wt 277 (mass spectrometry).

And, second, a viscous liquid (21 gm) bp 96-130° (5 mm). Glpc indicated that it was a mixture of many compounds. The major component isolated by glpc was identified at $(C_2H_5O)_2Si(C_3H_6N)Si^{OC_2H_5}(C_3H_6N)SiMe_2(OC_2H_5)$.

Anal. Calcd C, 47.29; H, 9.36; N, 6.90; Si, 20.68; mol wt 406.

Found: C, 46.92; H, 8.80; N, 6.83; Si, 20.30; mol wt 406 (mass spectrometry).

Its nmr spectrum had shown correct proton numbers and three different ethoxy groups.

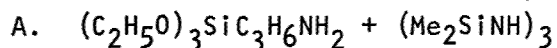
In a separate similar experiment, one component from the mixture isolated by glpc was identified as $(C_2H_5O)_2SiC_3H_6N-Me_2Si-O-Me_2SiOC_2H_5$.

Anal. Calcd C, 44.44; H, 9.68; N, 3.98; Si, 23.93; mol wt 351.

Found: C, 44.33, 44.49; H, 9.51, 9.56; N, 4.77, 4.77; Si, 23.51, 23.28; mol wt 351 (mass spectrometry).

The higher boiling substance left from these reactions was vacuum distilled and analyzed by mass spectrometry. Results are listed in Table I.

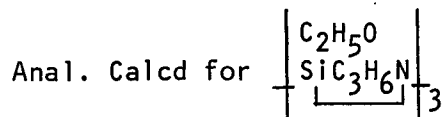
4. Preparation of Tri-(1-Ethoxy-1-Sila-2-Azacyclopentane) (Trimer D)



A mixture of 3-aminopropyltriethoxysilane (93.0 gm, 0.42 mol), hexamethylcyclotrisilazane (31.0 gm, 0.14 mol), and lithium nitride (0.53 gm, 0.015 mol) was heated under N_2 to initiate the reaction. The ammonia generated from the reaction was periodically neutralized by standard HCl solution to the methyl red end point. In 5 days, 0.43 mol of ammonia was collected. At the end of ammonia generation, the reaction mixture was distilled under N_2 to removed the diethoxydimethylsilane (58 gm, yield 93.5%) bp 110-115° (Reference 14, 113-114°).

70-114

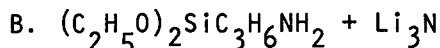
The remaining reaction mixture, upon vacuum distillation gave 14.0 gm (yield 26%) of the expected compound, bp 120-140°/0.05 mm. Sample for analysis was purified by glpc.



C, 46.51; H, 8.53; N, 10.85; Si, 21.71; mol wt 387.

Found: C, 46.41, 46.04; H, 9.19, 8.81; N, 10.59, 10.56; Si, 21.55, 21.47; mol wt 387 (mass spectrometry).

Its nmr indicated the correct proton numbers and slightly splitting ethoxy groups.



Into warm 3-aminopropyltriethoxysilane (151.0 gm, 0.68 mol) in a three necked flask, equipped with mechanical stirrer, condenser, and N_2 inlet and outlet tubes, was added slowly 14.3 gm (0.41 mol) of lithium nitride. Reaction took place with generation of heat and ammonia (not collected). At times the outside heating had to be removed to ensure a smooth reaction. After complete addition, the reaction mixture was heated to reflux for 2 hrs, then cooled to room temperature. The lithium ethoxide was precipitated by the addition of 250 ml of xylene and removed by filtration under N_2 . The filtrate was distilled at atmospheric pressure to remove the solvent. Vacuum distillation gave 28.0 gm of the expected compound bp 115-138°/0.05 mm. One vacuum distillation gave 24.0 gm (yield 30%) of relatively pure sample. Mol wt 387 (mass spectrometry) infrared and glpc retention time were identical to the sample obtained from A.

5. Condensation Reaction of 1,1-Diethoxy-2-(Ethoxydimethylsilyl)-1-Sila-2-Azacyclopentane

1,1-diethoxy-2-(ethoxydimethylsilyl)-1-sila-2-azacyclopentane (29 gm) and lithium nitride (0.0050 gm) were heated together under N_2 overnight. The reaction mixture, after cooling to room temperature, was vacuum distilled. There was obtained 14.2 gm of starting material and a small amount of trimer D identified by the retention time of glpc identical to that of the above sample.

6. Alcoholysis of Trimer D

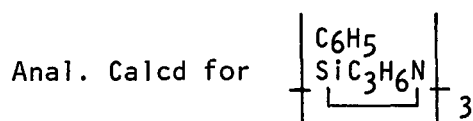
A mixture of anhydrous ethyl alcohol (50 ml) and a few drops of benzene was distilled under N_2 until the distillation temperature reached 78° . After the alcohol was cooled to room temperature, trimer D (11.0 gm) was added. The reaction mixture was heated to reflux for 10 hrs. Upon vacuum distillation, 4.8 gm of 3-aminopropyltriethoxysilane was obtained (yield 25%).

In another similar experiment, a small piece of lithium was put into the alcohol before the addition of 12.5 gm of trimer D. Upon refluxing for one hour and vacuum distillation, it gave 17.0 gm of pure 3-aminopropyltriethoxysilane (yield 79%). Its identify was proved by infrared and glpc retention time.

7. Arylation of Trimer D

An excess of phenylbromide (35.0 gm) was added slowly to a mixture of trimer D (20.6 gm, 0.05 mol), 250 ml of anhydrous ether, and shredded lithium (2.24 gm, 0.32 mol). The reaction took place with the

generation of heat and proceeded smoothly during the addition (3 hrs). The lithium salts were precipitated by the addition of 200 ml of benzene and removed by filtration under N_2 . The filtrate, after removal of the benzene, was taken up in 100 ml of petroleum ether (bp 30-60°) and cooled. There was obtained 13.7 gm of tri-(1-phenyl-1-sila-2-azacyclopentane) mp 129-131°. Concentration of the mother liquid gave an additional 2.8 gm of the compound, yield 64%. One crystallization from hexane gave a pure sample mp 133°.

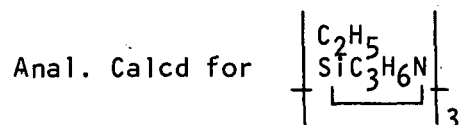


C, 67.08; H, 6.83; N, 8.70; Si, 17.39; mol wt 483.

Found: C 67.20, 67.03; H, 6.76, 6.86; N, 8.88, 8.90; Si, 17.31, 17.27; mol wt 483 (mass spectrometry).

8. Alkylation of Trimer D

Ethyl bromide instead of phenyl bromide was used in the above reaction. After the removal of lithium salts, the filtrate was concentrated and vacuum distilled. A fraction 5.5 gm (yield 45%) distilled over at 136-156° (0.1 mm) and was shown by glpc to be a pure sample of the expected compound.



C, 53.10; H, 9.73; N, 12.39; Si, 24.77; mol wt 339.

Found: C, 52.97; H, 10.15; N, 11.86; Si, 25.13; mol wt 339 (mass spectrometry).

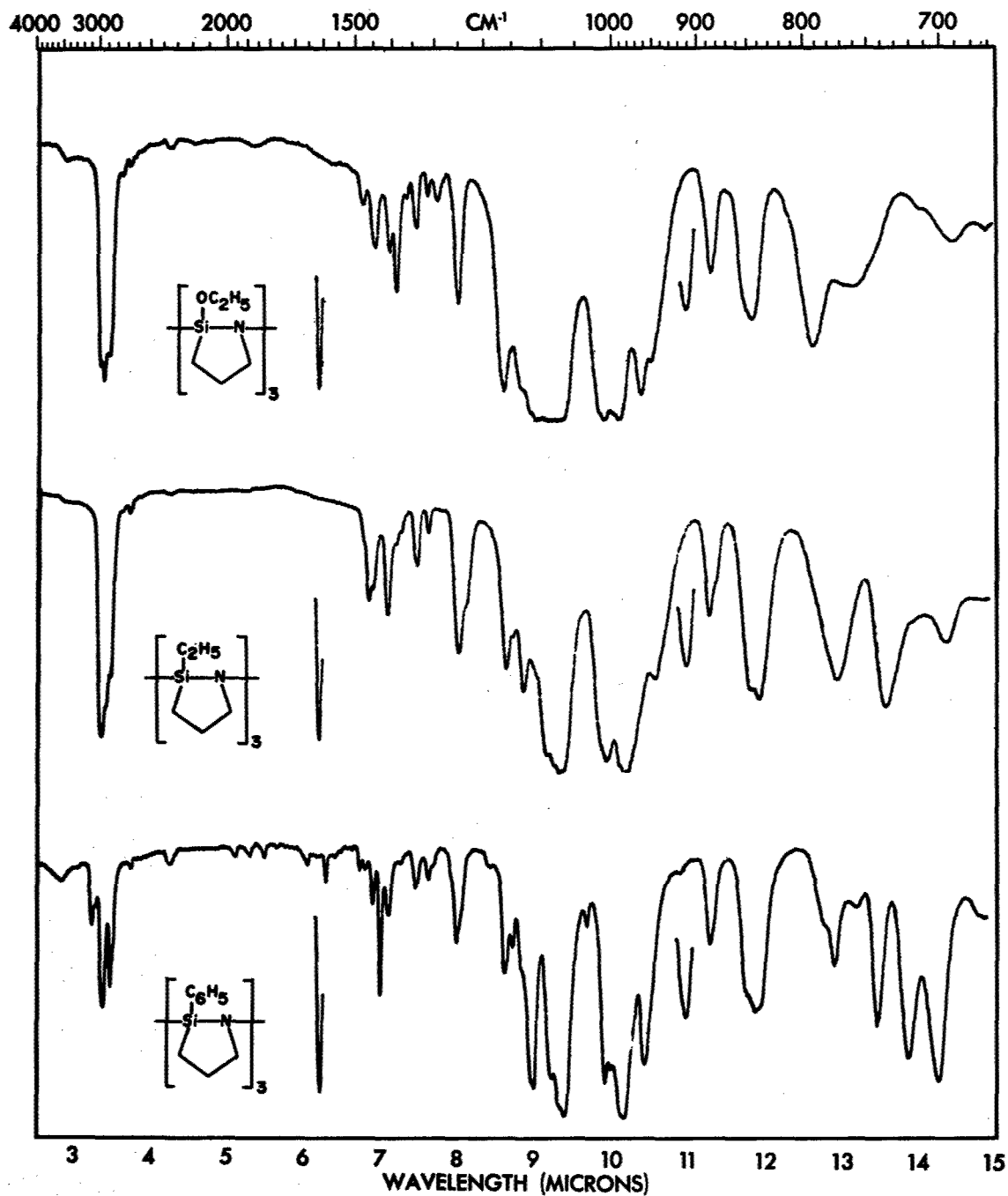


Figure 1. Infrared Spectra of Tri-(1-Ethoxy-1-Sila-2-Azacyclopentane), Tri-(1-Ethyl-1-Sila-2-Azacyclopentane), and Tri-(1-Phenyl-1-Sila-2-Azacyclopentane)

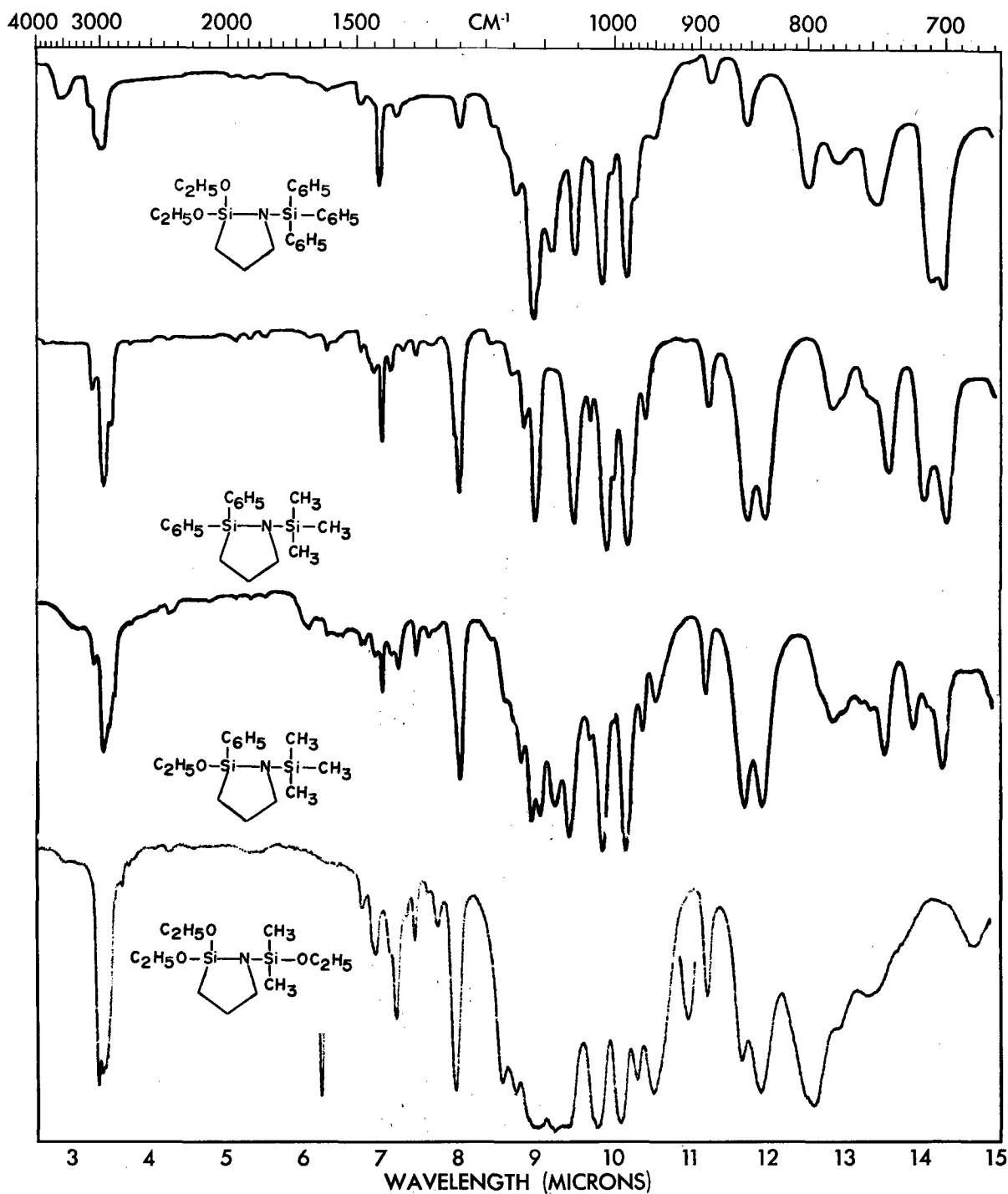


Figure 2. Infrared Spectra of 1,1-Diethoxy-2-(Triphenylsilyl)-1-Sila-2-Azacyclopentane, 1,1-Diphenyl-2-(Trimethylsilyl)-1-Sila-2-Azacyclopentane, 1-Phenyl-1-Ethoxy-2-(Trimethylsilyl)-1-Sila-2-Azacyclopentane, and 1,1-Diethoxy-2-(Ethoxydimethylsilyl)-1-Sila-2-Azacyclopentane.

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13. ABSTRACT <p>New ring-compounds: 1,1-diethoxy-2-(triphenylsilyl)-1-sila-2-azacyclopentane, 1,1-diethoxy-2-(ethoxydimethylsilyl)-1-sila-2-azacyclopentane, and tri-(1-ethoxy-1-sila-2-azacyclopentane) were prepared and isolated. The third compound reacts with ethyl alcohol to form 3-aminopropyltriethoxysilane; and with phenyllithium and ethyllithium to form tri-(1-phenyl-1-sila-2-azacyclopentane) and tri-(1-ethyl-1-sila-2-azacyclopentane), respectively.</p>			

14.

KEY WORDS

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1,1-diethoxy-2-(triphenylsilyl)-1-sila-
2-azacyclopentane

1,1-diethoxy-2-(ethoxydimethylsilyl)-1-sila-
2-azacyclopentane

tri-(1-ethoxy-1-sila-2-azacyclopentane)

tri-(1-phenyl-1-sila-2-azacyclopentane)

tri-(1-ethyl-1-sila-2-azacyclopentane)