analytically pure material, m.p. 287–288° dec. (see Table I).

*2-(α-Aminobenzyl)pyridine, dihydrochloride.*[9] Phenyl-2-pyridyl ketone, oxime, 120 g., (0.06 mol.) was dissolved in 800 ml. of glacial acetic acid containing 30 ml. of water. The solution was brought to reflux and 210 g. (3 mol.) of zinc dust was added portionwise at a rate sufficient to maintain reflux. The addition was complete in 1 hr. and heating was continued for an additional hour. The reaction mixture was then filtered and the filtrate was made strongly alkaline, causing the product to separate. It was taken up in ether, dried over sodium sulfate and the ether was removed *in vacuo*. The oily residue was distilled to yield 69 g., 62%, of a pale yellow liquid, b.p. 159–165° at 3 mm., $n_D^{20}$ 1.5961. On standing 1 day at room temperature it turned dark brown and emitted a strong odor of ammonia.

*Anal.* Calcd. for $C_{12}H_{12}N_2$: N, 15.22. Found: N, 14.38.

Because of its instability it was converted to a dihydrochloride salt, which was crystallized from ethanol ether, m.p. 242–244° dec.

*Anal.* Calcd. for $C_{12}H_{14}Cl_2N_2$: C, 56.10; H, 5.46; Cl, 27.55. Found: C, 56.46; H, 6.56; Cl, 27.44.

*1-(α-2-Pyridylbenzyl)-3-methyl-2-thiourea.* 2-(α-Aminobenzyl)pyridine, 5 g. (0.027 mol.); methyl ester of isothiocyanic acid, 2 g., (0.027 mol.), and 50 ml. of absolute ethanol were heated under reflux for 2 hr. Cooling and addition of ether caused 6 g., 86%, of product, m.p. 165–166° to precipitate. One recrystallization from methanol did not change the melting point.

*Anal.* Calcd. for $C_{14}H_{15}N_3S$: C, 65.37; H, 5.87; N, 16.30. Found: C, 65.43; H, 5.66; N, 16.24.

*1-(α-2-Pyridylbenzyl)-2,3-dimethyl-2-thiopseudourea, hydriodide.* 1-(α-2-Pyridylbenzyl)-3-methyl-2-thiourea was converted into its hydriodide by treatment with hydriodic acid in an acetone solution. The hydriodide, 4.1 g., (0.011 mol.), iodimethane, 2.1 g. (0.015 mol.), and 50 ml. of ethanol were heated under reflux for 3 hr. On cooling and addition of ether, 2.7 g., 61%, of product, m.p. 178–180° dec., precipitated. Two recrystallizations from ethanol raised the melting point to 182–183°.

*Anal.* Calcd. for $C_{15}H_{18}IN_3S$: N, 10.52; S, 8.02. Found: N, 10.30; S, 7.80.

*1-(2,2-Diphenyl-2-hydroxyethyl)-2-thiourea.* The fusion was carried out in the usual manner in refluxing xylene.[1] α-(Aminoethyl)benzhydrol, hydrochloride,[10] 7.5 g. (0.03 mol.) and the ammonium salt of thiocyanic acid, 2.4 g. (0.03 mol.), gave 3.2 g., 42% of product, m.p. 185–187° dec. One recrystallization from ethanol did not change the melting point.

*Anal.* Calcd. for $C_{15}H_{16}N_2OS$: C, 66.14; H, 5.92; N, 10.03; S, 11.77. Found: C, 65.72; H, 5.96; N, 10.63; S, 11.90.

*1-(2,2-Diphenyl-2-hydroxyethyl)-2-methyl-2-thiopseudourea, hydriodide.* The methylation was carried out in the usual manner with iodomethane.[1] 1-(2,2-Diphenyl-2-hydroxyethyl)-2-thiourea, 2.1 g. (0.008 mol.), gave 2.2 g., 69%, of the product, m.p. 150–151° dec. One recrystallization from isopropanol did not change the melting point.

*Anal.* Calcd. for $C_{16}H_{19}IN_2OS$: N, 6.77; S, 7.74; I, 30.63. Found: N, 6.68; S, 7.60; I, 30.97.

*Benzophenone, 3-methyl-3-thioisosemicarbazone, hydriodide.* The methylation procedure was identical to that used for the methylation of thioureas with iodomethane.[1] Benzophenone, thiosemicarbazone,[11] 2.6 g. (0.01 mol.), gave 3.7 g., 93%, of product, m.p. 192–194° dec. One recrystallization from isopropanol did not raise the melting point.

*Anal.* Calcd. for $C_{15}H_{16}IN_3S$: N, 10.58; S, 8.07; I, 31.94. Found: N, 10.59; S, 8.09; I, 31.96.

*Acknowledgments.* The authors wish to thank Dr. C. I. Chappel of our Laboratories for the pharmacological data, Mr. W. J. Turnbull for the analyses and Dr. Gilles Papineau-Couture and Mrs. J. Jachner for numerous infrared absorption spectra.

Montreal, Canada

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(9) 3-(α-Aminobenzyl)pyridine was prepared by La Forge in a similar manner, *J. Am. Chem. Soc.*, **50**, 2487 (1928).

(10) K. Thomas and F. Bettziecke, *Z. Physiol. Chem.*, **140**, 246 (1924).

(11) P. Chabrier and E. Cattelain, *Bull. soc. chim. France*, 48 (1950).

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[Contribution from the Silicones Division, Union Carbide Corporation]

# Preparation and Properties of β-Cyanoethyltrichlorosilane[1]

R. A. PIKE, J. E. McMAHON,[2] V. B. JEX,[2a] W. T. BLACK,[2b] AND D. L. BAILEY

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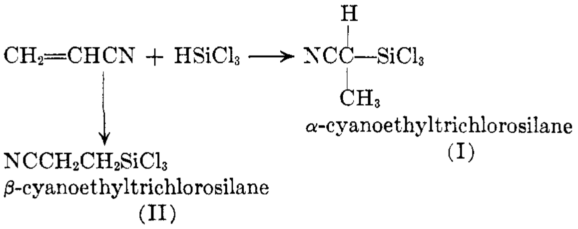
Directive catalysts, organic derivatives of Group V A elements, are described for the addition of trichlorosilane to acrylonitrile, which produce only β-cyanoethyltrichlorosilane. The effect of the cyano group on the rate of hydrolysis of the chlorosilane and heat stability of the corresponding silicone polymer are discussed and the utilization of β-cyanoethyltrichlorosilane as a starting material for the preparation of β-carbethoxyethyl silanes and β-carboxyethyl silicones is illustrated.

Trichlorosilane can add to acrylonitrile with formation of two possible isomeric adducts:



$$CH_2{=}CHCN + HSiCl_3 \longrightarrow NC\overset{H}{\underset{CH_3}{C}}{-}SiCl_3$$

α-cyanoethyltrichlorosilane (I)

$$\downarrow$$

$$NCCH_2CH_2SiCl_3$$

β-cyanoethyltrichlorosilane (II)

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(1) Presented at the 134th meeting of the American Chemical Society, Division of Organic Chemistry, at Chicago, Ill., Sept. 10, 1958.

(2) (a) Present address: Linde Co., Division of Union Carbide Corp., Tonawanda, N. Y. (b) Patent Department, Union Carbide Corp., 30 East 42nd St., New York, N. Y.

Under the conditions normally used for the addition of SiH compounds to olefins (*i.e.*, peroxide or platinum catalyzed), the reaction of trichlorosilane with acrylonitrile gives predominantly the α-isomer (I),[3,4] as well as polyacrylonitrile and other addition products. The α-isomer is hydrolytically unstable and of little synthetic value compared to the β-isomer (II).[5]

This paper reports the results of an investigation of new directive catalysts for the addition of trichlorosilane to acrylonitrile and some of the properties of the resulting β-cyanoethyltrichlorosilane (II).

It was discovered that tertiary phosphines, under conditions studied, appear to be the most effective catalysts for the addition with exclusive formation of the β-isomer (II). In general, organic compounds derived from Group V A elements were found to have some catalytic activity in the reaction. The relative effectiveness of representative catalysts is shown in Table I.

TABLE I

CATALYSTS FOR ADDITION OF $HSiCl_3$ TO ACRYLONITRILE[a]

| Catalyst | Product (Wt. % Conversion) | Remarks[6] |
|---|---|---|
| None | 42 | α- adduct |
| $(n\text{-}C_4H_9)_3P$ | 55 | β- adduct |
| $(C_6H_5)_2PCl$ | 47 | β- adduct |
| $C_6H_5PCl_2$ | 39 | β- adduct |
| $(C_6H_5)_3P$ | 56 | β-isomer, no α |
| $(C_6H_5)_3N$ | 29.8 | Mixture of α and β |
| $Et_3N$ | 30.7 | β- adduct |
| $(C_6H_5)_3As$ | 25.8 | Mainly α, some β |
| $(C_6H_5)_3Bi$ | 10.5 | Mixture of α and β |
| $(C_6H_5)_3Sb$ | Trace | Mixture of α and β |

[a] 1:1 mole ratio reactants; 200°; 2 hr.; 2 wt. % catalyst, in 50-ml. stainless steel pressure vessel.

In addition to tertiary phosphines, tertiary amines, as previously reported,[3] are effective directive catalysts; the aliphatic amines gave only β-isomer, while the aryl amines gave a mixture of isomers. The tertiary arsines, bismuthines, and stibines are less effective and also gave mixtures. Variations in time, temperature, and catalyst concentration, on the addition reaction using triphenyl phosphine catalyst (Table II) showed that catalyst concentration and time of reaction over a small range has little effect on yield of product, whereas temperature affects the yield to a marked degree. This effect is partially due to the increase in polymer formation at the higher temperatures. This can be controlled to some extent by the addition of a polymerization inhibitor such as hydroquinone to the system.

TABLE II

EFFECT OF TEMPERATURE, TIME, AND CATALYST CONCENTRATION ON $HSiCl_3:CH_2{=}CHCN$ REACTION USING $(C_6H_5)_3P$ CATALYST[a]

| (Wt. %) Catalyst Concentration | Temp. | Time (Hr.) | % Conversion Product |
|---|---|---|---|
| 2 | 200 | 2 | 56 |
| 2 | 150 | 2 | 65 |
| 1 | 150 | 2 | 67 |
| 0.5 | 150 | 2 | 59 |
| 1 | 100 | 0.5 | 71 |
| 1 | 100 | 2 | 70 |
| 1 | 75 | 2 | 13 |

[a] 1:1 mole ratio of reactants; 300 ml. stainless steel pressure vessel.

The other catalysts listed in Table I would undoubtedly exhibit the same temperature dependence as shown by triphenylphosphine, and several of them might be equally effective catalysts under optimum conditions.

To demonstrate the influence of the cyano group on the properties of chlorosilanes, measurements of the rate of hydrolysis of II were made and compared to the corresponding nonorganofunctional chlorosilanes. The relative rates of hydrolysis of the first chlorine as determined in dimethyl "Cellosolve"–water mixtures for several chlorosilanes[7] and β-cyanoethyl trichlorosilane, Table III, show the cyano group markedly increases the rate of hydrolysis, the relative increase being greater at higher temperatures.

TABLE III

RELATIVE RATES OF HYDROLYSIS OF FIRST CHLORINE IN ORGANOCHLOROSILANES IN DIMETHYL "CELLOSOLVE"[7]

| $RSiCl_3$ | At −51° | At −20° | At 0° |
|---|---|---|---|
| $C_2H_5SiCl_3$ | 1.0 | 1.0 | 1.0 |
| $CH_3SiCl_3$ | 2.2 | 0.59 | 0.30 |
| $C_6H_5SiCl_3$ | 4.1 | 2.0 | 1.4 |
| $NCCH_2CH_2SiCl_3$ | 62.0 | 116.0 | 153.0 |

This increase may be due to the effect of the cyano group on the electropositive character of the silicon atom. By inductive effect (−I effect) the Si atom would become more positive and thus subject, to

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(3) S. Nozakura and S. Konotsure, *Bull. Chem. Soc., Japan*, **29**, 326 (1956).

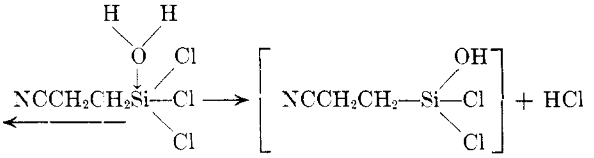
(4) L. Goodman, R. M. Silverstein, and A. Benitez, *J. Am. Chem. Soc.*, **79**, 3073 (1957).

(5) S. Nozakura and S. Konotsure, *Bull. Chem. Soc. Japan*, **29**, 322 (1956).

(6) Positive identification of α- and β- adducts of acrylonitrile and chlorosilane can be made on the basis of characteristic infrared absorption bands at 6.86μ (CH deformation) and 11.5μ in the spectrum of the α- adduct, and at 6.97μ (CH deformation) and 11.0μ in the spectrum of the β- adduct. Also the spectrum of the α- adducts shows a characteristic peak at 3.48μ which can be ascribed to the tertiary C—H stretching; this band does not appear in the β- adduct spectrum.

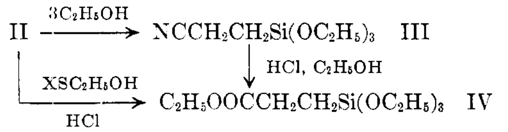
(7) L. H. Shaffer and E. M. Flanigen, *J. Phys. Chem.*, **61**, 1591, 1595 (1957).

a greater extent than in alkyl silanes, to nucleophilic attack by water.[8]



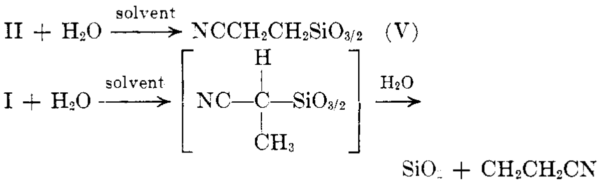
The β-cyanoethyl group attached to silicon undergoes the normal reactions of an aliphatic cyano compound. The silicon portion of the molecule may or may not enter into the reaction, depending upon the conditions employed. The synthesis of other silane monomers clearly demonstrates this versatility.

β-Cyanoethyltriethoxy silane[3] (III), a water-white, high boiling liquid, was obtained by addition of a stoichiometric amount of ethanol to the chlorosilane (II). The cyano group was converted to the ethyl ester by further reaction with ethanol, using an acid catalyst to give β-carbethoxyethyltriethoxy silane (IV). The ester (IV) can be prepared in one step by reaction of β-cyanoethyltrichlorosilane with excess alcohol, the hydrogen chloride liberated during the esterification being used as the acid catalyst.



$$\begin{tikzcd} \mathrm{II} \arrow[r, "3C_2H_5OH"] \arrow[dr, "XSC_2H_5OH", "HCl"'] & NCCH_2CH_2Si(OC_2H_5)_3\ \mathrm{III} \arrow[d, "HCl,\ C_2H_5OH"] \\ & C_2H_5OOCCH_2CH_2Si(OC_2H_5)_3\ \mathrm{IV} \end{tikzcd}$$

Since the importance of an organofunctional silane depends to a great extent upon the ease of preparation and stability of the silicone polymer which can be derived from it, the polymers corresponding to the silanes described above were prepared and their properties investigated. β-cyanoethyl silicone,[5] $NCCH_2CH_2SiO_3/_2$ (V) was prepared by hydrolysis of β-cyanothyltrichlorosilane (II), using an ether-water system,[9] showing that under usual hydrolysis conditions no silicon-carbon bond cleavage occurs. However, hydrolysis of α-cyanoethyltrichlorosilane (I) using a similar procedure gave silica and propoinitrile.[5] Nearly pure α-cyanoethyl silicone can be obtained, however, if the hydrolysis is carried out at 0°.



$$\mathrm{II} + H_2O \xrightarrow{\text{solvent}} NCCH_2CH_2SiO_{3/2}\ (\mathrm{V})$$

$$\mathrm{I} + H_2O \xrightarrow{\text{solvent}} \left[ NC\text{—}\overset{H}{\underset{CH_3}{C}}\text{—}SiO_{3/2} \right] \xrightarrow{H_2O} SiO_2 + CH_2CH_2CN$$

β-Cyanoethyl silicone (V) was found to have surprising thermal stability when heated in air at 250° for 100 hr. As shown in Table IV, the β-cyanoethyl group has nearly the same thermal stability, measured as percent retention of carbon, as methyl silicone, and markedly superior stability compared to ethyl or α-cyanoethyl silicones. The stability may be partially due to coordination of the cyano group with the silicon atom, either inter- or intramolecularly, involving expansion of the silicon valence shell.[10] No experimental data to support this postulate has been obtained to date.

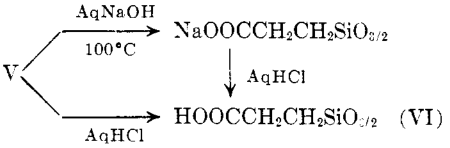
TABLE IV

COMPARISON OF SILICONE OXIDATIVE STABILITY IN AIR AT 250°

| Siloxane | % Original C. Remaining after 100 Hr. |
|---|---|
| Diphenyl | 98.4 |
| Methyl | 96.7 |
| β-cyanoethyl | 88.3 |
| Dimethyl | 81.5 |
| α-cyanoethyl | 12.6[a] |
| Ethyl | 6.7 |

[a] After 25 hr. at 250°.

As was the case with the chlorosilane monomers, the cyano group in the silicone polymer can also undergo reaction. This was illustrated by synthesis of the corresponding carboxyalkyl silicones. The carboxy acid was prepared from β-cyanoethyl silicone (V) using either alkaline or acidic conditions. Using alkali the salt of the acid was obtained, which was easily neutralized to form the acid, β-carboxyethyl silicone (VI).



$$\begin{tikzcd} & NaOOCCH_2CH_2SiO_{3/2} \arrow[d, "AqHCl"] \\ \mathrm{V} \arrow[ur, "AqNaOH", "100°C"'] \arrow[r, "AqHCl"'] & HOOCCH_2CH_2SiO_{3/2}\ (\mathrm{VI}) \end{tikzcd}$$

The acid (VI) is a white solid, soluble in hot water, sparingly soluble in cold water. The warm, concentrated solutions of the acid, when cooled, formed gel similar to that obtained with silicic acid in water. The compound is also soluble in methanol and dimethyl formamide, but insoluble in carbon tetrachloride, diethyl ether, and acetone. The acid can also be prepared from β-carbethoxyethyltriethoxy silane (IV) by saponification with aqueous base followed by neutralization of the resulting sodium carboxylate.

## EXPERIMENTAL

*β-Cyanoethyltrichlorosilane* (II). All runs were made with equimolar mixtures of acrylonitrile and trichlorosilane mixed with the specified catalyst, and heated in a 300-cc. stainless

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[8] A similar mechanism has been postulated for the effect of trifluoromethyl group attached to silicon through a $CH_2$ chain. O. W. Steward and O. R. Pierce, Abstract of Papers, Organic Division of the American Chemical Society, Chicago, Ill., Sept. 10, 1958, 50P.

[9] E. G. Rochow, *Chemistry of the Silicones*, J. Wiley and Sons, Inc., New York, N. Y., 2nd ed., p. 53.

[10] N. Sidgwick, *The Electronic Theory of Valency*, Oxford University Press, London, 1927, pp. 159 and 160.

steel pressure vessel placed in a rocking furnace for the specified temperature and time. At the end of the reaction the vessel was cooled, discharged, and the product filtered, using a fritted-glass filter. The filtrate was concentrated and distilled under reduced pressure through a glass-helix packed column to give β-cyanoethyltrichlorosilane (II), b.p. 57° (1 mm.), m.p. 36°. Hydrolyzable chlorine: theory, 56.4%; found, 56.0%. Literature value[3] b.p. 109° (30 mm.), m.p. 34.6–35.1°.

The α-isomer (I) was similarly prepared using no catalyst and had the following properties: b.p. 37° (1.0 mm.), $n_D^{25}$ 1.4513. Hydrolyzable chlorine: theory 56.4%; found 55.5%. Literature value,[5] b.p. 96–8° (41.5 mm.).

*β-Cyanoethyltriethoxysilane* (III). In a 500-ml., three necked flask fitted with a reflux condenser, dropping funnel, gas inlet tube, and stirrer, was charged 36.35 g. (0.19 mol.) of β-cyanoethyltrichlorosilane and 75 ml. of diethyl ether. Ethanol (26.7 g., 0.58 mol.) was added slowly with stirring over a 0.5-hr. period. The stirring was continued for 3 hr. after the addition was complete with the system under a slow argon purge. The last traces of hydrogen chloride were neutralized by bubbling gaseous ammonia through the reaction mixture. The mixture was then filtered under reduced pressure through a Büchner funnel and the filtrate fractionally distilled through a 26-cm. column packed with 10 × 12 mesh alumina. There was obtained 24.21 g. (58%) $n_D^{25}$ 1.4153, $D_4^{25}$, 0.970, b.p. 102° (3.8 mm.) of β-cyanoethyltriethoxysilane.

*Anal.* Calcd. for $C_9H_{19}NO_3Si$: C, 49.74; H, 8.81; $OC_2H_5$, 62.21. Found: C, 49.5; H, 8.7; $OC_2H_5$, 62.4.

Previous work[3] reports b.p. 111.5–112.5° (10 mm.), $D_4^{24}$ 0.9699, $n_D^{25}$, 1.4103.

*β-Carbethoxyethyltriethoxysilane* (IV). A 500-ml., three necked flask, fitted with a dropping funnel, thermometer, magnetic stirrer, and a reflux condenser, was charged with 168.6 g. (0.89 mol.) of β-cyanoethyltrichlorosilane. Over the course of 2 hr., with stirring, was added 164.2 g. (3.58 mol.) of ethanol. Hydrogen chloride was evolved and nitrogen was used to purge the residual hydrogen chloride from the reaction mixture. Additional ethanol (115 ml., 197 mol.) was then added, and the solution heated at reflux for 16 hr. The solution was then concentrated under reduced pressure and the residue taken up in diethyl ether and filtered to separate the ammonium chloride. The filtrate was concentrated and the residue distilled under reduced pressure through a glass-helix packed column to give 150 g. (63.5%), b.p. 124–125° (10 mm.), $n_D^{25}$ 1.4125, of β-carbethoxyethyltriethoxysilane.

*Anal.* Calcd. for $C_{11}H_{24}O_5Si$: C, 50.0; H, 9.15; Si, 10.6. Found: C, 50.3; H, 9.8; Si, 10.4.

*β-Cyanoethylsilicone* (V). In a 500-cc. beaker was charged 400 cc. of cracked ice and 100 ml. of diethyl ether. β-cyanoethyltrichlorosilane (15.46 g.) dissolved in 100 ml. of diethyl ether was added with stirring to the ice-solvent mixture. The hydrolysis was extremely rapid. The ether was allowed to evaporate from the aqueous acid layer, and the resulting oil layer separated and washed with distilled water until the water washings were neutral. The viscous oil was further concentrated under reduced pressure and the residue dried at 80° for 2 hr. to give 6.38 g. (99.7%) of a white, amorphous powder.

*Anal.* Calcd. for $C_3H_4NO_{1.5}Si$: Si, 26.44; N, 13.2. Found: Si, 25.8; N, 12.4.

*β-Carboxyethylsilicone* (VI) *from β-cyanoethyltrichlorosilane* (II). In a 2000-ml. beaker was charged 255 g. of β-cyanethyltrichlorosilane dissolved in 800 ml. of isopropyl ether. To the ether solution was added, with constant stirring, 600 g. of crushed ice. Hydrogen chloride was evolved and a white precipitate formed, which was crude $NCCH_2CH_2SiO_{3/2}$. Sodium hydroxide solution (32.5%, 400 ml.) was then added and the mixture stirred until the solid dissolved in the aqueous layer. The aqueous layer was separated by means of a separatory funnel and neutralized by addition of concentrated hydrochloride acid at 10–20°. A heavy white precipitate formed which was filtered on a Büchner filter, washed with four 250-ml. portions of distilled water and dried under vacuum to give 147.3 g. (86.5%) of β-carboxyethylsilicone. Equivalent weight by titration of an aqueous solution with base: Theory: 125; Found: 127.4.

*Anal.* Calcd. for $C_3H_5SiO_7$: C, 28.8; Si, 22.4. Found: C, 28.3; Si, 21.3.

*β-Carboxyethylsilicone* (VI) *from β-carbethoxyethyltriethoxysilane* (IV). β-carbethoxyethyltriethoxysilane (88.9 g.) and 200 ml. of water were mixed in a 1-l., round bottomed flask, equipped with a reflux condenser and thermometer. The mixture was refluxed for 1 hr., but no noticeable reaction occurred. A small amount (30 ml.) of 3*N* hydrochloric acid was added, and the refluxing continued for 2 hr. The solution became cloudy during this period. The solution was then concentrated under reduced pressure to give a solid residue. The solid was ground to a white powder and dried at 100° for 6 hr. in a vacuum oven to remove traces of water. The yield of β-carboxyethylsilicone was 39.6 g. (93.8%).

*Anal.* Calcd. for $C_3H_5SiO_{7/2}$: C, 28.8; Si, 22.4. Found: C, 28.3; Si, 21.4. Equivalent weight by titration, 127.4; theory, 125.

*α-Cyanoethylsilicone* (I). By the procedure described above for β-cyanoethylsilicone, 19.6 g. of α-cyanoethyltrichlorosilane b.p. 55° (4 mm.) was hydrolyzed to give an amorphous solid. The chemical analysis showed that some cleavage of the carbon-silicon bond had occurred during hydrolysis.

*Anal.* Calcd. for $C_3H_4NO_{1.5}Si$: C, 33.95; H, 3.80; N, 13.2; Si, 26.44. Found: C, 28.8; H, 3.6; N, 8.6; Si, 27.8. After heating the sample in air at 250° for 25 hr. the analysis was: C, 3.0; H, 1.1; N, 1.3; Si, 40.8. Theory, Si for $SiO_2$, 46.6%.

*Heat stability tests.* These tests were run by heating a weighed sample of the material in a 150-ml. beaker in a forced draft oven at 250°. Samples were analyzed after every 24-hr. heating cycle. The values listed in Table IV are final results obtained on carbon analysis after 100 hr.

*Acknowledgment.* The authors wish to thank Dr. E. R. Shull and Mr. D. M. Ruttenbur of the Molecular Spectroscopy Group, Linde Company, Division of Union Carbide Corp. Tonawanda, N. Y., for the interpretation of the infrared data.

TONAWANDA, N. Y.