

and in a nitrogen atmosphere, explosions occurred which shattered the apparatus and caused fires. Apparently a vapor phase reaction between ethylsilane and bromine in diffuse daylight can occur explosively even at low temperatures.

The reaction of triphenylsilanol with silicon tetrabromide, used to prepare triphenylbromosilane, is a new reaction in silicon chemistry. The reaction occurred readily on simple mixing of the reactants in ether solution and gave good yields of product. While the generality of this reaction remains to be established it is interesting to note that similar reactions have been reported in carbon¹¹ and in boron chemistry.¹²

The failure of hydrogen bromide to cause replacement of chlorine by bromine in the chlorosilanes points up a difference between silicon halides and titanium halides. Titanium tetrachloride is readily converted to the tetrabromide by reaction with hydrogen bromide. The reaction of alumi-

(11) W. Dilthey, *Ber.*, **36**, 923 (1903).

(12) A. B. Burg, *THIS JOURNAL*, **62**, 2232 (1940).

num bromide on trimethylchlorosilane gave rise to a number of compounds which could not be separated by fractional distillation. The fact that one of the products was probably dimethyldibromosilane indicates that the reaction involves cleavage of the silicon-carbon bond as well as replacement of chlorine by bromine.

In the determination of the melting points of these compounds several were found to solidify readily in a metastable form, as indicated in Table I. The method² used for the melting point determinations is favorable for the separation and observation of metastable forms. A similar metastable form was previously observed² for silicon tetrabromide.

The calculated and observed molar refractions are in good agreement except for the *p*-methoxyphenyltribromosilane. In the case of this compound an exaltation of molar refraction results from electronic interaction between the *p*-methoxyphenyl and tribromosilyl groups.

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Dialkylsilanediols¹

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RECEIVED NOVEMBER 1, 1952

Diethylsilanediol, di-*n*-propylsilanediol and di-*n*-butylsilanediol have been synthesized, and their chemical and physical properties have been investigated. Methods are described for obtaining these labile compounds in good yields by hydrolysis of the corresponding dichlorosilanes. The diols are ordinary white solids, which are stable when pure; they exhibit typical silanol reactions.

Some years ago we reported³ the synthesis and some properties of diethylsilanediol. We now wish to give a more complete account of our work on dialkylsilanediols.⁴ While organosilanediols had been known for over forty years,⁵⁻⁷ no dialkylsilanediol had been reported. The non-isolation of this type compound had generally been attributed to rapidity of intermolecular condensation to form silicones.⁸ Hydrolysis studies on diethyl-

dichlorosilane had previously failed to give the diol.⁹

By careful control of conditions during the hydrolysis and the working up of the product we have prepared diethylsilanediol, di-*n*-propylsilanediol and di-*n*-butylsilanediol in good yield from the corresponding dichlorosilanes. Key features of the synthesis involve employment of dilute solutions, low temperatures and short reaction times. Best results were obtained with dilute alkali or saturated sodium bicarbonate solution; however, other media such as concentrated sodium chloride solution may also be used. One of the preferred methods is exemplified by the following synthesis of diethylsilanediol.

A 6% solution of diethyldichlorosilane in dry ether was added during five minutes with stirring to the theoretical amount of 1.5 *N* alkali at 0°. Immediate separation of the ether layer and ether extraction of the water layer were followed by drying over anhydrous potassium carbonate for a short time in the cold. The ether solution of the product was then concentrated at low temperature. Addition of an equal volume of pentane followed by cooling gave a 65% yield of diethylsilanediol. Similarly di-*n*-propylsilanediol and di-*n*-butylsilane-

(1) Paper 32 in a series on organosilicon chemistry; for paper 31, see *THIS JOURNAL*, **73**, 882 (1951).

(2) Deceased.

(3) (a) Communication to the Editor: P. D. George, L. H. Sommer and F. C. Whitmore, *THIS JOURNAL*, **68**, 344 (1946); (b) P. D. George, paper presented before the Division of Organic Chemistry at the 109th Meeting of the American Chemical Society at Atlantic City, N. J., April 10, 1946.

(4) Taken from a dissertation submitted by P. D. George to the Graduate School of the Pennsylvania State College in partial fulfillment of the requirements for the Ph.D. degree, February, 1947.

(5) For references to diphenylsilanediol see C. A. Burkhard, *THIS JOURNAL*, **67**, 2173 (1945).

(6) For the preparation of the phenylethyl, benzylethyl, benzylphenyl and dibenzyl compounds see R. Robinson and F. S. Kipping, *J. Chem. Soc.*, **101**, 2148 (1912); R. Robinson and F. S. Kipping, *ibid.*, **101**, 2156 (1912).

(7) The isolation of dicyclohexylsilanediol in small yield as an unexpected by-product, N. W. Cusa and F. S. Kipping, *ibid.*, **121**, 2205 (1932), had been considered somewhat questionable until it was recently confirmed, C. Eaborn, *ibid.*, **141**, 2840 (1952).

(8) See for example E. Krause and A. von Grosse, "Die Chemie der metallorganischen Verbindungen," Borntraeger Geb., Berlin, 1937, p. 284; F. G. Rochow, *Chem. Eng. News*, **23**, 614 (1945).

(9) G. Martin and F. S. Kipping, *J. Chem. Soc.*, **95**, 313 (1909); J. F. Hyde and R. C. De Long, *THIS JOURNAL*, **63**, 1194 (1941); T. Alfrey, F. J. Honn and H. Mark, *J. Polymer Sci.*, **1**, 102 (1946).

diol were obtained in somewhat better yield; these latter compounds required less care because of their greater stability.

Hydrolyses of diethyldichlorosilane under a variety of conditions are described in the Experimental part. The results show that while diethylsilanediol is sensitive to strong acids and bases, it may be isolated from such media by use of dilute solutions, low temperatures and short reaction times. Basic hydrolysis media are preferred to acidic ones; this conclusion is in agreement with the fact that triethylsilanol is more stable toward base than toward acid.¹⁰

Diethyldiethoxysilane is not a suitable intermediate for the synthesis of diethylsilanediol. It appears that this compound is so relatively stable to hydrolysis that the conditions required to hydrolyze it are sufficiently drastic to polymerize any diol formed. A trace of diethylsilanediol was obtained when diethyldiethoxysilane was stirred for 12 hours with 1 *N* sodium hydroxide. Diethyldiethoxysilane was not perceptibly hydrolyzed upon being stirred for 24 hours with sodium bicarbonate solution at room temperature.

Diethylsilanediol is a white, orthorhombic, crystalline solid which melts at 96° and boils with decomposition at about 140°. It can be stored in ordinary corked bottles for months; even prolonged exposure to a laboratory atmosphere gave no change. The diol was isolated from a dilute sodium bicarbonate solution after standing at room temperature for a week. Solubilities are tabulated below. In general, diethylsilanediol is soluble at room temperature in water and in low molecular weight oxygen-containing organic solvents; it is insoluble in saturated aqueous sodium chloride, hydrocarbons, chloroparaffins and nitroparaffins. Diethylsilanediol may be recrystallized from acetone-pentane, chloroform, or carbon tetrachloride; however ether-pentane is the most reliable recrystallizing solvent. Refractive indexes and other crystal properties are given below.

Diethylsilanediol burns easily with a flame characteristic of organosilicon compounds, and it reacts readily in ether with sodium and with phosphorus pentachloride. Reaction with thionyl chloride in the presence of quinoline gave a 50% yield of diethyldichlorosilane. Concentrated hydrochloric acid polymerizes diethylsilanediol readily even in the cold. Thermal dehydration, performed in very dilute di-*n*-butyl ether solution to encourage intramolecular reaction, gave only polydiethylsiloxanes; no diethyloxosilane was isolated. Diethylsilanediol was recovered unchanged after being refluxed for one-half hour with a dilute ether solution of iodine; however, refluxing for two hours in benzene containing iodine gave quantitative dehydration.

Di-*n*-propylsilanediol and di-*n*-butylsilanediol were both obtained as soft, fluffy, white, fibrous needles melting at 100 and 96°, respectively. The butyl compound was insoluble in water, but it dissolved readily in oxygenated organic solvents. It was insoluble in the cold but soluble when heated

in ligroin, benzene, higher ethers and chlorinated solvents. An anhydrous ether solution reacted readily with sodium and with phosphorus pentachloride.

Our work on dialkylsilanediols was undertaken because they probably play a part in the mechanism of formation of industrially important silicones and also because of the possibility of copolymerizing the diols with other organic and organosilicon monomers. Our method of hydrolysis has since been applied to the preparation of other silanols.¹¹ The hydrolysis of dimethyldichlorosilane by another method to form tetramethyldisiloxane-1,3-diol¹² has since been described. Higher dimethylpolysiloxanediols have been obtained from the reaction of dimethylpolysiloxanes with water at elevated temperature and pressure.¹³ The synthesis of silanediols containing *t*-butyl groups has been reported,¹⁴ and the condensation of such diols with diethoxysilanes has been described as an improved method for making certain silicone copolymers.¹⁵ Other branched dialkylsilanediols have recently been reported.¹⁶

Experimental

Diethylsilanediol. Method A. Hydrolysis of Diethyldichlorosilane with Aqueous NaOH.—In a two-liter, three-neck flask equipped with stirrer, thermometer and dropping funnel and surrounded by a salt-ice-bath was placed 300 ml. of water and 16 g., 0.40 equivalent, of sodium hydroxide pellets. In a separate vessel, 30 g., 0.38 equivalent, of diethyldichlorosilane¹⁷ was dissolved in 500 ml. of anhydrous ether. When the reactants had been cooled to 0°, the halide solution was added with vigorous stirring over a period of four minutes to the aqueous alkali layered with 100 ml. of ether. After the addition was complete, the reaction temperature was 4°; the phenolphthalein previously placed in the aqueous layer remained pink throughout the addition.

The ether solution of product plus a single ether extract of the aqueous layer were placed over anhydrous potassium carbonate within ten minutes after the addition was complete. After filtration two hours later, the cold solution was concentrated to about 250 ml. by evaporation with a stream of air on the steam-bath. The flow of air and steam was so regulated that the temperature of the evaporating solution was maintained below 10°. The colorless concentrate was heated to boiling, and about 250 ml. of boiling pentane was added. In a few minutes micro-crystals began to appear and the solution was refrigerated.

Several hours later a 13-g. crop of white, elongated plates melting at 95–96° was obtained. Concentration of the mother liquor as before to 75 ml. gave, upon chilling, 2 g. of shiny, white, microplates melting at 95–96°.¹⁸ Evaporation of the second mother liquor *in vacuo* at 50° left only a smear. The two crops constituted a 65% yield of diethylsilanediol.

Repetition of the above run with a two-hour addition period at 0–5° gave only a 30% yield. A similar hydrolysis done in 40 minutes at room temperature gave a 44% yield.

(11) M. Sveda, U. S. Patents 2,561,429 and 2,562,000 (July 24, 1951).

(12) G. R. Lucas and R. W. Martin, U. S. Patent 2,600,307 (June 10, 1952); G. R. Lucas and R. W. Martin, *THIS JOURNAL*, **74**, 5225 (1952).

(13) E. L. Warrick, U. S. Patent 2,607,792 (August 19, 1952).

(14) L. J. Tyler, Doctoral Dissertation, The Pennsylvania State College, 1948.

(15) L. J. Tyler, U. S. Patent 2,605,274 (July 29, 1952).

(16) C. Eaborn, *J. Chem. Soc.*, **141**, 2840 (1952).

(17) Available from Dow Corning Corp., Midland, Michigan. It is desirable that the dialkyldichlorosilanes used in this synthesis be free of iron, else troublesome emulsions may be encountered.

(18) The heat sensitivity of the material made it desirable to determine the melting point by inserting the sample capillary into a bath preheated to 75° and then raising the temperature at the rate of 5° per minute.

(10) L. H. Sommer, E. W. Pietrusza and F. C. Whitmore, *THIS JOURNAL*, **68**, 2282 (1946).

When the order of addition was reversed, so that the alkali was added to the chlorosilane solution in one hour at 0°, only polydiethylsiloxanols were obtained.

Method B. Hydrolysis of Diethyldichlorosilane with Aqueous NaHCO₃.—In a two-liter, three-neck flask equipped with stirrer, thermometer and dropping funnel was placed 50 g., 0.60 equivalent, of sodium bicarbonate, 400 ml. of tap water, 100 ml. of ether and a few drops of methyl red indicator. The dropping funnel was filled with 500 ml. of ethereal solution containing 23 g., 0.29 equivalent, of diethyldichlorosilane. The addition of the halide solution to the bicarbonate suspension was carried out over a period of 40 minutes at 20°. The indicator remained orange during the entire addition.

The yellow ether layer and two 100-ml. ether extracts of the aqueous layer were placed over a mixture of anhydrous potassium carbonate and Norit A activated carbon; the aqueous layer was discarded. After five minutes, the ether solution of product was filtered, and the colorless filtrate was concentrated to about 200 ml. by evaporation with a stream of air while heating on the steam-bath. The flow of air and steam was so regulated that the temperature of the evaporating solution was kept below 10°. The concentrate was heated to boiling and 200 ml. of boiling pentane was added.

After cooling overnight, there was obtained 11 g. of shiny white needles melting at 95–96°. Addition of 100 ml. of pentane to the mother liquor and further chilling gave only 0.3 g. more of crystals, the melting point of which was not determined. Evaporation of the mother liquor to dryness left only about 1 g. of viscous oil. The 11-g. first crop constituted a 63% yield of diethylsilanediol.

Repetition of the above run using only about one-third as much water gave a 63% yield of diethylsilanediol, m.p. 94–95°. Another hydrolysis using cooling and an aqueous medium saturated both with sodium bicarbonate and sodium chloride gave a 65% crude yield of diethylsilanediol. In this case the reaction was carried out so rapidly that the hydrolysis medium was acidic during the 3-minute addition period. In a similar run in which the sodium chloride was omitted, the yield of diethylsilanediol was about the same.

Diethylsilanediol boils with decomposition at about 140° at atmospheric pressure. The determination was performed in a micro boiling point tube according to the method described by Shriner and Fuson.¹⁹

The solubilities of diethylsilanediol in a variety of solvents are tabulated below: s, soluble at least to the extent of 0.1 g. in 3 ml. of solvent; i, no visible solution; p, partly soluble; y, recrystallization took place on cooling the hot solution; dash (–) = no observation made.

SOLUBILITIES OF (C₂H₅)₂Si(OH)₂

Solvent	Cold ^a	Hot ^b	Solvent	Cold ^a	Hot ^b
Water ^c	s	...	Chloroform	i	s y
Satd. aq. NaCl	i	...	Carbon tetra-		
Methanol	s	...	chloride	i	s y
95% ethanol	s	...	Acetone	s	...
Isopropyl alcohol	s	...	Ethyl acetate	s	...
<i>t</i> -Butyl alcohol	s	...	Glacial acetic	s	...
Pentane	i	i	Dioxane	s	...
Ligroin	i	p y	Di- <i>n</i> -butyl ether	i	s y
Benzene	i	s y	Dry pyridine	s	...
Xylene	i	s y	Nitromethane	i	s y
Ethyl bromide	i	i	2-Nitropropane	i	s y
Methylene chloride	i	p y	Camphene	i	s

^a Room temperature. ^b At boiling point of solvent if less than 60°; otherwise at 60°. ^c The aqueous solution of diethylsilanediol was neutral to both red and blue litmus.

Diethylsilanediol may be recrystallized from ether-pentane, acetone-pentane, benzene, xylene, chloroform or carbon tetrachloride. Of these solvents, the most reliable was found to be ether-pentane; spontaneous dehydration was found to occur occasionally with some of the other solvents. The recrystallization of diethylsilanediol is somewhat tricky;

(19) Shriner and Fuson, "Identification of Organic Compounds," John Wiley and Sons, New York, N. Y., 1940, p. 93.

it should be borne in mind that heating time, particularly at temperatures above 40°, should be kept to a minimum.

The crystal properties of diethylsilanediol were determined by Dr. M. L. Willard of this Laboratory. The crystals obtained from ether-pentane were clear-cut, thin, transparent, orthorhombic plates. They exhibited empty mother liquor inclusions and oriented small crystals on the surface. Optical anomalies were observed which indicated growth under strain or structural instability. The optical axial plane was 001 $X = a$, and $B_{X_a} = \alpha$. The 100 face was length fast and $Y = c$ and $Z = b$. $2V = 60^\circ$ and refractive indexes were $\alpha = 1.413$, $\beta = 1.493$ and $\gamma = 1.517$.

Anal. Calcd. for C₄H₁₂O₂Si: C, 40.0; H, 10.1; Si, 23.3; mol. wt., 120; H₂O of dehydration, 15.0. Found: C, 39.9, 40.0; H, 10.1; Si, 23.0, 23.1, 22.7, 22.9; mol. wt., 139, 142, 139²⁰; H₂O of dehydration, 14.7.²¹

Other Hydrolyses of Et₂SiCl₂.—In addition to the above described preferred methods, other hydrolyses of diethyldichlorosilane were investigated. A few of these are briefly described; procedural details were similar to those described above.

(a) **With H₂O.**—Addition of a 9% solution of diethyldichlorosilane in ether to water at room temperature gave only polydiethylsiloxanols.

(b) **With Saturated Aqueous NaCl.**—Addition of a 6% solution of diethyldichlorosilane in ether to a saturated aqueous sodium chloride solution at 0–5° gave a 40% yield of diethylsilanediol, m.p. 93°. From which a yellow impurity, presumably ferric chloride, was removed with decolorizing carbon.

(c) **With Concentrated Hydrochloric Acid.**—Stirring 15 ml. of diethyldichlorosilane with 20 ml. of concentrated hydrochloric acid for one hour at 0–5° resulted in 50% hydrolysis. The major product isolated by distillation was crude tetraethyl-1,3-dichlorodisiloxane.

(d) **With Ammonium Hydroxide.**—When a 50% solution of diethyldichlorosilane in ether was added to 20% excess concentrated ammonium hydroxide over a period of three minutes with no cooling, there was obtained only about 1 g. of crystalline material. In another run addition of a 25% solution of diethyldichlorosilane in ether to excess 1:1 ammonium hydroxide over a period of 11 minutes at 0° also gave poor results. There was obtained what appeared to be about one-quarter of the usual amount of crystalline material; however, on standing in contact with the mother liquor the product underwent spontaneous dehydration.

Hydrolysis of Diethyldiethoxysilane. (a) **With Aqueous NaOH.**—A mixture of 30 g. of diethyldiethoxysilane and 450 ml. of 1 *N* sodium hydroxide was stirred vigorously for 12 hours at room temperature. The 12 g. upper layer was then discarded. Concentration *in vacuo* of five dried 100-ml. ether extracts of the aqueous layer gave a few grams of oily residue containing about 0.1 g. of minute crystals. This material, after recrystallization from ether-ligroin, melted at 94°¹⁸ and gave no depression with an authentic sample of diethylsilanediol.

(b) **With Aqueous NaHCO₃.**—A mixture of 30 g. of diethyldiethoxysilane, 400 ml. of water and 50 g. of sodium bicarbonate was stirred vigorously for 24 hours at room temperature. On concentrating *in vacuo* the dried solution composed of the upper layer and five 100-ml. ether extracts of the aqueous layer, there was obtained 25 g. of ether-free residue. No crystals were visible. Distillation showed that over 95% of this material boiled below 160° thus indicating that little or no hydrolysis of the diethyldiethoxysilane had occurred.

Di-*n*-propylsilanediol.—In the usual apparatus was placed 400 ml. of 0.75 *N* sodium hydroxide. To this solution was added over a period of five minutes with vigorous

(20) Determined cryoscopically in redistilled stock dioxane whose constant (4.90) was checked with benzene. While the values obtained were somewhat high, they served well to differentiate between silanediol and disiloxanediol.

(21) A 15-g. sample was heated for two hours in 100 ml. of boiling benzene containing about 0.5 g. of iodine. The water evolved was measured volumetrically. Prolonged heating after the theoretical amount of water had been obtained gave no further dehydration. The liquid dialkylsilicones were isolated by evaporation of the benzene after removal of the iodine with antimony or sodium sulfite followed by treatment with decolorizing carbon: diethylpolysiloxane n^{20}_D 1.4375, di-*n*-propylpolysiloxane n^{20}_D 1.4412, and di-*n*-butylpolysiloxane n^{20}_D 1.4460.

stirring at 0–10°, 25 g., 0.27 equivalent, of di-*n*-propyldichlorosilane. The ether layer was separated immediately and placed over anhydrous potassium carbonate together with a 200-ml. ether extract of the aqueous layer.

After being dried for about 15 minutes, the ether solution of product was evaporated to dryness with a stream of air while being heated on the steam-bath. The white, solid residue was taken up in 350 ml. of ligroin and the solution was distilled for a moment until the distillate came over clear.

On cooling slowly to room temperature, the solution set to a solid, white, crystalline mass. Chilling to 0° followed by filtration gave 14.7 g. of fluffy, soft, white, fibrous needles melting at 99–100°¹⁹ before and after sublimation *in vacuo*. This material constituted a 74% yield of di-*n*-propylsilanediol.

Anal. Calcd. for C₆H₁₆O₂Si: C, 48.6; H, 10.9; mol. wt., 148; H₂O of dehydration, 12.2. Found: C, 48.5, 48.9; H, 10.6, 11.1; mol. wt., 178²⁰; H₂O of dehydration, 12.0.²¹

Di-*n*-butylsilanediol.—To solution of 13 g., 0.33 equivalent, of sodium hydroxide in 400 ml. of water was added at 0–10° with vigorous stirring over a period of ten minutes 33 g., 0.31 equivalent, of di-*n*-butyldichlorosilane dissolved in 400 ml. of anhydrous ether. After immediate separation of the layers, the ether solution of product and a 200-ml. extract of the aqueous layer were dried for about 15 minutes over anhydrous potassium carbonate.

The dried ether solution of product was then evaporated to dryness with a stream of air while being heated on the steam-bath. The white solid residue was taken up in 350 ml. of ligroin and the solution was boiled a moment until the distillate came over clear.

On cooling slowly to room temperature, the solution deposited no crystals; however after short chilling in the refrigerator, the solution became completely solid with white, crystalline material. Filtration gave 20.3 g. of fluffy, soft, white, fibrous needles melting at 95–97°.¹⁸ This material constituted a 75% yield of di-*n*-butylsilanediol.

Anal. Calcd. for C₈H₂₀O₂Si: C, 54.6; mol. wt., 176; H₂O of dehydration, 10.2. Found: C, 54.3; mol. wt., 199²⁰; H₂O of dehydration, 10.0.²¹

Di-*n*-butylsilanediol was insoluble in water, but it was readily soluble in ether, methanol, 95% ethanol, acetone, dioxane and pyridine. The diol was insoluble cold but soluble hot in methylene chloride, chloroform, carbon tetrachloride, ligroin, benzene, di-*n*-butyl ether, nitromethane and 2-nitropropane. An anhydrous ethereal solution of the diol reacted vigorously with sodium and with phosphorus pentachloride. Di-*n*-butylsilanediol burned readily with a flame characteristic of organosilicon compounds.

Reactions of Diethylsilanediol. (a) Dehydration.

After being refluxed for one-half hour in a dilute ether solution of iodine, diethylsilanediol was recovered unchanged. Refluxing for five minutes in benzene also gave no dehydration. However, when iodine was added, quantitative dehydration occurred in two hours.²¹ The diol was also quantitatively dehydrated upon boiling for four hours in di-*n*-butyl ether. Even though a 1% solution was used in order to encourage intramolecular dehydration, no diethyloxosilane was obtained. Careful fractional distillation gave nothing boiling below di-*n*-butyl ether, and the early fractions of the latter contained no silicon.

(b) **With Thionyl Chloride.**—In a 500-ml. flask equipped with stirrer and dropping funnel and surrounded with an ice-bath was placed 50 g. of dry quinoline, 100 ml. of anhydrous ether and 37 g. of freshly distilled thionyl chloride. When these reactants were combined, there was some reaction, and a brick-red lower layer separated.

To the mixture was added with vigorous stirring over a period of one-half hour 12 g., 0.10 mole, of diethylsilanediol dissolved in 200 ml. of anhydrous ether. A white solid formed momentarily at the point of mixing, but it rapidly changed into a red oil. After all the diol had been added, the cloudy yellow ether layer was decanted from the brick-red sticky residue. Fractional distillation gave 8 g., 50% yield, of diethyldichlorosilane, b.p. 128–129 at 735 mm.²²

Anal. Calcd. for C₄H₁₀Cl₂Si: Cl, 45.1; neut. equiv., 78.6. Found: Cl, 45.0, 45.0; neut. equiv., 79.2, 79.2.

(c) **Hydrochloric Acid.**—Addition of diethylsilanediol to concentrated hydrochloric acid at 0° resulted in rapid polymerization. No diethyldichlorosilane or low molecular weight diethylchloropolysiloxanes were obtained.

(d) **Other Reactions.**—Diethylsilanediol burned readily with a flame characteristic of organosilicon compounds. An anhydrous ethereal solution of the diol reacted vigorously with sodium and with PCl₅. Diethylsilanediol appeared to be quite stable in slightly basic solution at room temperature; the compound was isolated from a dilute sodium bicarbonate solution after standing at room temperature for a week.

Acknowledgment.—We wish to thank the following for providing analyses and intermediates: D. L. Bailey, E. W. Balis, G. T. Kerr, N. S. Marans, L. J. Tyler and R. N. Walter.

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(22) A. Ladenburg, *Ann.*, **164**, 310 (1872); G. Martin and F. S. Kipping, *J. Chem. Soc.*, **95**, 302 (1909).