

II. THE REACTION OF CHLOROSILANES WITH 2-METHOXYETHANOL

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The reaction of organochlorosilanes with 2-methoxyethanol has been shown to give good yields of 2-methoxyethoxyorganosilanes. The formation of silyl ethers by reaction of an organochlorosilane with alcohols has been described (2); however, the ethers which have been reported in previous publications have been insoluble in water, or at most have shown only slight solubility. In contrast, the introduction of the 2-methoxyethoxy radical very greatly increases the water solubility of the resulting silyl ether as compared to the corresponding *n*-butoxy ether, or even the ethoxy ether. The extent to which increased solubility is effected is dependent upon the size and number of hydrocarbon radicals that are directly attached to the silicon atom. The silyl ethers that contain the 2-methoxyethoxy radical are of interest in those cases where a homogeneous aqueous system of silanes is desired.

TABLE I
2-ALKOXYETHOXY ETHERS OF ORGANOSILANES

COMPOUND	B.P., °C. (MM)	n_D^{20}	d_4^{20}	MR_D^a		ANALYSIS				SOLUBILITY IN WATER
						Calc'd		Found		
				Calc'd	Found	C	H	C	H	
Methyltris-2-methoxyethoxysilane	145 (16)	1.4200	1.0454	65.1	65.0	44.75	9.02	43.8 43.4	8.9 8.9	∞
Dimethylbis-2-methoxyethoxysilane	203-204	1.4114	0.9663	53.5	53.6	46.13	9.68	45.8 46.3	9.5 9.6	∞
Trimethyl-2-methoxyethoxysilane	128	1.3952	.8492	41.9	41.9	48.61	10.88	49.0	11.0	ca. 5%
Azeotrope ^b	118	1.3988	.8936							
Methylbenzylbis-2-methoxyethoxysilane	181-185 (15)	1.4795	1.0141	77.5	79.6	59.12	8.51	59.1	8.7	ca. 5%
Methyl-2-pentylbis-2-methoxyethoxysilane	146-152 (15)	1.4298	0.9454	71.1	72.2	54.50	10.67	53.4	10.0	ca. 5%
Phenyltris-2-methoxyethoxysilane	204 (15)	1.4727	1.0818	84.1	85.6	54.52	7.93	54.0	8.1	ca. 25%
Dimethylbis-2-ethoxyethoxysilane	136 (30)	1.4131	0.9368	62.0	62.9	50.81	10.24	51.7	10.6	ca. 50%

^a Sauer, *J. Am. Chem. Soc.*, **69**, 701 (1947).

^b This is an azeotrope of trimethyl-2-methoxyethoxysilane and 2-methoxyethanol (approx. 50-50).

The preparation of *tetrakis*-2-methoxyethoxysilane and *tris*-2-methoxyethoxychlorosilane from 2-methoxyethanol and silicon tetrachloride was described in a previous publication (1). Both of these compounds are miscible with water in all proportions.

These new silyl ethers appear to possess reasonable hydrolytic stability. Aqueous solutions of dimethylbis-2-methoxyethoxysilane remain clear for approximately fifteen to thirty minutes before clouding is observed. The extent of clouding increases slowly as the solution is allowed to stand. After 24 hours only very little dimethylsilicone oil separates from the solution. If a small amount of dilute acid or base is added to the clear aqueous solution of dimethylbis-2-methoxyethoxysilane an oil layer separates almost immediately.

The 2-ethoxyethoxy radical also promotes water solubility of the silyl ethers but to a somewhat lesser extent than the 2-methoxyethoxy radical.

The 2-methoxyethoxy ethers of the following organochlorosilanes have been prepared: methyltrichloro-, dichlorodimethyl-, chlorotrimethyl-, benzylmethyldichloro-¹, methyl-2-pentyldichloro-, and phenyltrichloro-silane. The 2-ethoxyethoxy ether of dichlorodimethylsilane was also prepared. The physical properties and analyses of these compounds are tabulated in Table I.

Trimethyl-2-methoxyethoxysilane and 2-methoxyethanol form an azeotrope whose composition is approximately 50% 2-methoxyethanol.

EXPERIMENTAL

The following procedure was used in the preparation of the ethers.

The organochlorosilane is placed in a three-necked flask, which is fitted with a dropping-funnel, thermometer, and condenser. A tube filled with Drierite is attached at the end of the condenser. A slight excess of the 2-alkoxyethanol is added dropwise while the mixture is heated at reflux with an electric mantle. The heating is continued after complete addition of the alcohol until hydrogen chloride evolution ceases. The products are then distilled.

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REFERENCES

- (1) BURKHARD, *J. Org. Chem.*, **13**, 879 (1948).
- (2) BURKHARD, ROCHOW, BOOTH, AND HARTT, *Chem. Revs.*, **41**, 97 (1947).

¹ Dr. B. A. Bluestein supplied the sample of benzylmethyldichlorosilane. He will describe this compound in a future publication.