UNION COLLEGE - GRADUATE STUDIES

Schenectady, New York

A STUDY OF SODIUM PHENYLSILICONATE

A thesis presented to the Committee on Graduate Studies and the Department of Chemistry of Union College, Schenectady, New York, in partial fulfillment of the requirements for the degree of Master of Science.

by John Henry Daane MS 1966

By John H. Daane

Approved by Kevork V. Nahabedian
Thesis advisor

Approved by
Committee on Graduate Studies

Date May 26, 1966

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DEDICATION

This thesis is dedicated to my wife Gail for her encouragement and patience along the way.

To

ACKNOWLEDGEMENTS

I am indebted to Dr. K. V. Nahabedian of Union College and University for his interest and help. Likewise, I am indebted to Dr. J. F. Brown, Jr., of the General Electric Research and Development Center for suggesting the problem, supplying spectra and samples, helpful discussions and for the opportunity to examine his manuscript on the subject prior to its publication. I would thank the people of the General Electric Silicone Products Department for their assistance: Drs. A. Torkelson and R. Mansfield for permission to use company facilities and materials;
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ABSTRACT

The application of modern silicone chemistry knowledge and new analytical techniques to the elucidation of the structure of the hitherto uncharacterized, crystalline salt, sodium phenysiliconate, has been achieved. This salt, produced in 1914 by J. A. Meads and F. S. Kipping,. had been left structurally uncharacterized. This thesis builds upon the early work by applying to the problem the recent knowledge developed by J. F. Brown, Jr., and coworkers at the General Electric Research Laboratory.

Derivatives of the salt were made and characterized by a variety of analytical techniques. The silanols produced by the acidification of the sodium phenylsiliconate are shown to yield the same derivatives as those made from *cis*-*cis*-*cis*-2,4,6,8-tetraphenycyclotetrasiloxanetetrol. The nature of the sodium phenylsiliconate, as dictated by the experimental results, is discussed. The use of gel permeation chromatography has made it relatively easy to distinguish between the production of a monomeric or polymeric reaction product.

I. INTRODUCTION

Early in the development of silicone chemistry, before the day of the polymer chemist, various compounds were produced, some of whose structures were not elucidated (1). By applying the knowledge and instrumental techniques available today, structural analyses of these compounds are now readily accomplished. This work attacks one of these previously uncharacterized compounds, namely, the salt sodium phenylsiliconate (2) using infrared spectroscopy, proton magnetic resonance, gel permeation chromatography, molecular weight determination, melting point, etc. on derivatives of the salt. The structure of the salt is narrowed down to several possibilities.

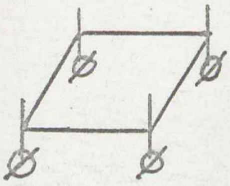
II. HISTORICAL BACKGROUND

In 1914 and 1915, Meads and Kipping reported the preparation of several compounds obtained from trichlorosilanes by various hydrolysis procedures. They referred to these products as siliconic acids (2,3). Hydrolyses were performed in ice water, steam, cold and warm dilute ammonium hydroxide and an aqueous solution of potassium hydroxide. Molecular weight determinations confirmed their suspicions that the products were polymeric materials.

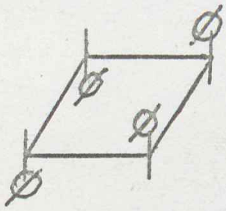
Needlelike, colorless crystals were obtained by reacting either the alcohol-soluble fraction of the phenyltrichlorosilane hydrolyzate obtained using cold water or the alcohol-insoluble hydrolyzate obtained using steam with an ethanol solution of sodium ethoxide. Both products yielded identical sodium analyses consistent with the empirical formula for a siliconate salt, $NaC_6H_5SiO_2$. However, the molecular weights of the soluble hydrolysis products themselves indicated a mixture of polymers in the trimer to hexamer range. No further work on the crystals is recorded.

A recent study of the hydrolysis of phenyltrichlorosilane in cold aqueous acetone has resulted in the preparation, separation and identification of a series of cyclic phenylsiloxanes (4). A sequence of steps was proposed for the polycondensations which accounted for these products. The product of the first step, phenylsilanetriol, was isolated previously by Tyler (5). The first condensation product of phenylsilanetriol is 1,3-diphenyldisiloxanetetrol, which was prepared and characterized by Brown and Slusarczuk (6). A subsequent condensation product of this, whose isolation and identification was the most significant finding so far as this thesis is concerned, was *cis*-*cis*-*cis*-2,4,6,8-tetraphenylcyclotetrasiloxanetetrol.

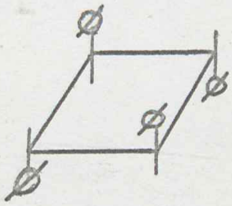
The alternative configurations that are possible for a cyclic tetramer tetrol are:



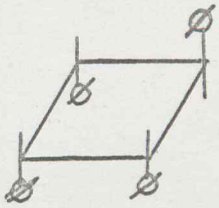
cis-*cis*-*cis* tetrol



cis-*trans*-*cis* tetrol

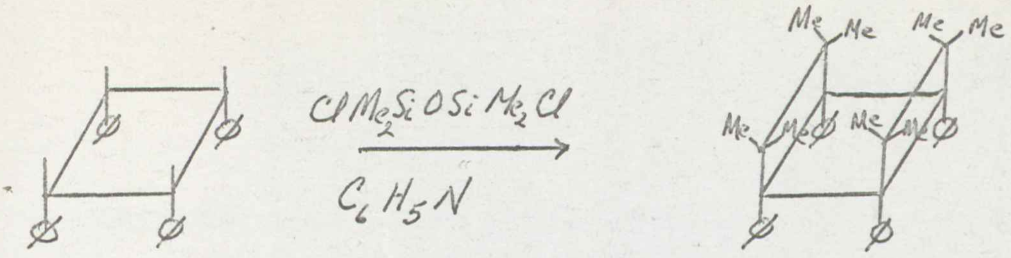


trans-*trans*-*trans* tetrol

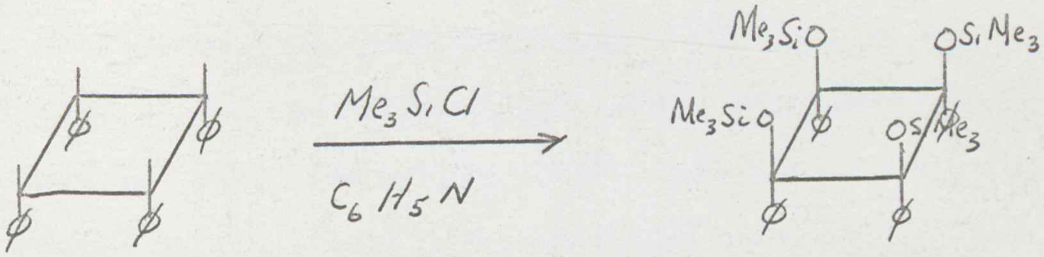


cis-*trans*-*trans* tetrol

where the above parallelograms represent cyclotetrasiloxane rings with the silicon atoms at each corner connected through oxygen atoms, i.e. siloxane linkages. The vertical lines represent hydroxyl groups which are on the opposite side of the ring from the phenyl groups. Brown prepared the 1,3-dichlorotetramethyldisiloxane derivative of his condensation product and characterized it as *cis*-*syn*-*cis*-octamethyl-1,3,9,11-tetraphenyltricyclo$[9.5.1.1^{3,9}]$octasiloxane (4):



(Polycyclic siloxanes are numbered according to the methods used for carbon compounds of the same nature (7). Thus numbers are assigned to the ring atoms by beginning at a bridgehead and moving along the bridges (8). The longest bridge is numbered first, the second largest next, etc.) Brown also produced and characterized the corresponding trimethylmonochlorosilane derivative, *cis*-*cis*-*cis*-2,4,6,8-tetrakis(trimethylsiloxy)tetraphenylcyclosiloxane (9):



III. DESCRIPTION OF APPARATUS

Infrared spectra were obtained on a Perkin-Elmer Model 21 Infrared Spectrophotometer. A Perkin-Elmer Model 137 Infrared Spectrophotometer was used during the course of the work for rapid checks. A Varian A-60 Proton Magnetic Resonance Spectrometer was used to obtain the proton magnetic spectrum. Gel permeation chromatograms were obtained on a Water's Associates, Inc. Gel Permeation Chromatograph equipped with a 10^4, 8×10^3, 3×10^3 and 250 A^o column combination set with toluene as solvent. Number average molecular weights were obtained on a Mechrolab Vapor Pressure Osmometer using acetone as solvent.

Gel permeation chromatography (GPC) is a recent addition to the tools available to the polymer chemist for polymer characterization (10). Instrumentally, GPC is analogous in several ways to vapor phase chromatography (VPC). A solution of sample is carried through a column or series of columns where separation is to be achieved. The fluid then passes on in the stream to a differential detector, in this case a refractometer, where comparison with the solvent itself is made. The differential signal is fed to a strip chart recorder where appearance versus retention time or elution volume may be ascertained.

GPC utilizes the difference in permeability shown by a swollen polystyrene gel bead toward molecular species which differ in size (11). Since larger species can diffuse into less of the heteroporous gel volume, the larger species emerge first. Calibration with substances of the same molecular type and having known sizes permits molecular size analysis barring association effects. Generally a plot of the logarithm of molecular weight versus elution volume yields a straight line over a considerable range, i.e. several orders of magnitude.

A simplified diagram of the Water's Gel Permeation Chromatograph flow system is shown in Figure I. The instrument operates in the following manner: the solvent stream, after first being degassed in a heated chamber, is divided into two streams. One stream consists of a closed loop between the solvent reservoir, reference column and detector. The sample stream flows past a loop sample valve whereby the stream can be diverted through the loop to introduce the sample (in solution already) into the system.

The sample is transmitted through the analytical columns and to the detector. The effluent passes into a five milliliter self-syphon whose draining initiates a photocell circuit signal to the recorder. This signal serves as a measure of the flow rate (5 ml./count) and is used for determining retention volumes of samples.

The detector is a highly sensitive differential refractometer capable of measuring to within one thousandth of a refractive index unit (± .0000005) (12). The chart recorder gives a record of the difference in refractive index between the solvent and the sample solution as well as the elution volume of the sample.

Vapor pressure osmometry is a misnomer applied to an instrument manufactured by Mechrolab Inc., a division of Hewlett Packard. It is based on the measurement of the temperature difference between a pair of matched thermistor bead detectors situated in a thermostatted solvent saturated vapor chamber. It is assumed that the temperature difference generated by a solvent droplet on one bead and a sample solution droplet on the other bead is determined by the vapor pressure lowering which is proportional to the molar concentration of the solute. A Wheatstone bridge circuit is used to measure this difference. Calibration with known molecular weight materials leads to a correlation factor, different for each solvent and proportional in sensitivity to the solvent's heat of vaporization (13). The value obtained by this method is the number average molecular weight ($\overline{M}_n$) which is simply the number of molecules times their molecular weight divided by the total number of molecules.

There has been controversy over the operating principle of the vapor pressure osmometer. Is is a transport or a thermodynamic process? One well known polymer characterization expert goes along with the transport process (14). Recently the manufacturer, who has claimed it is a thermodynamic process, has published an article defending its stand and acknowledging that the technique only approaches (but closely) a true thermodynamic measure of vapor pressure lowering (15). Both sides, however, agree as to its accuracy and usefulness.

IV. EXPERIMENTAL PROCEDURE

Initially, much effort was spent in seeking to achieve the production of the colorless, needlelike crystals of sodium phenylsiliconate for this study. Methanol and denatured ethanol were found to be unsuitable as solvents. A polymeric material rather than a crystalline material separated out as evidenced by the extremely high concentrations required to get a deposit. Methanol was initially tried because a carefully prepared sodium methoxide-methanol solution was available. A variety of molar ratios of sodium methoxide versus phenyltrichlorosilane hydrolyzate were tried in methanol but with no success. These experiments are not described here.

The following procedure is essentially a duplication of that used by Meads and Kipping.

Preparation of Sodium Phenylsiliconate. Phenyltrichlorosilane (146.9 g.) of 99.0 % purity was slowly added via a Drierite capped dropping funnel to two liters of a rapidly stirred ice slurry. The aqueous phase was split in half and each half extracted with one pound of diethyl ether. The ether portions were each washed twice with 500 ml. of cold distilled water. The second wash in each case was neutral to pH paper.

The combined ether solution was partially evaporated by immersing a two liter beaker containing the ether solution in a dish of warm water in a hood. The remaining solution was placed in a weighed 250 ml. beaker which was then placed in a vacuum desiccator and pumped on through a liquid nitrogen trap via a rotary oil pump. The polymeric mass resulting after most of the ether had evaporated began to froth as stated in Kipping's paper. Pumping had to be done intermittently until no final frothing occurred. The final product was 40.2 g. (43.7 %) of flaky phenyltrichlorosilane hydrolyzate.

After repeated attempts to produce the sodium phenylsiliconate, the following combination produced a mass of needlelike crystals. Five grams of the phenyltrichlorosilane hydrolyzate was dissolved in 100 ml. of absolute ethanol. A threefold molar excess of 3.07 M methanolic sodium methoxide (36.5 ml.) was added to the solution. The mixture was placed in a vacuum desiccator along with potassium hydroxide pellets in a separate beaker and then evacuated.

Two days later, the solution had reduced to a volume of 90 ml. and crystals were forming in the beaker. The following day, with the volume down to 70 ml., the solution contained a large mass of colorless, needlelike crystals. These were filtered in a dry nitrogen purged dry bag and washed with cold ethanol. Approximately 8.6 g. of solvated crystalline product was obtained.

Vacuum drying at 120°C for 6 hrs. was required for complete removal of solvent. Sodium analyses were performed on the crystals by titration of the base generated upon aqueous dissolution of the salt. The sodium analysis thus obtained was 14.2 ± 0.3 % ($[C_6H_5SiO_2Na]_x$ requires 14.4 % Na; $HO(C_6H_5SiO_2Na)_4H$ requires 14.0 % Na). Microscopic examination of the salt, now present as a fine powder after drying, revealed a crystalline structure.

Hydrolysis of Sodium Phenylsiliconate. In a typical generation of the silanol derivative, 1.037 g. (.0065 moles Na) of the sodium phenylsiliconate was added slowly to a rapidly magnetically stirred 100 ml. diethyl ether-30 ml. dilute acetic acid (.00714 moles, 10 % excess) solution two phase system in a 250-ml. Erlenmeyer flask. This procedure should satisfy the requirement that the preparation of stable silanols be done at neutral or near neutral conditions (16). The phases were separated after 1/2 hr. and the ether was vacuum evaporated off. The residue was washed with carbon disulfide, filtered and the solvent vacuum evaporated, leaving 0.340 g. (38 % yield) of product.

One sample of silanol generated by this procedure (Preparation A) was recrystallized from 20:80 acetone-water solution at 0°C to give a product melting at 174-179°C. An infrared spectrum, run in a 9:1 carbon disulfide-acetone solution against a compensated cell (Figure II) shows silanol bands at 3300, 930 and 880 cm.$^{-1}$ The ν_a SiOSi band (in phase chain vibrational mode) occurred at 1090 cm.$^{-1}$ with a shoulder at 1050 cm.$^{-1}$ A Debye-Scherrer powder pattern showed x-ray reflections at 17 (s), 9(d), 8(d), 4.8(s), 4.5(s) and 4.05(w) A^{o}.

A second sample, generated by seemingly the same procedure, (Preparation B) shows bands at 3400, 1100 and 925 cm.$^{-1}$ in addition to uncompensated solvent bands near 1085, 910 and 860 cm.$^{-1}$ (Figure III). There was no shoulder at 1050 cm.$^{-1}$ in this spectrum.

1,3-Diphenyldisiloxanetetrol is reported to melt at 114°C which shows ν_a SiOSi at 1095 cm.$^{-1}$ and silanol bands at 915 cm.$^{-1}$ and 850 cm.$^{-1}$ (6). The ν_a SiOSi band checks with that in Preparation A, however, the silanol band positions were observed to be quite different, and the melting point is too far removed.

The all <u>cis</u>-cyclotetrasiloxane tetrol is known in at least two crystalline modifications, one of which does melt at 173-179°C, however neither form corresponded in its x-ray pattern to Preparation A. However, the reported infrared spectrum (silanol bands at 3400 and 925 cm.$^{-1}$ and ν_a SiOSi at 1102 cm.$^{-1}$) is identical for Preparation B.

<u>Trimethylmonochlorosilane Derivative</u>. A dispersion of 0.2785 g. of the vacuum dried sodium phenylsiliconate

in 30 ml. of dry benzene was stirred rapidly by a Teflon coated, magnetic activated stirrer bar. A stoichiometric amount of trimethylmonochlorosilane (0.326 cc.) of 99+ % purity was added. The 125 ml. Erlenmeyer flask was stoppered and stirred for 1/2 hr. at slightly above room temperature. The solution was filtered to remove the sodium chloride formed.

The benzene was evaporated by a nitrogen purge. The number average molecular weight of the product was determined (Mechrolab osmometer, acetone) and found to be 1050 ± 10 %. A gel permeation chromatographic examination (Figure IV) in toluene solvent was performed to check on the molecular weight distribution. It revealed a higher molecular weight and broader distribution than Brown's cis-cis-cis-2,4,6,8-tetrakis(trimethylsiloxy)tetraphenylcyclosiloxane compound. The width at half-height was found to be 2.0 counts (10 ml.) at a retention volume of 146 ml. whereas material obtained from Brown (9) eluted at 151.5 ml. with a width at half-height of one count (5 ml.). 1,1,3,3,5,5,7,7-octaphenylcyclotetrasiloxane eluted at 155.8 ml. with a width at half-height of one count (5 ml.).

The peak component on the GPC was trapped for infrared identification which was run as a carbon disulfide solution (Figure V). The SiOSi band was observed at 1054 $cm.^{-1}$ with indication of a band of lesser intensity at 1110 $cm.^{-1}$

The relative intensities of the 693 cm.$^{-1}$ and 1247 cm.$^{-1}$ bands indicate a twofold higher $C_6H_5SiO_{3/2}$ to $(CH_3)_3SiO$ molar ratio than in the cis-cis-cis-2,4,6,8-tetrakis (trimethylsiloxy)tetraphenylcyclosiloxane.

Generation of a trimethylsilylated derivative was accomplished by reacting 0.172 g. of silanol (Preparation A) with a twofold molar excess of an equimolar mixture of trimethylmonochlorosilane (0.40 cc.) and pyridine (0.26 cc.) in dry benzene (50 cc.) contained in a 125-ml. Erlenmeyer flask. The flask was stoppered and the solution stirred magnetically with a Teflon coated stirrer bar 3 hrs. Pyridine hydrochloride was filtered out and the benzene solution washed three times with equal volumes of water. The benzene, hexamethyldisiloxane (b.p. 105°C) formed from the hydrolysis of excess trimethylmonochlorosilane and the water remaining was removed by vacuum distillation at 20 mm. and 80°C, leaving a solid residue.

The trimethylsilylated derivative of the silanol (0.1795 g., 68.5 % yield) when examined on the gel permeation chromatograph revealed a major peak at 151.5 ml. elution volume with a higher molecular weight tail (Figure VI). It was recrystallized from a 50:50 ether-methanol mixture. GPC examination (Figure VII) then revealed a single peak at 151.5 ml. elution volume and a width at half-height of one count (5 ml.). The $\overline{M}_n$ was measured by the technique described earlier

(Mechrolab osmometer, acetone) and found to be 900 ± 10 % (840 theory). An infrared spectrum (Figure VIII) obtained as a carbon disulfide solution showed ν_aSiOSi bands at 1050 $cm.^{-1}$ and another at 1100 $cm.^{-1}$ This entire 2-15μ spectrum matched one run on an authentic sample of *cis*-*cis*-*cis*-2,4,6,8-tetrakis(trimethylsiloxy)tetraphenylcyclosiloxane kindly furnished by J. F. Brown, Jr., (9). Melting points were determined on both samples. Our sample melted at 198°C while Dr. Brown's melted at 196°C (Fisher-Johns Melting Point Apparatus). Thus the molecular weight, infrared spectrum, GPC and melting points reveal the identity of these compounds.

1,3-Dichlorotetramethyldisiloxane Derivative of cis-cis-cis-2,4,6,8-Tetraphenylcyclotetrasiloxanetetrol. To 0.2916 g. of silanol, (Preparation B) in a 125-ml. Erlenmeyer flask containing 12.65 ml. diethyl ether was added a stoichiometric amount of 1,3-dichlorotetramethyldisiloxane (0.219 g.) and pyridine (0.159 g.). The flask was stoppered and the mixture stirred magnetically for one hour. The solution was filtered to remove pyridine hydrochloride and a nitrogen purge used to evaporate the ether. A colorless fluid remained as the product.

A 50:50 ether-methanol mixture was used to dissolve the fluid and induce crystallization. Upon standing overnight with partial evaporation of the solvent mixture, crystallization occurred. The crystals (37 mg., 8.7 % yield) were filtered off and washed with methanol. After vacuum drying, an infrared spectrum in carbon disulfide solution and a proton magnetic resonance spectrum in carbon tetrachloride with cyclohexane as internal standard were recorded. The integral of the phenyl and methyl protons was run.

Infrared analysis (Figure IX) showed two ν_a SiOSi bands at 1102 $cm.^{-1}$ (vs) and 1060 $cm.^{-1}$ (m) with indications of perhaps a shoulder at 1090 $cm.^{-1}$ Brown reported (4) bands at 1062 (m), 1093 (sh) and 1105 (vs) $cm.^{-1}$ for cis-syn-cis-octamethyl-1,3,9,11-tetraphenyltricyclo$[9.5.1.1^{3,9}]$-octasiloxane. The proton magnetic resonance spectrum (Figure X) showed complex aromatic absorption in the τ2.4-2.9 region with two methyl absorptions in the τ 9.5-10.0 region (τ 9.90 and τ 9.65 as compared to Brown's of τ9.89 and τ 9.69). The chemical shift of cyclohexane relative to tetramethylsilane was taken to be τ1.43 ppm. The integral of the phenyl and methyl protons was determined. The relative number of protons found were phenyl -5 to methyl -6.

The derivative was recrystallized from a 50:50 ether-methanol solvent. Its melting point determined on a benzoic acid (m.p. 122°C) calibrated Fisher-Johns melting point apparatus was 126°C (Brown reported 124°C and 129°C). Carbon hydrogen analysis yielded 5.6 % hydrogen and 47.4 %

carbon (theoretical - 5.45 % hydrogen and 47.4 % carbon). Gel permeation chromatography yielded an elution volume of 154.5 ml. and a width of one count at half-height, suggesting a uniform molecular weight.

Since the analytical data (infrared spectrum, proton magnetic resonance spectrum, melting point, carbon hydrogen analysis) on our derivative matched that of cis-syn-cis-octamethyl-1,3,9,11-tetraphenyltricyclo[9.5.1.$1^{3,9}$]octasiloxane, it proves the identity of our derivatives.

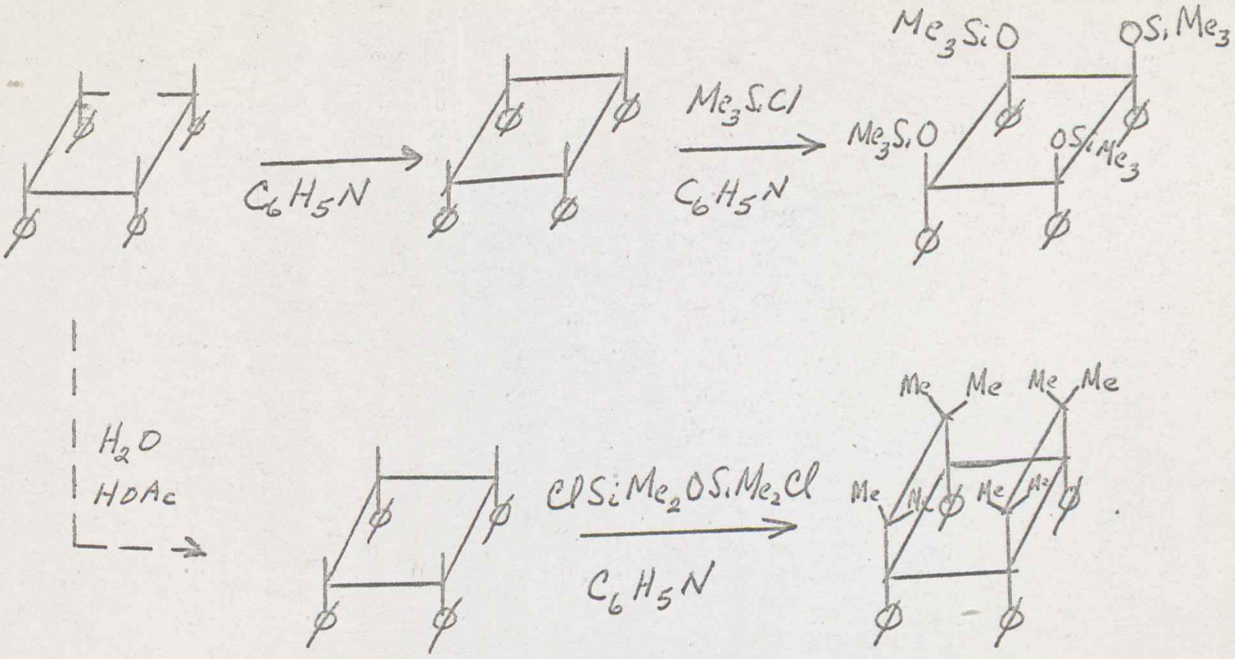
V. RESULTS AND DISCUSSION

The production of colorless needlelike crystals in ethanol solvent apparently depends on the solubility of the sodium salt. Perhaps the sodium methoxide requires sufficient time for siloxane bond rearrangement and then a concentration increase to initiate crystallization. The crystals produced in ethanol in this study would appear to be the same as those produced by Meads and Kipping. This is based on the time to crystallize under similar conditions, crystal form and sodium analysis.

The products of the direct reaction of trimethylmonochlorosilane with the sodium phenylsiliconate appear to be polymeric, based on the GPC results. The relative GPC detector response and infrared spectrum indicate a higher $C_6H_5SiO_{3/2}$ to $(CH_3)_3SiO$ molar ratio than one. The infrared spectrum and molecular weight suggest an average composition of $(C_6H_5SiO_{3/2})_6[(CH_3)_3SiO]_4$. Apparently condensation in the presence of excess trimethylmonochlorosilane takes place. This result was verified by varying the molar ratio of the chlorosilane to siliconate used but the same (infrared spectrum and GPC) material resulted in each case.

Acidification of the salt, on different occasions, gave at least two different silanols. One of these appears to correspond spectrally to <u>cis</u>-<u>cis</u>-<u>cis</u>-2,4,6,8-tetraphenylcyclotetrasiloxanetetrol (Preparation B) while the other (Preparation A) was different in spectrum and x-ray, although similar in melting point. Both silanols, however, gave derivatives identical to those from cis-cis-cis-2,4,6,8-tetraphenylcyclotetrasiloxanetetrol. The unknown silanol (Preparation A) treated with trimethylmonochlorosilane gave cis-cis-cis-2,4,6,8-tetrakis(trimethylsiloxy)tetraphenylcyclosiloxane. This indicates the presence of some structure which can yield cis-cis-cis-2,4,6,8-tetrakis(trimethylsiloxy) tetraphenylcyclosiloxane in the process of derivatization. The other sample of silanol (Preparation B), treated with 1,3-dichlorotetramethyldisiloxane gave cis-syn-cis-octamethyl-1,3,9,11-tetraphenyltricyclo$[9.5.1.1^{3,9}]$octasiloxane, as previously reported.

There are two possible structures for the unknown silanol (Preparation A). It could be a stereoisomer which undergoes epimerization under the conditions of the derivatization. The other possibility, which we favor, is that it is the open chain tetramer, 1,3,5,7-tetraphenyltetrasiloxanehexol. This would be expected to cyclize very readily under the conditions of either the silanol preparation or its reaction with trimethylmonochlorosilane and pyridine:



This conclusion would imply that the original salt had the composition $(\mathrm{HO}\left[\mathrm{phSiO_2Na}\right]_4\mathrm{H})$ rather than $(\mathrm{phSiO_2Na})_x$ as reported by Meads and Kipping. Both compositions are within experimental error of the sodium analysis.

Schematic of Water's Gel Permeation
Chromatograph Flow

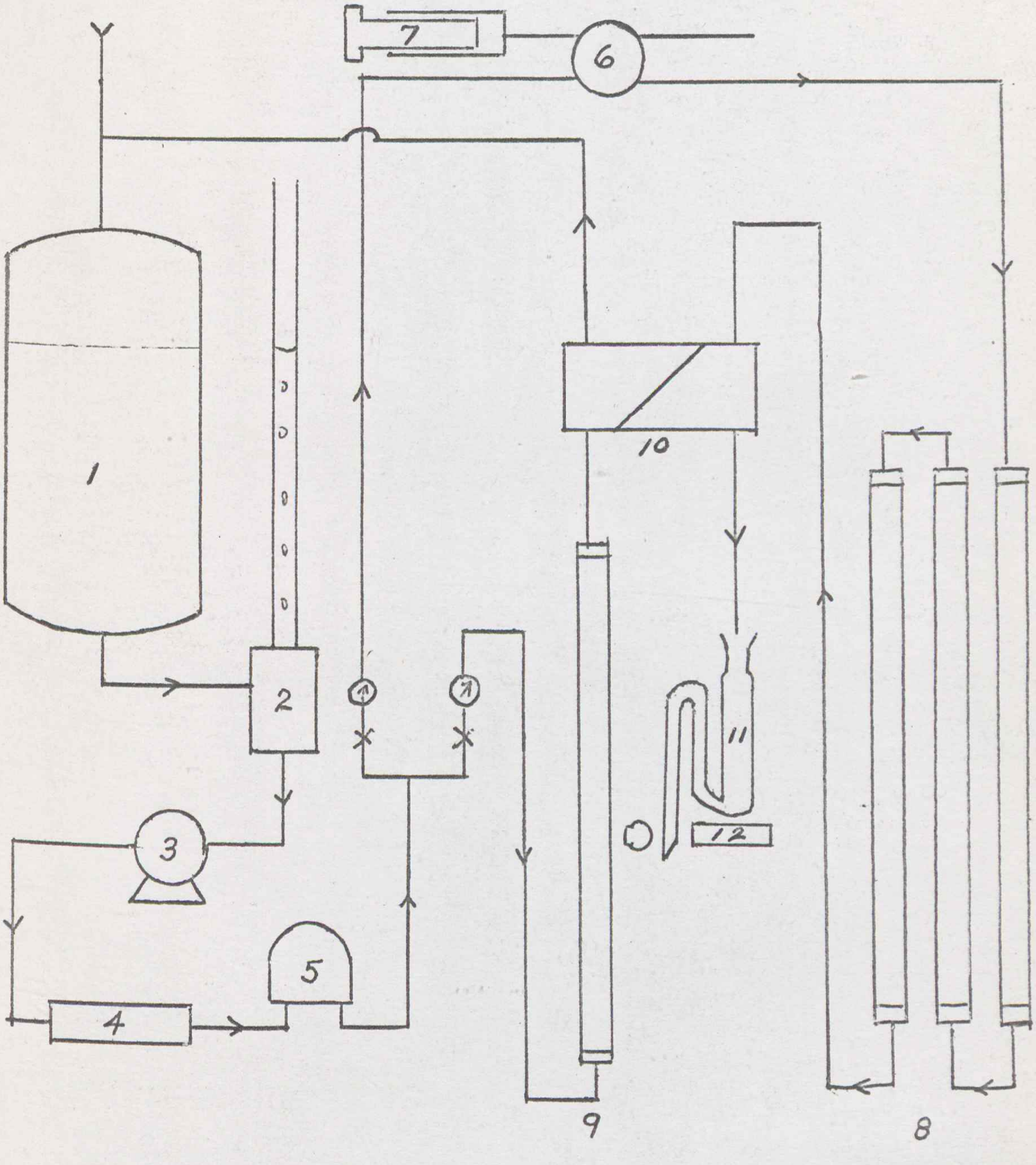
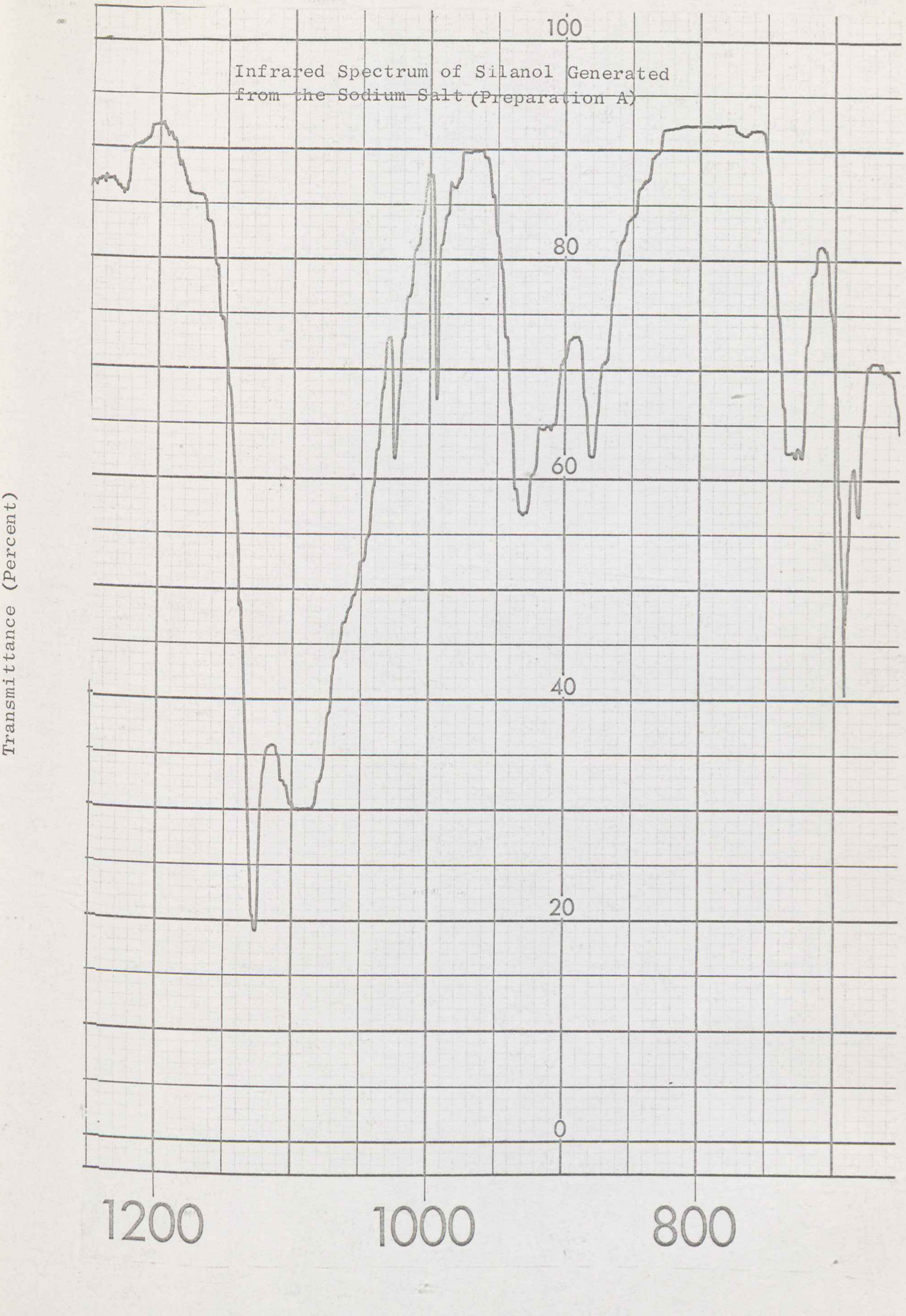


FIGURE I

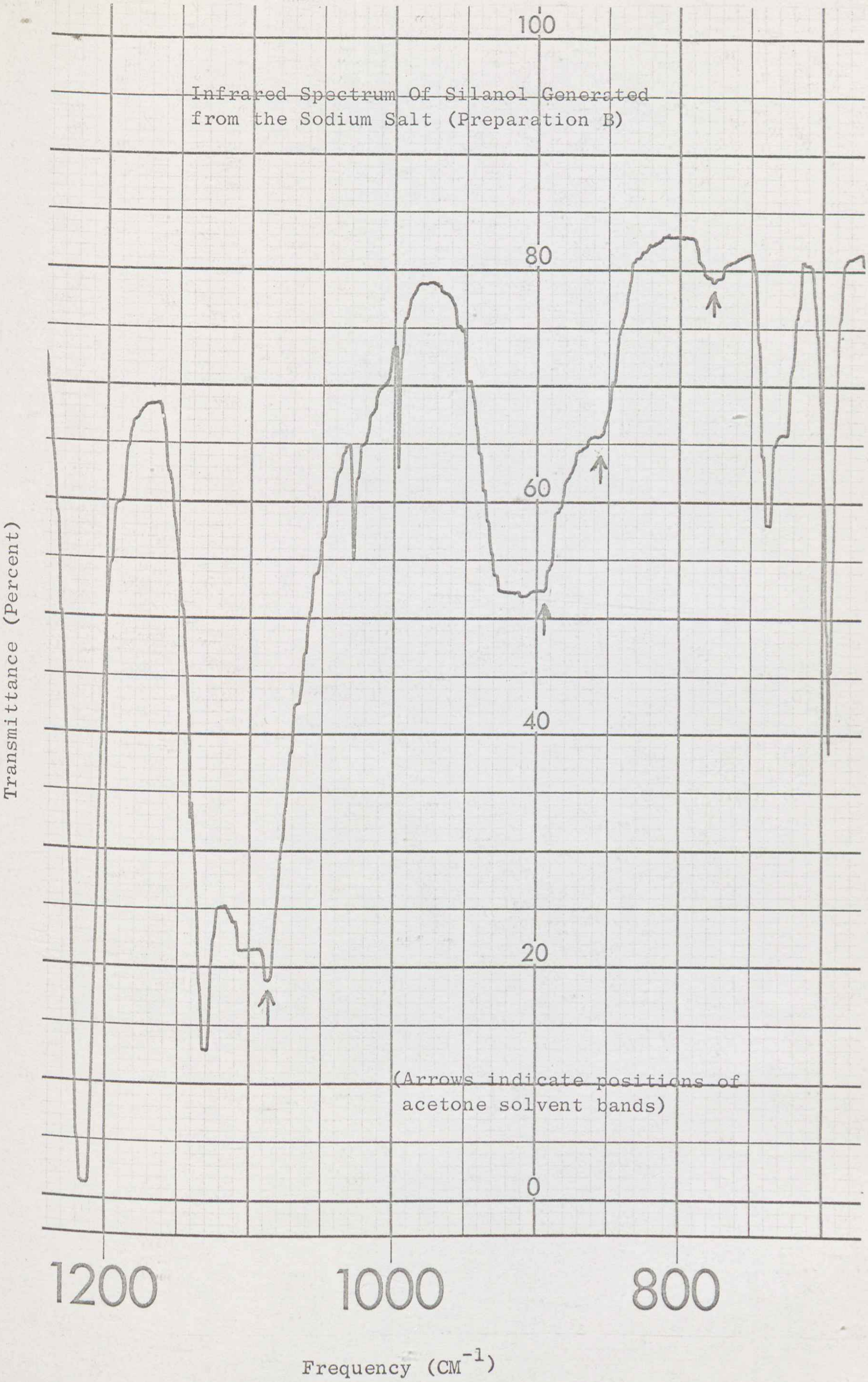
1. Solvent Reservoir
2. Degasser
3. Pump
4. Filter
5. Pressure Regulator
6. Sample Valve
7. Sample Syringe
8. Analytical Columns
9. Reference Column
10. Refractometer
11. Syphon
12. Photo Cell

FIGURE II



Infrared Spectrum of Silanol Generated from the Sodium Salt (Preparation A)

FIGURE III



Infrared Spectrum Of Silanol Generated from the Sodium Salt (Preparation B)

(Arrows indicate positions of acetone solvent bands)

Gel Permeation Chromatogram of the Direct Reaction Product of the Sodium Salt with Trimethylmonochlorosilane

FIGURE IV

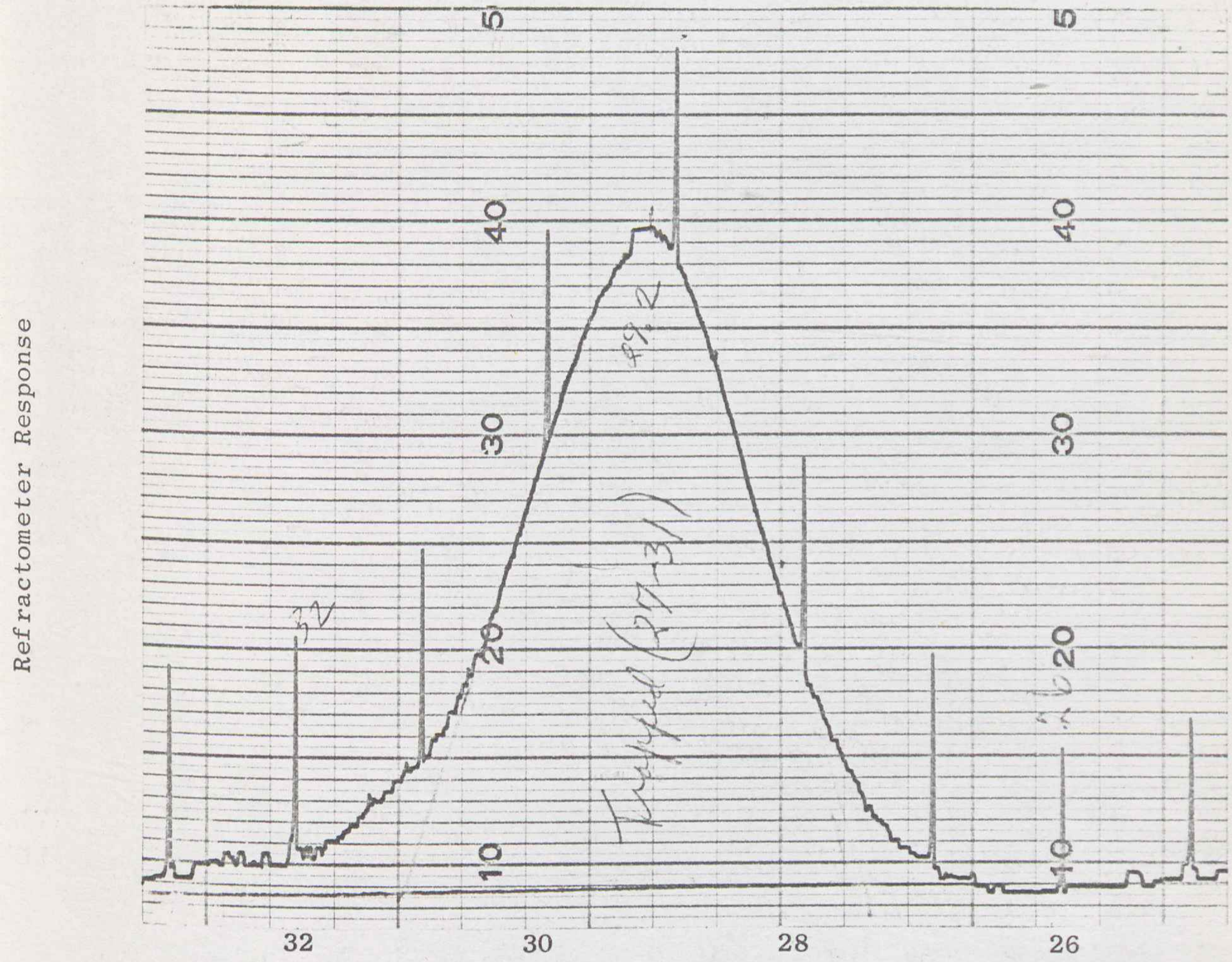
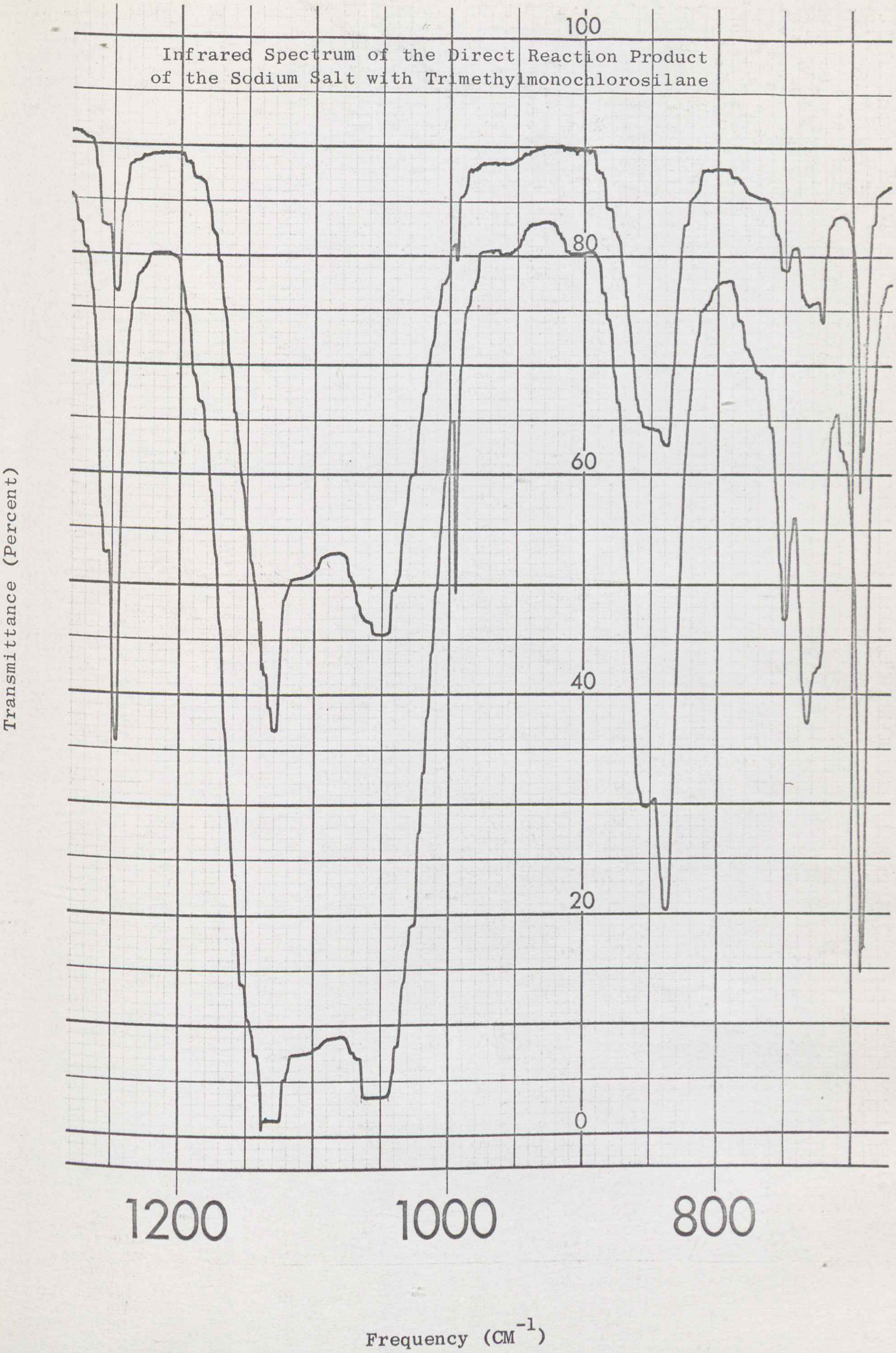
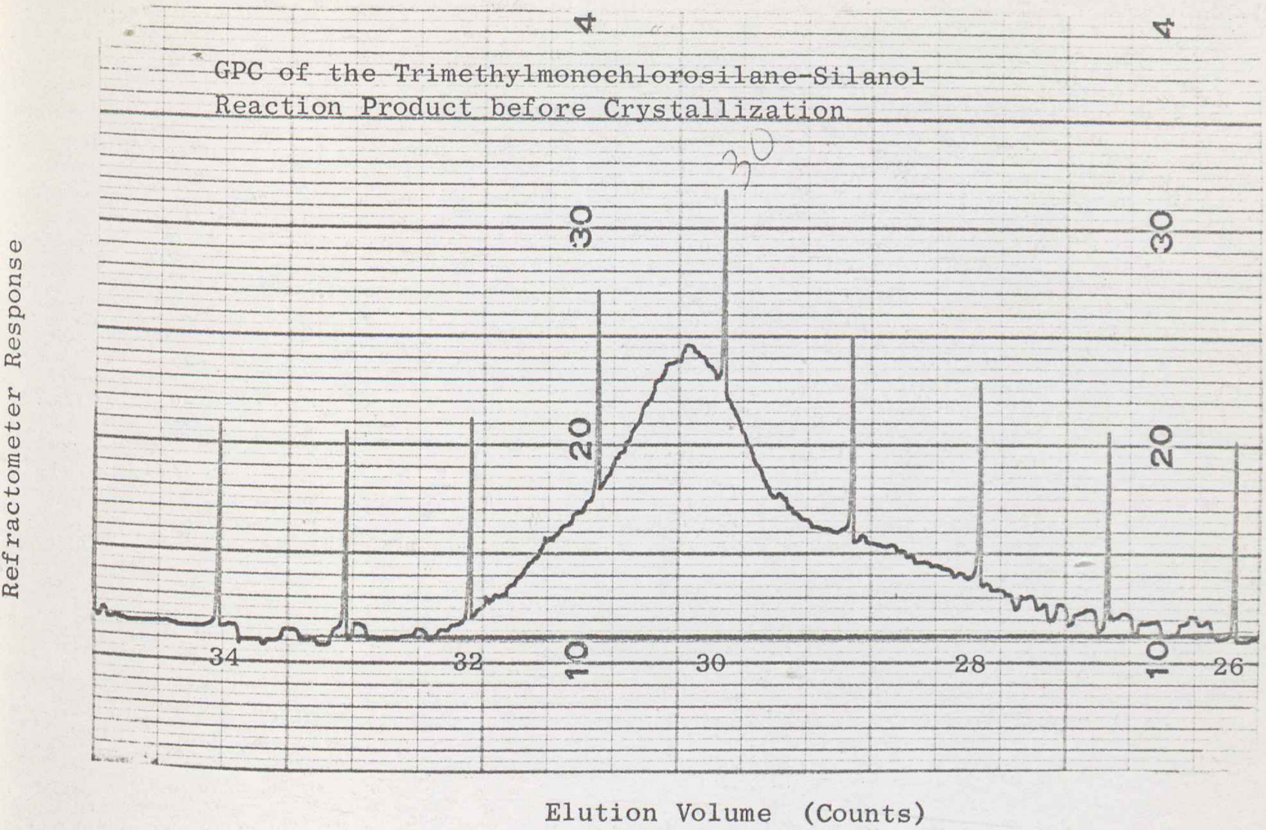


FIGURE V



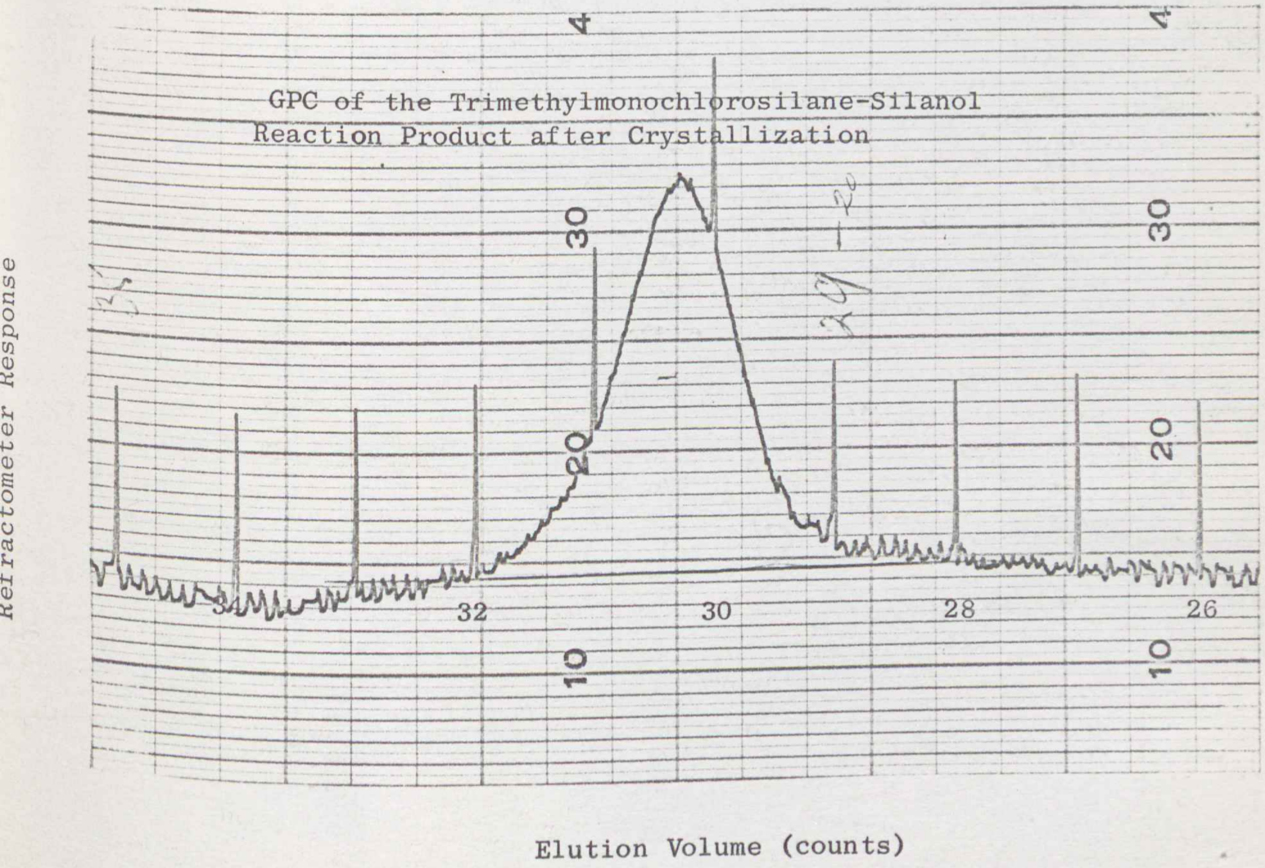
Infrared Spectrum of the Direct Reaction Product of the Sodium Salt with Trimethylmonochlorosilane

FIGURE VI



GPC of the Trimethylmonochlorosilane-Silanol Reaction Product before Crystallization

FIGURE VII



GPC of the Trimethylmonochlorosilane-Silanol Reaction Product after Crystallization

FIGURE VII

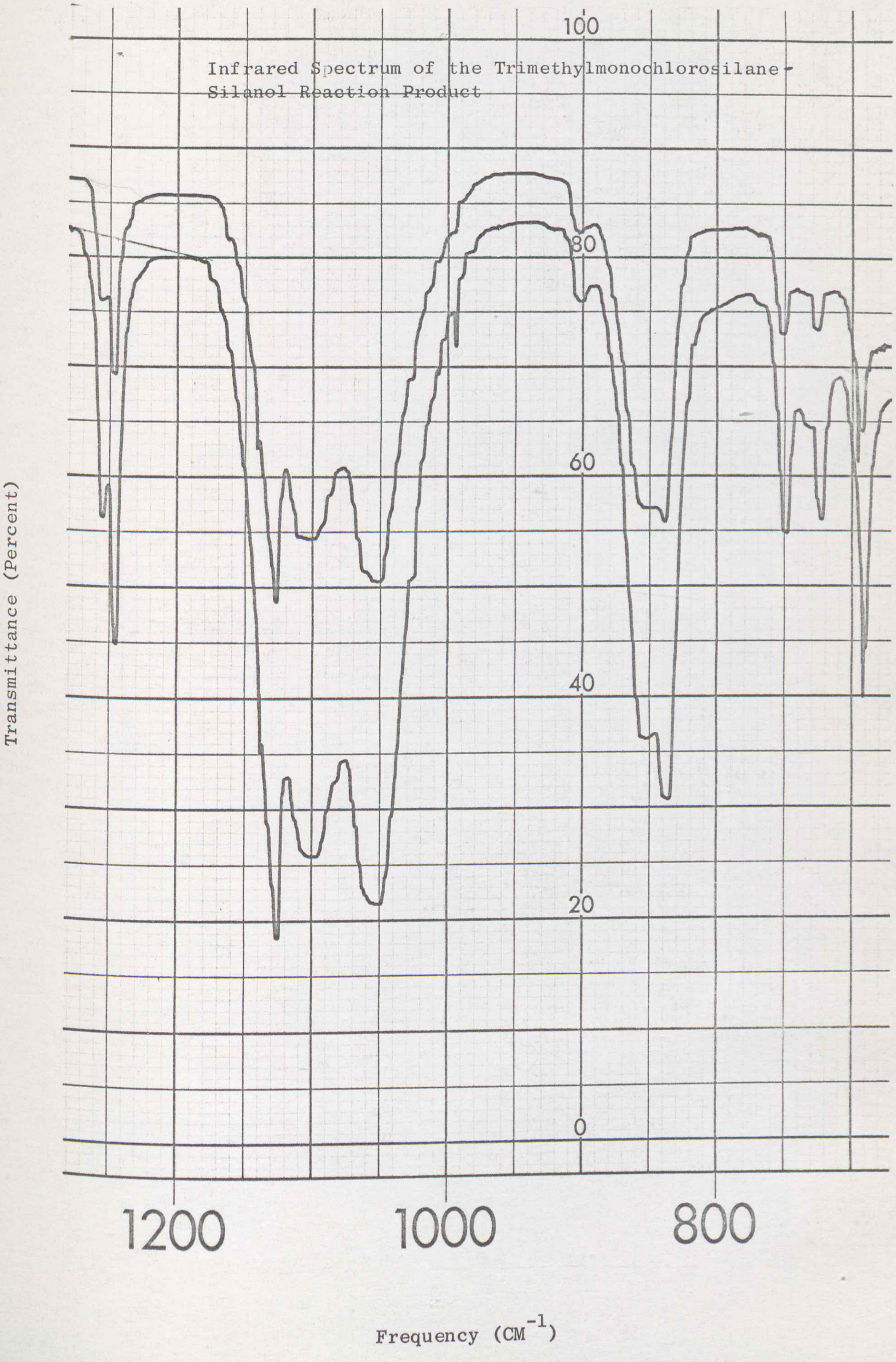
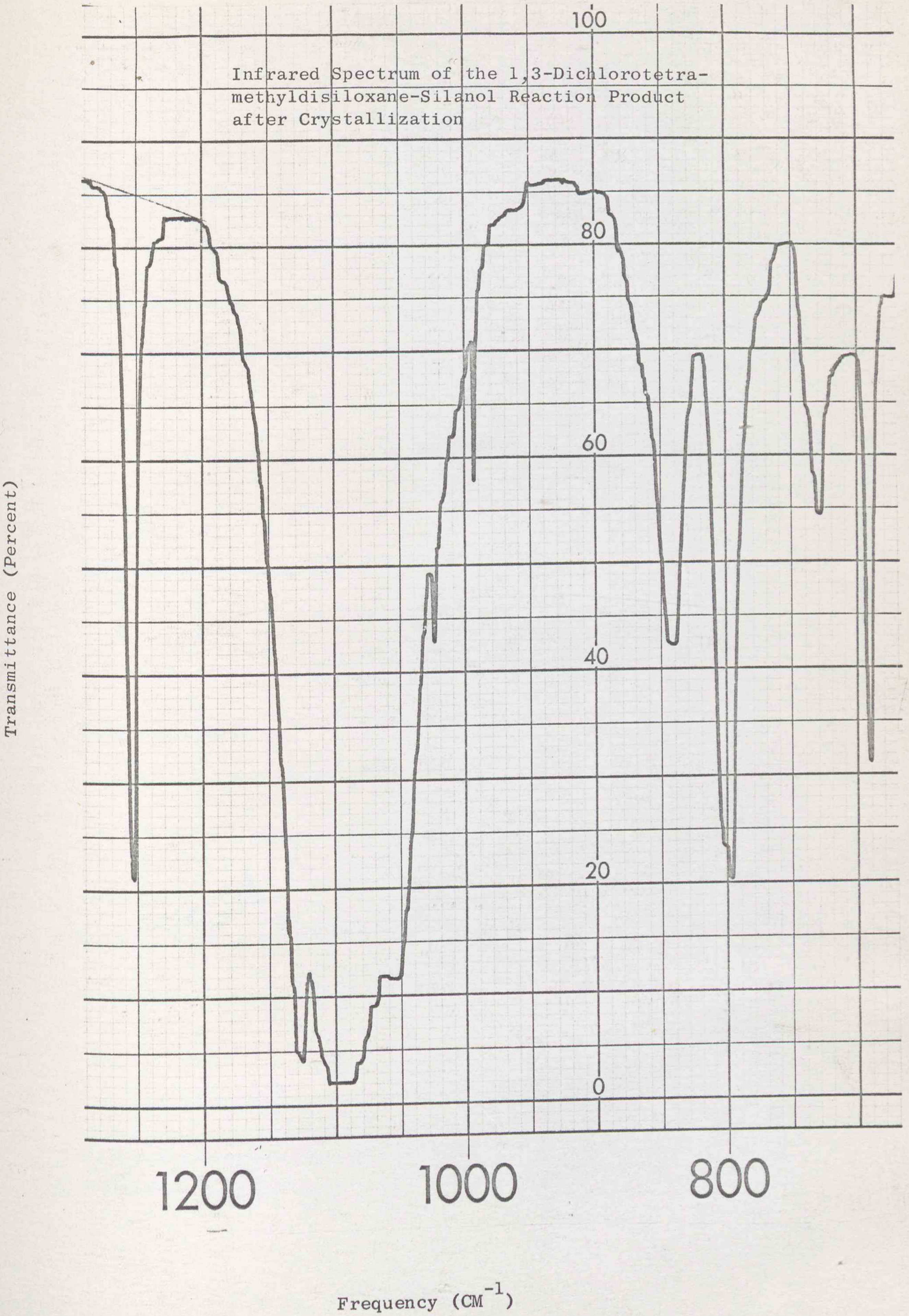

Infrared Spectrum of the Trimethylmonochlorosilane-Silanol Reaction Product
100
80
60
40
20
0
Transmittance (Percent)
1200
1000
800
Frequency (CM^{-1})

FIGURE IX



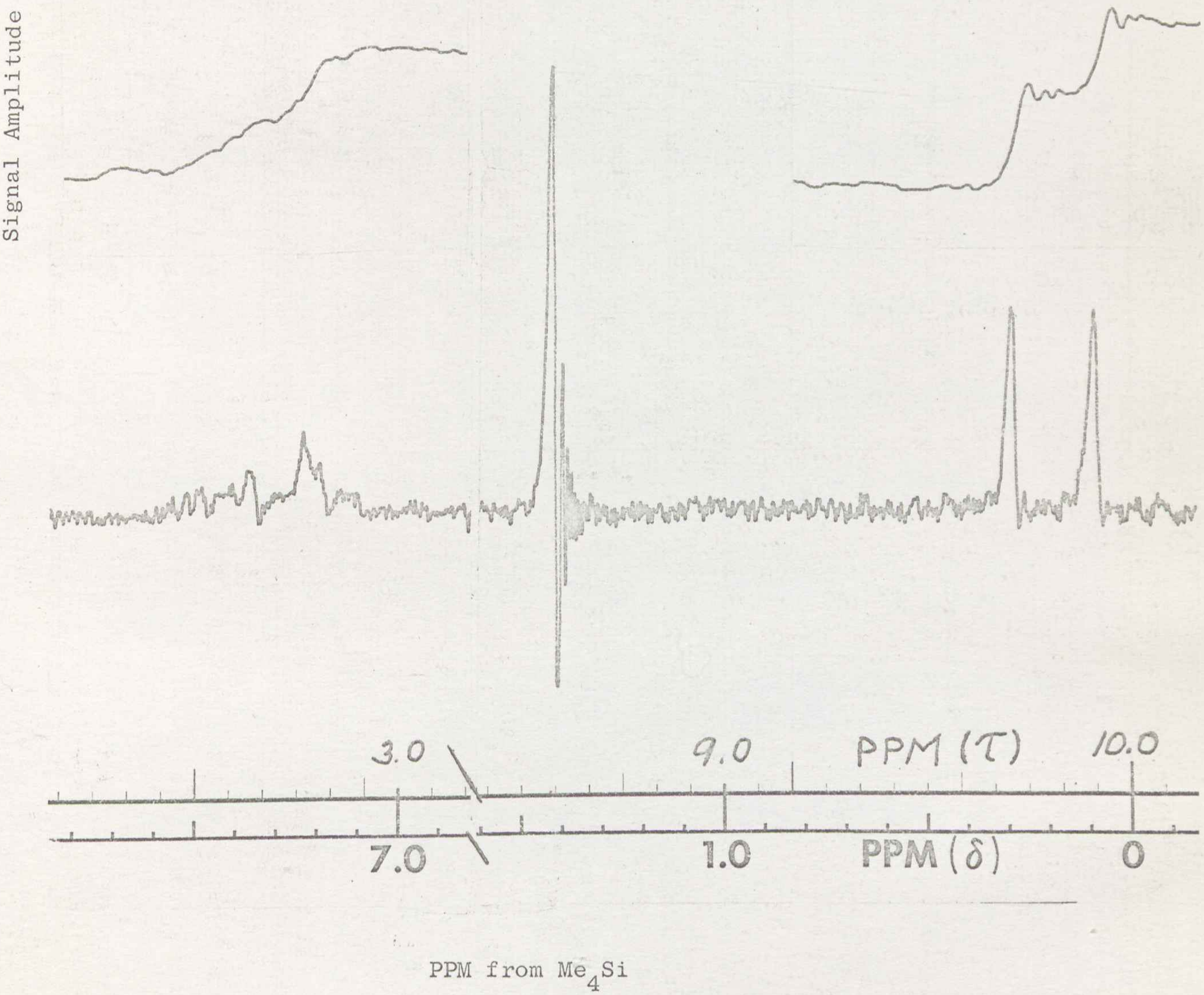
Infrared Spectrum of the 1,3-Dichlorotetramethyldisiloxane-Silanol Reaction Product after Crystallization

FIGURE X

Proton Magnetic Resonance Spectrum of the 1,3-Dichlorotetramethyldisiloxane Reaction Product

SOLVENT	CCl_4	
TEMPERATURE	RT	°C
FILTER BANDWIDTH	2	cps
R.F. FIELD	0.08	mG
SWEEP TIME	250	sec
SWEEP WIDTH	500	cps
SWEEP OFFSET	0	cps
SPECTRUM AMP.	3.2	
INTEGRAL AMP.		

REMARKS: C_6H_{12} Internal Std.
$\Delta\delta(C_6H_{12} - Me_4Si) = 85.8$ Hertz



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