$$dS/d[Cl^-] = (S/[Cl])(2\bar{n} - 9)/5 \qquad (5)$$

where S is the solubility (M) of the complex salt, $K_S = [(CH_3)_4N^+]^3[SbCl_4^-]^2[Cl^-]$, β_n is the over-all formation constant for $SbCl_n^{3-n} = [SbCl_n^{3-n}]/\{[Sb^{+3}][Cl^-]^n\}$, K_n is the step formation constant for formation of $SbCl_n^{3-n}$ from $SbCl_n^{4-n}$, $\bar{n}$ is the average ligand number for all $SbCl_n^{3-n}$ in solution, and $[Cl^-]_0$ is the chloride ion concentration (M) in the solvent before saturating it with solute.

Observations are summarized in Table II. The corrected values of $[Cl^-]$ were found by a method of successive approximations using eq. 4 and slopes of the solubility curve obtained at the data points. The corrected slope of -0.2 at $[Cl^-]_0 = 0$ is required by the equation. The minimum in the solubility curve denotes $\bar{n} = 4.5$, indicating that species with $n = 4.0$ must be present in significant concentrations. The value of $\bar{n}$ calculated from slopes of the curve using eq. 5 varies from 3.5 to 5.5.

TABLE II

SOLUBILITY DATA FOR $[(CH_3)_4N]_3Sb_2Cl_9$ AT VARIOUS $[Cl^-]$ AT 25°[a]

$[Cl^-]_0$, M	$[Cl^-]$,[b] M	S, M	$10^3S^{5/2}[Cl^-]^{1/2}$
0.00	0.150	0.0785	0.662
0.10	0.215	0.0700	0.598
0.20	0.300	0.0630	0.554
0.30	0.369	0.0585	0.513
0.40	0.465	0.0553	0.492
0.50	0.559	0.0525	0.472
0.60	0.656	0.0500	0.463
0.70	0.752	0.0492	0.466
0.80	0.852	0.0480	0.459
0.90	0.938	0.0473	0.469
1.00	1.031	0.0470	0.480
1.10	1.110	0.0475	0.511
1.20	1.20	0.0473	0.530
1.30	1.30	0.0460	0.490
1.50	1.50	0.0464	0.552
2.00	1.98	0.0470	0.694
3.00	2.95	0.0504	1.008
4.00	3.90	0.0550	1.508

[a] All solutions 4 M in H^+ assuming H_2SO_4 is a 1:1 electrolyte.
[b] Corrected value using eq. 4.

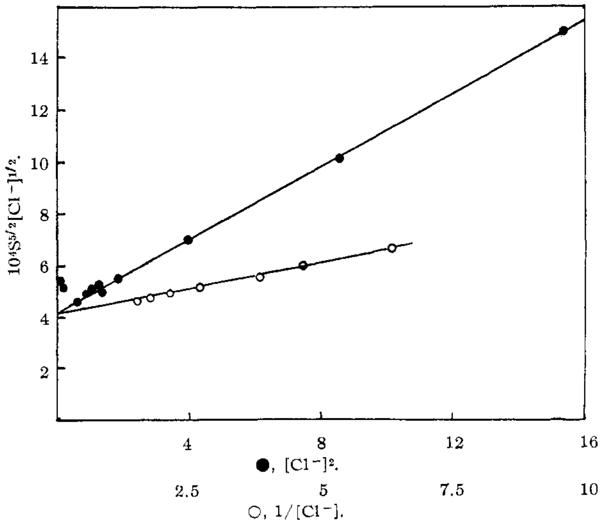


Figure 1.—Plots of the left-hand side of eq. 3 *vs.* $[Cl^-]^2$ (black dots) showing linearity at higher chloride ion concentrations, and *vs.* $1/[Cl^-]$ (open circles) showing linearity at lower chloride ion concentrations. The two plots should have a common intercept.

Figure 1 shows that a plot of the left-hand side of eq. 3 *vs.* $[Cl^-]^2$ gives a straight line over the range $0.8\ M < [Cl^-] \leqslant 4.0\ M$. This indicates that in this region the formation of $SbCl_6^{-3}$ from $SbCl_4^-$ is the dominant equilibrium in solution. A plot of the same quantity *vs.* $1/[Cl^-]$ gives a straight line over the range $0.15\ M < [Cl^-] < 0.8\ M$. This indicates that in this region formation of $SbCl_3$ from $SbCl_4^-$ is the dominant equilibrium in solution. No evidence of the formation of $SbCl_5^{-2}$ is found in these data. The two plots have a common intercept of 4.05×10^{-4} equal to $K_S^{1/2}/10.4$. From the slopes and the intercept the following constants are calculated: $K_5K_6 = [SbCl_6^{-3}]/\{[SbCl_4^-]\cdot[Cl^{-2}]\} = 0.17$; $K_4 = [SbCl_4^-]/\{[SbCl_3][Cl^-]\} = 10$; $K_S = 1.8 \times 10^{-5}$.

The constants are concentration constants and may be subject to activity corrections in view of the large change in solvent from nearly pure 4.0 M sulfuric acid to 4.0 M hydrochloric acid. Due to the relatively high solubility of the salt, the ionic strength is not quite constant but varies from 4.20 to 4.33. Also K_4 has been derived from data in the region where corrections using eq. 4 are large. However, there does appear to be strong evidence that the complex $SbCl_n^{3-n}$ of highest n is $SbCl_6^{-3}$ and not $SbCl_4^-$ as suggested by polarographic data.[3] The suggestion derived from the polarographic data that $SbCl_4^-$ is the only Sb(III) species present between 0.5 and 6.0 M HCl is contravened by observed changes in the absorption spectrum over this range. The possibility that the negatively charged complexes might be protonated is ruled out by the fact that partial substitution of lithium chloride for hydrochloric acid at constant $[Cl^-]$ has no effect on the absorption spectrum of antimony(III). The equilibrium between $SbCl_4^-$ and $SbCl_6^{-3}$ is analogous to that found for bismuth complexes.[5]

Acknowledgment.—This work was supported by grants from the Swarthmore College Faculty Research Fund and from the National Science Foundation.

CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
THE OHIO STATE UNIVERSITY, COLUMBUS, OHIO 43210

Palladium(II) Complexes of Secondary and Tertiary N-Substituted Ethylenediamines

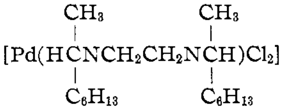
BY DEVON W. MEEK

Received September 17, 1964

Although simple tertiary amines are reported not to form stable transition metal complexes, the coordination stability of a tertiary amino group increases mark-

TABLE I

PROPERTIES AND ANALYSES OF THE Pd(II) COMPLEXES

Complex	Color	Yield, %	Molar conductance, cm.²/ohm M[a]	Analyses,[c] %: C		H		N		Cl	
				Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.	Found
$[Pd(CH_3NHCH_2CH_2NHCH_3)Cl_2]$	Light yellow	90	<2[b]	18.09	18.27	4.56	4.44	10.55	10.72		
$[Pd\{(CH_3)_2NCH_2CH_2N(CH_3)_2\}Cl_2]$	Yellow	85	<2[b]	24.54	24.77	5.49	5.56	9.54	9.10		
 $[Pd(H\overset{CH_3}{\underset{C_6H_{13}}{C}}NCH_2CH_2N\overset{CH_3}{\underset{C_6H_{13}}{C}}H)Cl_2]$	Light yellow	85	Insol.	46.79	46.72	8.73	8.52	6.07	6.15		
$[Pd(C_6H_5NHCH_2CH_2NHC_6H_5)Cl_2]$	Red-orange	95	<2[b]	43.15	43.04	4.14	4.19	7.19	7.14		
$[Pd(CH_3NHCH_2CH_2NHCH_3)_2]Cl_2$	White	60	182	27.17	26.95	6.84	7.09	15.85	15.63	20.07	19.84
$[Pd(CH_3NHCH_2CH_2NHCH_3)_2](NO_3)_2$	White	85	180	23.62	23.43	5.95	5.77	20.68	20.44		
$[Pd\{(CH_3)_2NCH_2CH_2N(CH_3)_2\}](NO_3)_2$	Light yellow	70	185	31.13	30.73	6.75	6.86	18.17	18.40		
$[Pd(C_6H_5NHCH_2CH_2NHC_6H_5)_2]Cl_2$	Light yellow	30	. . .	55.86	55.86	5.36	5.51	9.31	9.50	11.79	11.64

[a] The conductivity measurements were made on 1×10^{-3} M solutions in nitromethane. Typical molar conductance values for 1:1 and 2:1 electrolytes in 10^{-3} M nitromethane solutions are in the ranges 85–95 and 175–190 cm.²/ohm M, respectively. For example, $[(C_6H_5)_3AsCH_3]Cl = 85$ and $[Ni(DMSO)_6](ClO_4)_2 = 186$. [b] The resistance was too high to read accurately on the conductivity bridge. The values are upper limits. [c] Microanalyses were performed by Galbraith Laboratories Inc., Knoxville, Tenn.

edly when incorporated into a chelate ring.[1–4] For example, N,N,N′,N′-tetramethylethylenediamine forms 1:1 complexes with the strong Lewis acids $SiCl_4$,[5,6] $GeCl_4$,[6] TiF_4,[7] and "$Be(CH_3)_2$".[8]

Recently we[9,10] have prepared several stable metal complexes containing different secondary and tertiary amine ligands. Preparation and characterization of $[Ni(N,N'\text{-dimethylen})_3](ClO_4)_2$[9,11] suggested that the repulsion between N-methyl groups on adjacent N-substituted ethylenediamine ligands may not be as severe as originally claimed by Mann and Watson.[12] Indeed, examination of accurate molecular models indicated that two molecules of N,N,N′,N′-tetramethylethylenediamine actually could chelate to Pd(II) in a square-planar configuration. The present paper reports the preparation, characterization, and spectra of this and other mono- and bis-N-substituted ethylenediamine complexes of Pd(II).

Experimental

Materials.—The N-alkylethylenediamines were dried over barium oxide, decanted, and then distilled from fresh barium oxide. Potassium tetranitratopalladate(II), $K_2[Pd(NO_3)_4]$, was prepared by addition of a stoichiometric amount of KNO_3 to a solution of palladium in concentrated nitric acid.[13] Reagent grade methanol was refluxed over magnesium and then fractionated (b.p. 64.5°). Reagent grade N,N-dimethylformamide was dried for 24 hr. over barium oxide and then fractionated (b.p. 55° (20 mm.)). The nitromethane was washed with 5% aqueous $NaHCO_3$, dried over anhydrous Na_2SO_4, and then fractionated (b.p. 100–101°). Acetonitrile was purified according to the literature procedure.[14] Other reagent grade materials were used without further treatment.

Conductivity Measurements.—Conductivity determinations were performed at 24.6° using an Industrial Instruments, Inc., Model RC-16B conductivity bridge. The measurements were obtained in approximately 10^{-3} M solutions of nitromethane. The colors of the resulting solutions were the same as the colors of the original solids.

Spectral Measurements.—Spectra of finely ground solid samples were obtained with a Beckman DU spectrophotometer equipped with a standard Beckman reflectance attachment using U.S.P. $MgCO_3$ as a blank. The use of plastic cover plates permitted examination over the range from 1100 to 280 mμ. Readings were taken at 10-mμ intervals except in the regions of absorption maxima where readings were taken every 5 mμ. Absorption spectra of the complexes in solution were obtained with a Cary Model 14 recording spectrophotometer in matched sets of 1- and 5-cm. quartz cells.

The infrared spectra were obtained with a Perkin-Elmer Model 337 grating spectrophotometer with the samples mounted as KBr disks and in Nujol mulls. The polystyrene spectrum was used for calibration.

General Preparations of the Complexes. (a) **Pd(ligand)Cl_2, where ligand = N,N′-dimethylen, N,N,N′,N′-tetramethylen, N,N′-bis(1-methylheptyl)en, and N,N′-diphenylen.**[11]—To a filtered solution of palladium(II) chloride (0.89 g., 0.005 mole) dissolved in 25 ml. of DMF was added 0.005 mole of the appropriate ethylenediamine while stirring. The yellow to orange crystals, which formed slowly after complete mixing, were collected on a filter, washed with ether, and dried. The compounds were recrystallized from hot N,N-dimethylformamide; yields 85–95%.

(b) **$[Pd(N,N'\text{-dimethylen})_2](NO_3)_2$ and $[Pd(N,N,N',N'\text{-tetramethylen})_2](NO_3)_2$.**—$K_2[Pd(NO_3)_4]$[13] (2.16 g., 5 mmoles) dissolved readily in 25 ml. of N,N-dimethylformamide to produce a deep

---

(1) F. G. Mann and W. J. Pope, *J. Chem. Soc.*, 489 (1926).

(2) W. Gauss, P. Moser, and G. Schwarzenbach, *Helv. Chim. Acta*, **35**, 2359 (1952).

(3) G. Schwarzenbach and P. Moser, *ibid.*, **36**, 581 (1953).

(4) F. G. Mann and H. R. Watson, *J. Chem. Soc.*, 3950 (1957).

(5) I. R. Beatie, T. Gibson, M. Webster, and G. P. McQuillan, *ibid.*, 238 (1964).

(6) J. E. Fergusson, D. R. Grant, R. H. Hickford, and C. J. Wilkins, *ibid.*, 99 (1959).

(7) E. L. Muetterties, *J. Am. Chem. Soc.*, **82**, 1082 (1960).

(8) G. E. Coates and S. I. E. Green, *J. Chem. Soc.*, 3340 (1962).

(9) S. F. Pavkovic and D. W. Meek, *Inorg. Chem.*, **4**, 20 (1965).

(10) D. W. Meek and S. A. Ehrhardt, *ibid.*, in press.

(11) The following abbreviations are used throughout this paper for the ligands: en = ethylenediamine, N,N′-dimethylen = N,N′-dimethylethylenediamine, N,N,N′,N′-tetramethylen = N,N,N′,N′-tetramethylethylenediamine, N,N′-bis(1-methylheptyl)en = N,N′-bis(1-methylheptyl)ethylenediamine, and N,N′-diphenylen = N,N′-diphenylethylenediamine.

(12) F. G. Mann and H. R. Watson, *J. Chem. Soc.*, 2772 (1958).

(13) R. Eskenazi, J. Raskovan, and R. Levitus, *Chem. Ind.* (London) 1327 (1962).

(14) W. S. Muney and J. F. Coetzee, *J. Phys. Chem.*, **66**, 89 (1962).

red solution. The solution was filtered to remove potassium nitrate and then 4–5 ml. (large excess) of the appropriate ethylenediamine was added while stirring. Each of the solutions turned from red to yellow on complete mixing and crystals separated within 5 min. The white N,N′-dimethylethylenediamine complex was recrystallized from DMF, whereas the light yellow N,N,N′,N′-tetramethylethylenediamine complex was recrystallized from an acetonitrile–ether mixture.

(c) **[Pd(N,N′-dimethylen)$_2$]Cl$_2$ and [Pd(N,N′-diphenylen)$_2$]Cl$_2$.**—In each case a filtered solution of palladium(II) chloride (1.78 g., 0.01 mole) dissolved in 40 ml. of N,N-dimethylformamide was added to a DMF solution containing 100% excess of the ligand. The reactions were quite exothermic and white crystals precipitated during the addition. Each of the compounds was recrystallized from a mixture of ethanol and the respective ligand.

Discussion

Mann and Watson[12] reported that N,N,N′,N′-tetramethylethylenediamine and 1,4-dimethylpiperazine form the stable compounds M(amine)X$_2$ (where M = Pd, Pt, Ir; X = Cl or Br). However, they found no evidence for a bis-amine complex. Failure of Co^{3+}, Rh^{3+}, and Ni^{2+} to coordinate with N,N,N′,N′-tetramethylethylenediamine or 1,4-dimethylpiperazine and nonformation of a bis-amine complex with the platinum metals was attributed to repulsion between methyl groups on adjacent coordinated ligands. In contrast to the above results, the bis-N,N,N′,N′-tetramethylen complex [Pd{(CH$_3$)$_2$NCH$_2$CH$_2$N(CH$_3$)$_2$}$_2$](NO$_3$)$_2$ was prepared in 70% yield. The compound represents the first example of a transition metal ion bonded with four alkyl tertiary amino groups.

In order to discuss the steric requirements of the dimethylamino group, it is necessary to establish that both N,N,N′,N′-tetramethylethylenediamine molecules are chelated in the bis-amine complex of Pd(NO$_3$)$_2$. The electronic absorption spectra provide the most convincing evidence. Since a monodentate ligand produces a weaker ligand field than is produced when the same ligand atom is incorporated into a chelate, the λ_{max} of the hypothetical [Pd(N,N,N′,N′-tetramethylen)$_2$(NO$_3$)$_2$] (with the two amines coordinated only at one end) should occur at lower energy than for the [Pd(N,N,N′,N′-tetramethylen)Cl$_2$] complex. Instead, the λ_{max} of Pd(NO$_3$)$_2$·2N,N,N′,N′-tetramethylen is at much higher energy than for [Pd(N,N,N′,N′-tetramethylen)Cl$_2$]. A comparison of the diffuse reflectance spectrum of the solid with the spectrum of the complex dissolved in methanol shows that no structural change occurs on dissolution. In addition, both the conductance value in nitromethane and the infrared spectrum of the solid complex are consistent with ionic nitrate groups. Thus, all of the data indicate that both amine molecules are strongly chelated to the Pd(II) ion in a planar structure. Therefore, the steric requirements of four dimethylamino groups do not prohibit their coordination to Pd(II) as postulated by Mann and Watson.[4]

Further support for the chelation of both amines is derived from the data of the bis-N,N′-dimethylen complexes of PdCl$_2$ and Pd(NO$_3$)$_2$. Both the chloride and the nitrate complexes exhibit identical λ_{max} values and similar ϵ_{max} values in solution (Table II). The positions of the λ_{max} in the solution spectra are in excellent agreement with the reflectance spectra. These data, coupled with the behavior of the compounds as bi-univalent electrolytes, indicate that the anions are not coordinated and that the four-coordinate, square-planar [Pd(N,N′-dimethylen)$_2$]$^{2+}$ cation is present in both complexes.

Table II

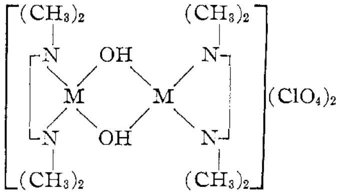
Spectral Data of the Complexes

Complex	λ_{max} (solid), mμ	λ_{max} (soln.), mμ^b
[Pd(N,N′-dimethylen)Cl$_2$]	385	388 (2990)^c
	475 sh^a	
[Pd(N,N,N′,N′-tetramethylen)Cl$_2$]	395	392 (3210)^c
	487 sh	
[Pd{N,N′-bis(1-methylheptyl)en}Cl$_2$]	390	Insol.
	475 sh	
[Pd(N,N′-diphenylen)Cl$_2$]	417	390 (495)^d
	512 sh	512 (59)
[Pd(N,N′-dimethylen)$_2$]Cl$_2$	295	294 (500)^d
	350 sh	350 (70)
[Pd(N,N′-dimethylen)$_2$](NO$_3$)$_2$	295	294 (480)^d
	350 sh	350 (68)
[Pd(N,N,N′,N′-tetramethylen)$_2$](NO$_3$)$_2$	337	326 (610)^e
	425 sh	425 (56)

[a] sh, shoulder on the main band. [b] The molar extinction coefficients, calculated from the equation log $I/I_0 = \epsilon Cl$, are given in parentheses. [c] DMF solution. [d] Ethanol solution. [e] Methanol solution.

Nonformation of bis-N,N,N′,N′-tetramethylethylenediamine complexes of platinum metal halides[12] can be attributed to the competition between the halide and the amine for coordination to the metal ion. This is substantiated by the fact that the bis-N,N,N′,N′-tetramethylen complex of PdCl$_2$ did not form even with a large excess of amine, whereas the bis-amine complex is obtained in good yield with Pd(NO$_3$)$_2$.

In contrast to the formation of the bis-N,N,N′,N′-tetramethylen complex of Pd(II), we[10] have been unable to isolate analogous bis-N,N,N′,N′-tetramethylen complexes of the smaller Cu^{2+} and Ni^{2+} ions, even by use of nitrate or perchlorate salts. Dinuclear compounds of the type



are found instead of the bis-amine complexes. Perhaps an ion of certain critical size (*i.e.*, a second or third row transition metal ion) is necessary for coordination of two N,N,N′,N′-tetramethylen molecules.

The position of the λ_{max} in the reflectance spectra of the complexes (Table II) produces the decreasing order of metal–ligand interaction: N,N′-dimethylen > N,N′-bis(1-methylheptyl)en > N,N,N′,N′-tetramethylen > N,N′-diphenylen. The same relative series is repeated for the three bis-amine complexes. The relative order of N,N′-dimethylen and N,N,N′,N′-tetramethylen suggests that steric considerations prevail over inductive factors[10] in the N,N,N′,N′-tetramethylen complexes since one would expect the increased electron density at the nitrogen atoms of N,N,N′,N′-tetramethylen to rank this ligand highest in coordinating ability, if the inductive property were the only consideration. Both steric and inductive factors operate in the same direction in determining the relative position of N,N′-diphenylen in the series.

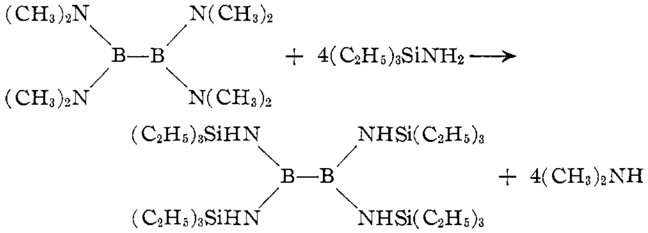
CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY,
DUKE UNIVERSITY, DURHAM, NORTH CAROLINA

The Preparation of Tetrakis(triethylsilylamino)diborane(4)[1]

BY A. L. ALSOBROOK,[2] A. L. COLLINS, AND R. L. WELLS

Received September 8, 1964

Tetra(amino)diborane(4) compounds have been synthesized and studied in some detail[3]; however, there has been no report made of a compound of this type containing silylamine groups. We wish to report the first preparation of such a compound, namely tetrakis(triethylsilylamino)diborane(4), by the direct transamination[3a,b] of tetrakis(dimethylamino)diborane(4) with triethylsilylamine.



Experimental

Reagents and Apparatus.—Research quantities of tetrakis(dimethylamino)diborane(4) were supplied by U. S. Borax Corp., Anaheim, Calif. Triethylsilyamine was prepared from triethylchlorosilane and liquid ammonia.[4] Volatile compounds were separated using a standard high vacuum line.[5] Elemental analyses were done by Schwarzkopf Laboratories, Woodside, N. Y. Infrared spectra were obtained using Perkin-Elmer 137 or 237 spectrophotometers. Proton nuclear magnetic resonance spectra were obtained using a Varian A-60 spectrometer. Mass spectra were obtained using a Bendix Model 14 time-of-flight mass spectrometer. An F and M Model 500 gas chromatograph equipped with a 2-ft. column containing 30% silicone gum rubber SE 30 substrate on 60/80 mesh Chromosorb-P was used for analytical purposes. Separation and isolation of products were accomplished using an Aerograph Autoprep Model A-70 gas chromatograph equipped with a 5-ft. column containing the same packing as noted above.

Preparation of $[(C_2H_5)_3SiNH]_4B_2$.—A 500-ml., one-neck, round-bottom flask was equipped with a Friedrich condenser and a side-arm gas inlet tube which was connected to a source of dry nitrogen. The condenser was connected to two traps, the first trap being cooled to $-80°$ and the second trap being cooled to $-195°$. Finally, the outlet of the trap at $-195°$ was connected to a mercury bubbler. A continuous flow of nitrogen was maintained in the system during the reaction.

For a typical reaction, 32.0 g. (0.240 mole) of triethylsilylamine, 8.50 g. (0.042 mole) of tetrakis(dimethylamino)diborane(4), and 300 ml. of toluene were added to the reaction flask. The reaction mixture was refluxed for 17 days, during which time the traps at low temperature were monitored with all volatile materials being collected. Dimethylamine (69.2 mmoles) and ammonia (38.6 mmoles) were isolated from the traps, these compounds being identified by their characteristic vapor pressures and/or infrared spectra. The solvent, some excess triethylsilylamine, and additional dimethylamine (5.0 mmoles) were removed from the reaction mixture at low pressures. Further separations of the solvent-free reaction mixture were made using gas chromatography techniques, and two major products with nearly identical infrared spectra were isolated. The product fraction having the longest retention time on the column was found to be tetrakis(triethylsilylamino)diborane(4) (yield 20%, estimated from gas chromatogram). The other major product has not been completely characterized.

Anal. Calcd. for $[(C_2H_5)_3SiNH]_4B_2$: C, 53.11; H, 11.89; B, 3.98; N, 10.32; Si, 20.70. Found: C, 53.40; H, 11.47; B, 3.97; N, 10.41; Si, 20.83.

The peak of highest mass in the mass spectrum appeared at 542.6 and 542.8 ± 0.5 as compared to two internal standards in two separate determinations. These data are in good agreement with the calculated molecular weight of 542.8. Also, the fragmentation pattern observed in the mass spectrum is consistent for the molecule $[(C_2H_5)_3SiNH]_4B_2$.

The infrared spectrum of the compound contained the following characteristic absorption bands: 3380 $cm.^{-1}$ (N–H), 838 $cm.^{-1}$ (Si–N), and 1313 $cm.^{-1}$ (B–N).

The proton n.m.r. spectrum was quite complex in the region 0.8–2.0 p.p.m. (*vs.* tetramethylsilane reference in a sealed capillary tube) and was not readily interpreted; however it was shown to be nearly the same as the spectrum of triethylsilylamine.

Discussion

Although the reaction time is quite long, and the yield is relatively low, it has been established that tetrakis(triethylsilylamino)diborane(4) is one of the major products formed in the reaction of triethylsilylamine with tetrakis(dimethylamino)diborane(4).

As expected, dimethylamine and ammonia were also generated during the course of the reaction. The dimethylamine is the primary volatile product in the transamination reaction; whereas the ammonia is a secondary product that is produced by the following reactions: (1) the condensation of triethylsilylamine to hexaethyldisilazane[6]; (2) the transamination reaction[7,8] of dimethylamine with triethylsilylamine to give N,N-dimethyltriethylsilylamine.[9] The infrared spectrum of one of the fractions separated from the reaction mixture gave evidence for the formation of

---

[1] $[(C_2H_5)_3SiNH]_2B–B[HNSi(C_2H_5)_3]_2$; name approved by *Chemical Abstracts*. See: "The Naming and Indexing of Chemical Compounds," *Chem. Abstr.*, **56**, paragraph 288 (1962).

[2] N.S.F. undergraduate research program participant, Sept. 1963–Aug. 1964.

[3] For example see the following and the references cited therein: (a) R. J. Brotherton, A. L. McCloskey, L. L. Petterson, and H. Steinberg, *J. Am. Chem. Soc.*, **82**, 6242 (1960); (b) M. P. Brown, A. E. Dann, D. W. Hunt, and H. B. Silver, *J. Chem. Soc.*, 4648 (1962); (c) L. L. Petterson and R. J. Brotherton, *Inorg. Chem.*, **2**, 423 (1963); (d) S. C. Malhotra, *ibid.*, **3**, 862 (1964).

[4] D. L. Bailey, L. H. Sommer, and F. C. Whitmore, *J. Am. Chem. Soc.*, **70**, 435 (1948).

[5] R. T. Sanderson, "Vacuum Manipulation of Volatile Compounds," John Wiley and Sons, Inc., New York, N. Y., 1948.

[6] R. M. Pike, *Rec. trav. chim.*, **81**, 28 (1962).

[7] R. Fessenden and D. F. Crowe, *J. Org. Chem.*, **26**, 4638 (1961).

[8] R. Fessenden and J. S. Fessenden, *Chem. Rev.*, **61**, 361 (1961).

[9] K. A. Andrianov, S. A. Golubstov, and E. A. Semenova, *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk*, 47 (1958); *Chem. Abstr.*, **52**, 11734 (1958).