

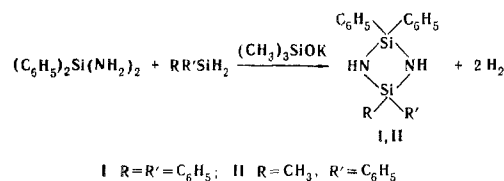
# CYCLIZATION DURING THE REACTION OF DIPHENYLDIAMINOSILANE WITH ORGANOSILANES

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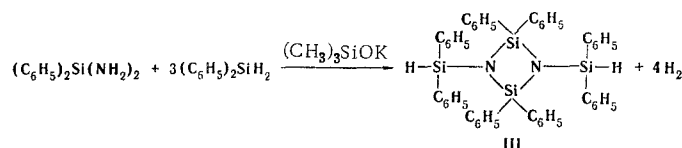
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Four-membered cyclosilazans were obtained as a result of the reaction of diphenyldiaminosilane with diphenylsilane and methylphenylsilane in the presence of potassium trimethylsilanolate.

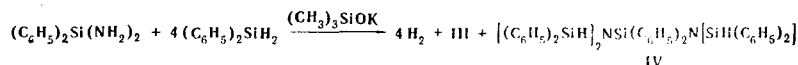
The formation of four-membered rings is observed in the reaction of organoaminosilanes or silazans with organosilanes [1]. However, organic or organosilyl groups were found to be attached to the nitrogen atoms in all of the rings obtained. We have studied the reaction of diphenyldiaminosilane with diphenylsilane and methylphenylsilane at various reagent ratios in and without anisole in the presence of potassium trimethylsilanolate as a catalyst. The chief reaction products when the molar ratio of diphenyldiaminosilane to diphenylsilane and methylphenylsilane is 1:1 are tetraphenylcyclodisilazan (I) and triphenylmethylcyclodisilazan (II), respectively.



When the molar ratio of diphenyldiaminosilane and diphenylsilane is 1:3, the major reaction product is N,N'-bis(diphenylsilyl)tetraphenylcyclodisilazan (III):



Approximately equal quantities of cyclodisilazan III and N,N,N',N'-tetrakis(diphenylsilyl)diphenyldiaminosilane (IV) were obtained when the molar ratio of diphenyldiaminosilane and diphenylsilane was 1:4:



All of the indicated reactions proceed in two steps. About 50% of the theoretical amount of hydrogen is evolved in the first step, which occurs at 20°C. The remainder of the hydrogen is evolved only on heating to 170°.

## EXPERIMENTAL

All of the reactions were carried out in a three-necked flask equipped with a hermetic seal and a device for introduction of the catalyst. After hydrogen evolution had ceased at 20°, the reaction flask was placed in a thermostat at 170°, and the reaction was allowed to proceed until hydrogen evolution ceased. The reaction products were isolated by recrystallization from petroleum ether. The molecular weight was determined by cryoscopy in camphor.

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1,1,3,3-Tetraphenylcyclodisilazan (I). The reaction of 3.10 g (14.5 mmole) of diphenyldiaminosilane and 2.63 g (14.5 mmole) of diphenylsilane in the presence of 8.81 g (81.5 mmole) of anisole and 0.080 g (0.62 mmole) of potassium trimethylsilanolate gave 3.1 g (52%) of tetraphenylcyclodisilazan I with mp 206°. The IR spectrum did not contain bands characteristic for  $\text{NH}_2$  and  $\text{Si-H}$  groups but did contain an  $\text{N-H}$  band at  $3395\text{ cm}^{-1}$ . Found: C 73.0; H 5.7; N 7.3; Si 14.1%. M 450.  $\text{C}_{24}\text{H}_{22}\text{N}_2\text{Si}_2$ . Calculated: C 73.1; H 5.6; N 7.1; Si 14.2%. M 394.

N,N'-Bis(diphenylsilyl)tetraphenylcyclodisilazan (III). The reaction of 7.13 g (33.3 mmole) of phenyldiaminosilane and 18.1 g (99.9 mmole) of diphenylsilane in the presence of 2.05 g (11 mmole) of anisole and 0.135 g (1.05 mmole) of potassium trimethylsilanolate gave 17.5 g (69%) of cyclodisilazan III with mp 224-225°. The IR spectrum did not contain an  $\text{N-H}$  band but did contain an  $\text{Si-H}$  band at  $2170\text{ cm}^{-1}$ . Found: C 75.8; H 5.7; N 3.6; Si 14.8;  $\text{H}_{\text{act}}$  0.3%. M 766.  $\text{C}_{48}\text{H}_{42}\text{N}_2\text{Si}_4$ . Calculated: C 75.9; H 5.8; N 3.7; Si 14.7;  $\text{H}_{\text{act}}$  0.3%. M 758.

N,N,N',N'-Tetrakis(diphenylsilyl)diphenyldiaminosilane (IV). The reaction of 3.07 g (14.3 mmole) of diphenyldiaminosilane and 10.5 g (57.2 mmole) of diphenylsilane in the presence of 0.65 g (6.0 mmole) of anisole and 0.077 g (0.60 mmole) of potassium trimethylsilanolate gave 5.0 g (37%) of cyclodisilazan (III) and 4.5 g (36%) of diaminosilane IV with mp 163-164°. The IR spectrum contained an  $\text{Si-H}$  band at  $2140\text{ cm}^{-1}$ , but there was no  $\text{N-H}$  band in the spectrum. Found: C 77.1; H 6.2; N 2.9; Si 13.4;  $\text{H}_{\text{act}}$  0.4%. M 898.  $\text{C}_{60}\text{H}_{54}\text{N}_2\text{Si}_5$ . Calculated: C 76.4; H 5.8; N 3.0; Si 14.9;  $\text{H}_{\text{act}}$  0.4%. M 943.

1,1,3-Triphenyl-3-methylcyclodisilazan (II). The reaction of 3.53 g (29.2 mmole) of methylphenylsilane and 6.26 g (29.2 mmole) of diphenyldiaminosilane in the presence of 0.048 g (0.38 mmole) of potassium trimethylsilanolate gave 3.5 g (36%) of cyclodisilazan II with mp 65°. Found: C 68.1; H 6.1; N 8.2; Si 17.5%. M 321.  $\text{C}_{19}\text{H}_{20}\text{Si}_2\text{N}_2$ . Calculated: C 68.2; H 6.0; N 8.5; Si 16.9%. M 332.

#### LITERATURE CITED

1. W. Fink, *Helv. Chim. Acta*, **47**, 498 (1964).