[CONTRIBUTION FROM THE GENERAL ELECTRIC RESEARCH LABORATORY]

TRIMETHYLSILYL DERIVATIVES OF POLYOLS

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Simple alkoxy derivatives of the alkyl and aryl silanes have proven very useful as intermediates for the preparation of more complex organo-silicon compounds, especially organopolysiloxanes. They are particularly useful where it is necessary to avoid the vigorous exothermic reactions normally encountered with organohalogenosilanes. For example, dialkylsilanediols are difficult to obtain from the corresponding dialkyldichlorosilanes, but can more easily be prepared from the dimethoxy or diethoxy derivatives (1).

Silyl derivatives of polyols are as yet little known. Kipping and Abrams (2) obtained bis-(tetraphenylethylenedioxy)silane in low yield when benzophenone and silicon tetrachloride were treated with magnesium in ether. Krieble and Burkhard (3) studied the reactions of dimethyldichlorosilane and dimethyldiethoxysilane with a number of glycols. Polymeric products were usually found. Heating these products under a vacuum gave cyclic, monomeric dimethylalkylenedioxysilanes; except in the case of ethylene glycol, which gave the 10-membered-ring dimer instead.

Staudinger and Hahn (4) investigated the reaction of glycols with diphenoxydichlorosilane and dicyclohexoxydichlorosilane in the presence of a tertiary base (pyridine). In addition to a number of low-polymeric products (DP = 4–9), they obtained several monomeric dialkoxy(polymethylenedioxy)silanes. From dicyclohexoxydichlorosilane and pinacol they obtained a very good yield of the monomeric product which has a 5-membered ring. Ethylene glycol, however, again gave the 10-membered-ring dimer rather than the 5-membered-ring monomer. They also prepared 1,6-bis-(triphenoxysiloxy)hexane from triphenoxychlorosilane and 1,6-hexanediol and the bis-(tricyclohexoxysilyl) derivatives of ethylene glycol, 1,6-hexanediol, 2-butyne-1,4-diol and 1,8-*p*-menthanediol.

Using similar methods, Hahn (5) prepared spirocyclic alkoxysilanes from silicon tetrachloride and glycols. Pinacol as well as 1,1′-dihydroxy-1,1′-bicyclohexyl gave monomers having two spiroid 5-membered rings. 1,3-Butanediol, 1,4-butanediol, and diethylene glycol similarly gave spiroid monomers having 6, 7, and 8 ring members.

Trialkylsilyl derivatives of the polyols should exist only as monomers. Despite this, only a few examples are known. Martin (6) separated the 4-mono-, 2,4-di-, and 2,4,6-trimethylol derivatives of phenol by fractional distillation of the trimethylsilyl derivatives obtained by treating the mixture with trimethylchlorosilane in pyridine and subsequent hydrolysis to the pure phenol. Breederveld, Steuns, and Waterman (7) prepared the tris-(trialkylsilyl) derivatives of glycerol, $R_3SiOCH_2CH(OSiR_3)CH_2OSiR_3$, where R was methyl, ethyl, *n*-propyl, and *n*-butyl. They are all liquids, easily distillable in a vacuum.

The present work involved the preparation of trimethylsilyl derivatives both of diols and polyols. There were two objectives in mind. First, it seemed desirable to study the simpler monofunctional silyl derivatives as a prelude to further work on the more complex polyfunctional products. Second, it was of interest to investigate further the feasibility of using trimethylsilyl derivatives to effect otherwise difficult purifications of polyols. Sugar alcohols were of particular interest in this respect. Trimethylchlorosilane has proved a very useful reagent for the separation of the hexitols from mixtures containing lower polyols. Substances such as galactitol are not very heat-stable and must be made into derivatives before they can be distilled from a mixture. Trimethylsilyl derivatives are easily prepared, are stable toward heat and oxygen, have considerably lower boiling points than the corresponding polyols, and are readily quantitatively hydrolyzed to yield the original material (6).

Trimethylsilyl derivatives of many glycols and polyols are conveniently prepared by addition of trimethylchlorosilane to an equivalent quantity of the alcohol containing a slight excess of a tertiary amine, such as pyridine, N,N-dimethylaniline, or tri-*n*-butylamine. Pyridine is, in general, the most convenient acid acceptor. The reaction generally proceeds favorably at room temperature, or shomewhat below. Pyridine hydrochloride, formed as a by-product, usually can be filtered; and the reaction products, normally liquid, are purified by vacuum fractionation.

2-Trimethylsiloxyethanol was easily prepared from ethylene glycol and trimethylchlorosilane if an excess of the glycol was used. It was easily separated by distillation from the disubstitution product, obtained as a by-product. However, with trimethylene glycol, tetramethylene glycol, and hexamethylene glycol the disubstitution products were formed predominantly even when a large excess of the glycol was used. In these three cases, the trimethylsiloxyalkanols formed azeotropes with the bis-(trimethylsiloxy)alkanes. 3-Trimethylsiloxypropanol was obtained in low yield as the higher-boiling component after distillation of the azeotrope. The homologous trimethylsiloxyalkanols were not obtained in a pure state. The bis-(trimethylsiloxy)alkanes, on the contrary, can be prepared easily and in good yield simply by employing essentially stoichiometric proportions of the reactants. In addition to those mentioned above, the bis-(trimethylsiloxy) derivatives of propylene glycol and neopentyl glycol thus were obtained.

Tris-(trimethylsiloxy)alkanes were prepared in excellent yield from glycerol, 1,1,1-trimethylolethane, and 1,1,1-trimethylolpropane; and a trimethylsilyl derivative of tris-(β-hydroxyethyl) cyanurate was similarly prepared. Other polyols that were converted to polytrimethylsilyl derivatives were erythritol, pentaerythritol, sorbitol, mannitol, and galactitol.

Trimethylsilyl derivatives of several bis-(β-hydroxyethyl) dicarboxylates were also prepared; namely, those derived from bis-(β-hydroxyethyl) oxalate, maleate, phthalate and terephthalate. Since these reactions were conducted by adding trimethylchlorosilane to crude reaction products containing the bis-(β-hydroxyethyl) dicarboxylate as the principal component [bis-(β-hydroxyethyl) dicarboxylates are very difficult to isolate in the pure state] the yields were not as good as in other examples described.

EXPERIMENTAL

Materials. The trimethylchlorosilane was obtained from the Silicone Products Department of the General Electric Company and from the Dow Corning Corporation. Reagent grade pyridine and Distillation Products "white label" grade tri-*n*-butylamine were used.

Ethylene glycol and glycerol were reagent grade; the other polyols were commercial chemicals, except galactitol which was prepared by the hydrogenation of galactose with Raney nickel and purified by recrystallization from water. Tris-(β-hydroxyethyl) cyanurate was very kindly furnished by Dr. M. Cohen of the Thomson Laboratory of the General Electric Company. The preparation of the bis-(β-hydroxyethyl) dicarboxylates is described later.

2-Trimethylsiloxyethanol. Four moles (248 g.) of ethylene glycol were dissolved in 185 g. (1.0 mole) of tri-*n*-butylamine. The flask was surrounded by an ice-bath. To the rapidly stirred solution was added, from a dropping-funnel, 98 g. (0.91 mole) of trimethylchlorosilane during 40 minutes at 20–35°. The reaction mixture then was allowed to warm to room temperature, and finally was heated to reflux for 10 minutes. The product was distilled directly out of this mixture, until the boiling point reached 160°. Some tri-*n*-butylamine that was carried over appeared as a lower layer, and was discarded. The crude material was fractionated carefully to give 92 g. (75.5%) b.p. 151–152°, n_D^{20} 1.4117, d_4^{20} 0.902.

This compound was also prepared from trimethylethoxysilane and excess ethylene glycol in the presence of Amberlite IR-100H ion exchange resin (Rohm and Haas Company), but the yield was low (33%) because the ethoxysilane was partly removed from the reaction mixture as an azeotrope with the ethanol formed.

1,2-Bis-(trimethylsiloxy)ethane. One mole (62 g.) of ethylene glycol and 2.2 moles (407 g.) of tri-*n*-butylamine were mixed, cooled in an ice-bath, and stirred rapidly. Two moles (217 g.) of trimethylchlorosilane were added during 40 minutes at 10–55°. The reflux condenser was replaced by a Vigreux column and the product was distilled directly from the reaction mixture. The crude product (218 g., b.p. 150–183°) contained some tri-*n*-butylamine and a little tri-*n*-butylamine hydrochloride. It was filtered and carefully redistilled, giving 9 g. of 2-trimethylsiloxyethanol and 134.5 g. (65%) of 1,2-bis-(trimethylsiloxy)ethane, b.p. 165–166°, n_D^{20} 1.4034, d_4^{20} 0.846.

A better yield (*ca.* 90%) was obtained when the reaction was run again, with reagent grade chemicals and pyridine as the acid acceptor.

3-Trimethylsiloxy-1-propanol—1,3-bis-(trimethylsiloxy)propane azeotrope. Four moles (304 g.) of trimethylene glycol, dissolved in 174 g. (2.2 moles) of pyridine, were treated dropwise with 217 g. (2.0 moles) of trimethylchlorosilane at 11–17°. After heating at 140–145° for 10 minutes, the mixture was chilled. Pyridine hydrochloride did not precipitate. The mixture therefore was extracted with ether (200 ml., then three 100-ml. portions). The combined ether extracts, when dried, concentrated and distilled, gave 26 g. of a mixture of trimethylsilanol and hexamethyldisiloxane, and 179 g. of crude reaction product, b.p. 145–188°. Acetylation of successive fractions with acetic anhydride in pyridine indicated that a mixture of the mono- and di-substitution products was present. Redistillation in a 20-plate still gave 134.5 g. of an azeotrope, b.p. 78°/22 mm., n_D^{20} 1.4100–1.4104.

Anal. Calc'd for 33% $Me_3SiO(CH_2)_3OH$ + 67% $Me_3SiO(CH_2)_3OSiMe_3$: Si, 23.4; OH, 3.8.
Found: Si, 23.8; OH, 3.8 (by Grignard analysis).

The reaction was repeated, using 14 moles of trimethylene glycol to 3.5 moles of trimethylchlorosilane and 3.85 moles of pyridine. The yield was 290 g., b.p. 171–174°. On redistillation, the major fraction (249 g.) was again the 1:2 azeotrope (b.p. 78°/23 mm., n_D^{20} 1.4100–1.4104). A minor fraction (16.5 g., b.p. 81.5–82.0°/23 mm., n_D^{20} 1.4178, d_4^{20} 0.899) was apparently the monotrimethylsiloxypropanol.

1,3-Bis-(trimethylsiloxy)propane. This compound was prepared by treating trimethylene glycol with 10% more than the stoichiometric proportion of trimethylchlorosilane in an excess of pyridine. The yield was about 90% (Table I).

1,2-Bis-(trimethylsiloxy)propane. This substance was prepared similarly in about 90% yield from propylene glycol, trimethylchlorosilane and excess pyridine (Table I).

TABLE I

TRIMETHYLSILYL DERIVATIVES OF GLYCOLS

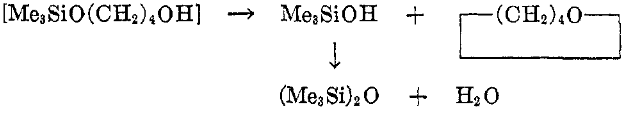
Compound	Formula	b.p., °C.	Press., mm.	n_D^{20}	d_4^{20}	MRD		Analyses							
								Calculated				Found			
						Calc'd	Found	C	H	Si	Mol Wt	C	H	Si	Mol Wt[a]
2-Trimethylsiloxyethanol	$C_5H_{14}O_2Si$	151–152	atm.	1.4117	0.902	37.2	37.1	44.8	10.5	20.9	—	45.5	11.0	20.4	—[b]
1,2-Bis-(trimethylsiloxy)-ethane[c,e]	$C_8H_{22}O_2Si_2$	165–166	atm.	1.4031	.841	59.1	59.9	46.5	10.7	27.2	206.4	46.7	11.1	27.4	—
		89	50	1.4034	.846		59.6					46.4	10.7	26.7	188
3-Trimethylsiloxy-1-propanol	$C_6H_{16}O_2Si$	81.5–82.0	23	1.4178	.899	41.8	41.5	—	—	18.9	148.3	—	—	18.5	152[d]
1,3-Bis-(trimethylsiloxy)propane[e]	$C_9H_{24}O_2Si_2$	69	11	1.4066	.843	64.7	64.3	49.0	11.0	25.5	—	49.4	10.7	25.1	—
1,2-Bis-(trimethylsiloxy)propane[e]	$C_9H_{24}O_2Si_2$	97	58	1.4031	.834	64.7	64.5	49.0	11.0	25.5	—	49.0	11.5	25.4	—
1,4-Bis-(trimethylsiloxy)butane	$C_{10}H_{26}O_2Si_2$	93.5–94.0	17	1.4114	.845	69.2	69.0	51.3	11.2	24.0	234.5	51.9	11.6	24.1	210
1,3-Bis-(trimethylsiloxy)-2,2-dimethylpropane[e]	$C_{11}H_{28}O_2Si_2$	73	11	1.4073	.829	74.0	73.8	53.2	11.4	22.6	—	53.1	11.5	22.6	—
1,6-Bis(trimethylsiloxy)hexane	$C_{12}H_{30}O_2Si_2$	126–128	20–22	1.4185	.845	78.6	78.4	54.9	11.5	—	262.5	55.3	12.0	—	232

[a] Determined cryoscopically in benzene. [b] Hydroxyl equivalent: Calc'd, 134.2; Found, 137.5. [c] The two sets of data describe products prepared by different workers. [d] Hydroxyl equivalent: Calc'd, 148.3; Found, 147.5. [e] The infrared spectra of these compounds were recorded, using a Perkin-Elmer Infrared Spectrophotometer, Model 21. These spectra are more useful in identification of unknowns than are those of the parent polyols.

4-Trimethylsiloxy-1-butanol—1,4-bis-(trimethylsiloxy)butane azeotrope. The reaction was carried out using 360 g. (4.0 moles) of tetramethylene glycol, 217 g. (2.0 moles) of trimethylchlorosilane, and 174 g. (2.2 moles) of pyridine. After cooling the reaction mixture, two liquid layers were formed. The lower layer was extracted 3 times with 200-ml. portions of ether. The ether solution was dried and distilled, giving 172 g., b.p. 88–96°/14–18 mm. Redistillation in an efficient still gave 152 g., b.p. 93–94°/17 mm., n_D^{20} 1.4160–1.4162. This was again a 1:2 azeotrope.

Anal. Calc'd for 33% $Me_3SiO(CH_2)_4OH$ + 67% $Me_3SiO(CH_2)_4OSiMe_3$: Si, 21.8; OH, 3.5. Found: Si, 22.1; OH, 3.5.

The preparation was repeated with tri-*n*-butylamine as the acid acceptor, and the reaction mixture was distilled directly. The only products isolated were trimethylsilanol, hexamethyldisiloxane, and tetrahydrofuran. These could be formed by disproportionation, as follows:



$$[Me_3SiO(CH_2)_4OH] \rightarrow Me_3SiOH + \boxed{(CH_2)_4O}$$
$$Me_3SiOH \downarrow (Me_3Si)_2O + H_2O$$

The 1:2 azeotrope was subjected to base-catalyzed ester interchange with methyl benzoate and the unaffected bis-(trimethylsiloxy)butane subsequently was recovered by distillation (b.p. 88.0–88.5°/17 mm., n_D^{20} 1.4240.

1,4-Bis-(trimethylsiloxy)butane then was prepared from tetramethylene glycol (2 moles) and trimethylchlorosilane (4 moles) in the presence of pyridine (4.4 moles). Ether (500 ml.) was added to aid filtration of the pyridine hydrochloride. Distillation gave 353 g. (75.5% yield) of 1,4-bis-(trimethylsiloxy)butane, b.p. 91–94° at 16–17 mm., n_D^{20} 1.4113–1.4133. A mid-cut of 144.5 g. boiled at 93.5–94.0° at 17 mm.

6-Trimethylsiloxy-1-hexanol—1,6-bis-(trimethylsiloxy)hexane azeotrope. The previous procedure was followed using 4 moles of hexamethylene glycol, 2 moles of trimethylchlorosilane and 2.2 moles of pyridine. Some pyridine hydrochloride (68 g.) separated and was filtered before ether extraction. The yield was 208.5 g., b.p. 106–115°/11 mm. Redistillation gave 152.5 g. of a 2:3 azeotrope, b.p. 103–105°/6.0–7.5 mm., n_D^{20} 1.4234–1.4237.

Anal. Calc'd for 40% $Me_3SiO(CH_2)_6OH$ + 60% $Me_3SiO(CH_2)_6OSiMe_3$: OH, 3.3. Found: OH, 3.3.

The azeotrope again was subjected to base-catalyzed ester interchange, and the unaffected disubstitution product was recovered by distillation, b.p. 126–128°/20–22 mm., n_D^{20} 1.4185, d_4^{20} 0.845.

1,3-Bis-(trimethylsiloxy)-2,2-dimethylpropane. Neopentyl glycol (2,2-dimethyl-1,3-propanediol) and trimethylchlorosilane, similarly, gave the corresponding bis-(trimethylsiloxy) derivative in approximately 90% yield (Table I).

POLYTRIMETHYLSILOXY DERIVATIVES OF POLYOLS

General procedure. A 1-liter, 3-neck flask with a coarse sintered glass disc sealed in the bottom was equipped with a sealed stirrer, dropping-funnel, and condenser fitted with drying tubes. Dry nitrogen was introduced through the sintered disc and the flask was charged with about 500 ml. of pyridine. Enough polyol to react with about 2 moles of trimethylchlorosilane was dissolved or slurried in the pyridine. About a 10% excess of trimethylchlorosilane then was added dropwise and with stirring. Stirring was continued until the reaction mixture had cooled to room temperature. The nitrogen inlet was replaced by a side-arm vacuum connection and a 1-liter flask. The pyridine solution of the product was filtered into the lower flask. The pyridine hydrochloride cake was washed twice with 100-ml. portions of 65–110° petroleum ether. The combined filtrate and washings were stripped of trimethylchlorosilane, petroleum ether, hexamethyldisiloxane, and pyridine. The trimethylsilyl derivative was purified by vacuum fractionation. Yields were approximately 90% based on the polyol. (Table II).

Bis-β-(trimethylsiloxyethyl) dicarboxylates. The bis-(β-hydroxyethyl) dicarboxylates

TABLE II

TRIMETHYLSILYL DERIVATIVES OF POLYOLS

Polyol	Formula of Derivative	b.p., °C.	Press., mm.	n_D^{20}	d_4^{20}	MRD		Analyses							
								Calculated				Found			
						Calc'd	Found	C	H	Si	Mol Wt	C	H	Si	Mol Wt[a]
Glycerol	$C_{12}H_{32}O_3Si_3$[b,d]	96	10	1.4149	0.874	88.9	88.4	46.7	10.4	27.3	308.6	46.8	10.6	27.2	297
1,1,1-Trimethylolethane	$C_{14}H_{36}O_3Si_3$	123–124	16	1.4155	0.864	98.2	97.4	50.0	10.8	25.0	336.6	49.9	10.7	25.9	331
1,1,1-Trimethylolpropane	$C_{15}H_{38}O_3Si_3$	124–125	10	1.4201	0.870	102.8	102.0	51.4	10.9	24.1	350.6	51.7	10.9	25.0	362
Tris-(β-hydroxyethyl) cyanurate	$C_{18}H_{39}N_3O_6Si_3$	180–181	0.3	1.4618	1.043	127.0	126.0	45.3	8.2	—	—	44.7	8.5	—[c]	—
Erythritol	$C_{16}H_{42}O_4Si_4$[d]	99	1.1	Solid	—	—	—	46.8	10.3	27.3	—	46.7	10.5	27.3	—
Pentaerythritol	$C_{17}H_{44}O_4Si_4$[d]	128–129	5	1.4179	0.877	122.7	122.1	48.1	10.4	26.4	424.9	48.4	10.7	27.0	429
Galactitol	$C_{24}H_{62}O_6Si_6$[d]	149	1.6	Solid	—	—	—	46.8	10.2	27.4	—	47.1	10.3	26.4	—
Mannitol	$C_{24}H_{62}O_6Si_6$[d]	149	1.7	1.4310	0.914	176.1	174.2	46.8	10.2	27.4	—	47.0	10.3	27.3	—
Sorbitol	$C_{24}H_{62}O_6Si_6$[d]	145	1.2	1.4310	0.915	176.1	174.1	46.8	10.2	27.4	—	46.9	10.4	26.8	—

[a] Determined cryoscopically in benzene. [b] This compound was prepared previously by Breederveld, Steuns, and Waterman; ref. 7. [c] Nitrogen: Calc'd, 8.8; Found, 9.1. [d] The infrared spectra of these compounds were recorded, using a Perkin Elmer Infrared Spectrophotometer, Model 21. See footnote[e], Table I.

TABLE III

BIS-(β-TRIMETHYLSILOXYETHYL) DICARBOXYLATES

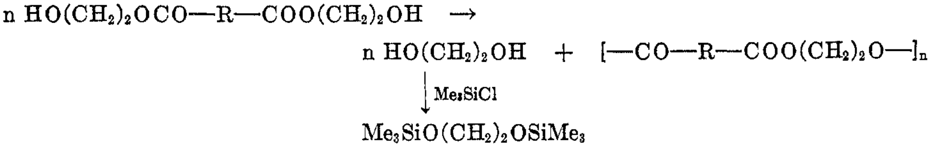
Dialkyl dicarboxylate	Formula of Product	Yield, %	b.p., °C.	Press., mm.	n_D^{20}	d_4^{20}	Analyses						Sapon. Equiv.		MRD	
							Calculated			Found						
							C	H	Si	C	H	Si	Calc'd	Found	Calc'd	Found
Diethyl oxalate	$C_{12}H_{26}O_6Si_2$	50	130	1.0	1.4310	1.023	44.7	8.1	17.4	44.4	8.2	17.4	161.2 322.4[a]	160.5 320.0[a]	82.05	81.60
Diethyl maleate	$C_{14}H_{28}O_6Si_2$	38	148–150	0.6–1.0	1.4460	1.053	48.3	8.1	—	48.9	8.2	—	174.3	170.0	90.8	88.3
Diethyl phthalate	$C_{18}H_{30}O_6Si_2$	50	156–162	0.5	1.4770	1.058	54.2	7.6	14.1	54.5	8.2	14.0	199.3	201.0	106.2	106.5
Dimethyl terephthalate	$C_{18}H_{30}O_6Si_2$	37	170–174	0.3–0.4	1.4812	1.062	54.2	7.6	14.1	53.5	7.9	14.1	199.3	198.0	106.2	106.9

[a] During titration with alcoholic alkali in the cold, only one ester group was hydrolyzed.

were prepared as follows: one mole of the dimethyl or diethyl dicarboxylate, 4 moles of ethylene glycol, and 0.5 g. of sodium methoxide were heated with stirring under a Vigreux column and the alcohol (MeOH or EtOH) was distilled continuously as formed. The final reaction temperature was approximately 200°. The system was then put under a vacuum and the 2 moles of excess glycol were distilled (b.p. ~76–83°/5 mm.). An abrupt rise in the pot temperature occurred at the end of the distillation. This indicated that little, if any, ethylene glycol had been produced by disproportionation. It was not necessary to remove the alkaline catalyst, since the next step involved reaction in presence of an amine. The syrupy reaction product was filtered through a coarse sintered glass disc. Pyridine (2.2 moles) and the calculated quantity (2.0 moles) of trimethylchlorosilane then were added. After completion of the reaction, 300–400 ml. of ether was added to aid filtration and the pyridine hydrochloride was filtered. The ether and excess pyridine were removed at atmospheric pressure and the product was distilled under a vacuum. Some bis-(trimethylsiloxy)ethane always was obtained as a by-product. A typical sample had b.p. 71–72°/5 mm., n_D^{20} 1.4034.

Anal. Calc'd for $C_8H_{22}Si_2O_3$: C, 46.5; H, 10.7; Mol. wt., 206.4.
Found: C, 46.6; H, 11.2; Mol. wt., 188 (in benzene).

There was an appreciable distillation residue, either a viscous syrup or a brittle resin. The yields of bis-(β-trimethylsiloxyethyl) dicarboxylates were 40–50% of the theoretical amount. Disproportionation occurred, at some stage of the reaction, to give 1,2-bis-(trimethylsiloxy)ethane and a polyester, presumably as follows:



$$n\,HO(CH_2)_2OCO{-}R{-}COO(CH_2)_2OH \rightarrow$$
$$n\,HO(CH_2)_2OH + [{-}CO{-}R{-}COO(CH_2)_2O{-}]_n$$
$$\Big\downarrow{\scriptstyle Me_3SiCl}$$
$$Me_3SiO(CH_2)_2OSiMe_3$$

Table III summarizes the data obtained in this series of experiments.

Acknowledgment. We wish to thank Dr. E. W. Balis and associates for the ultimate analyses and molecular weight determinations.

SUMMARY

Trimethylsilyl derivatives of polyhydric alcohols are conveniently prepared in good yield from the polyol and trimethylchlorosilane in the presence of a tertiary amine such as pyridine. They are very stable, easily distillable liquids, or low-melting solids. Representatives of each of the following classes of trimethylsilyl derivatives were prepared: mono- and bis-(trimethylsilyl) derivatives of glycols; polytrimethylsilyl derivatives of tri-, tetra-, and hexa-hydric alcohols; trimethylsilyl derivatives of bis-(β-hydroxyethyl) dicarboxylates. These substances are particularly useful in problems involving the separation and purification of difficultly isolable polyols.

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