

## LETTERS TO THE EDITOR

# Application of *N,N'*-Bis(trimethylsilyl)carbodiimide in the Synthesis of New Heterocyclic Compounds

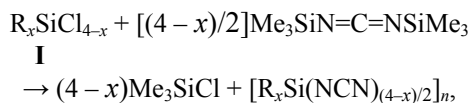
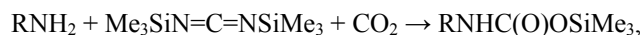
A. D. Kirilin, A. V. Gavrilova, M. G. Shamina, and V. G. Lakhtin

Lomonosov Moscow State University of Fine Chemical Technology, pr. Vernadskogo 86, Moscow, 119571 Russia  
e-mail: kirilina@rambler.ru

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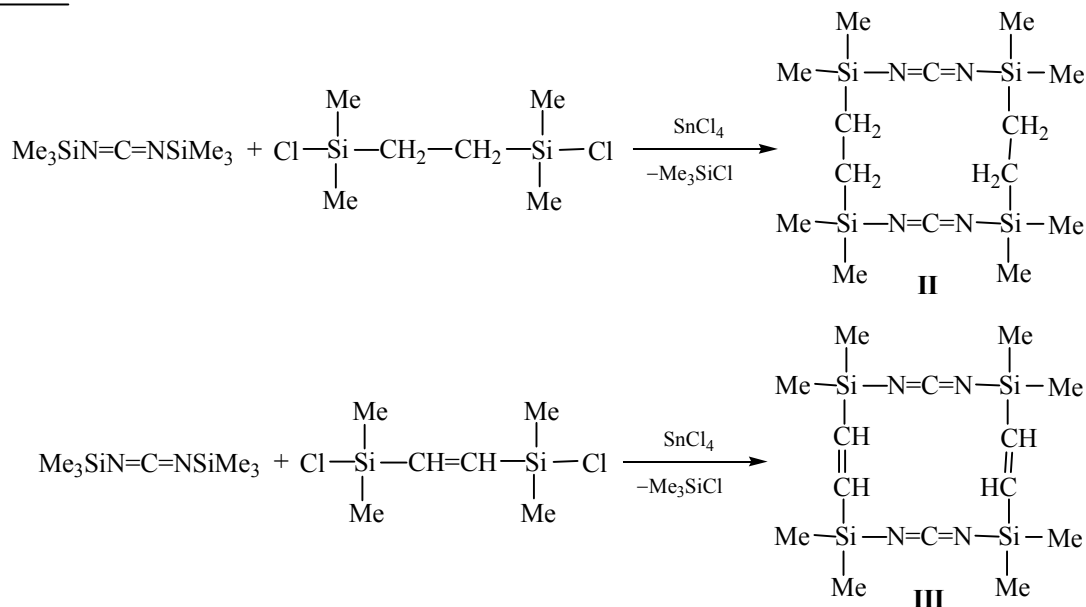
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*N,N'*-bis(trimethylsilyl)carbodiimide has been shown earlier to be useful as a *N*-siloxyacylating reagent [1] in conjunction with carbon dioxide for obtaining *O*-silyl uretanes, and in conjunction with the organochlorosilanes of  $R_nSiCl_{4-n}$  type in the synthesis of oligoorganosilylcarbodiimides [2].



We found that using *N,N'*-bis(trimethylsilyl)carbodiimide and organylchlorosilanes the silicon-containing heterocyclic compounds can be easily synthesized.

It turned out that the replacement of organochlorosilanes **I** by 1,2-bis(dimethylchlorosilyl)ethane or 1,2-bis(dimethylchlorosilyl)ethylene does not change the nature of the process: The same condensation of the starting materials proceeds followed by the release of trimethylchlorosilane. However, instead of the formation of oligoorganosilylcarbodiimides the process ends with the formation of stable novel heterocyclic compounds **II** and **III**.



All starting compounds and solvents were thoroughly dried before use and purified by distillation. Synthetic operations, identifying and sampling for analysis of the

substances was carried out in an atmosphere of dry nitrogen. The reaction mixtures and compositions of individual compounds were monitored by GLC on a

Shimadzu G-8 instrument (stainless steel column 1500×3 mm, stationary phase SE-30 on Chromaton N-AW, carrier gas helium). IR spectra were recorded on a Specord IR-75 instrument from thin layers. Gel-permeation chromatography was performed on the liquid chromatograph equipped with a Waters Ultra Styragel 10-3A column for exclusive chromatography, a Knauer WellChrom K-120 pump, with photometric detection at 254 nm. Mobile phase was tetrahydrofuran, graduation was performed on polystyrene standards with molecular masses of 580, 1700, 5050, 11 600 Da.

**1,1,5,5,8,8,12,12-Octamethyl-1,5,8,12-tetrasil-2,4,9,11-tetraazacyclotetradeca-2,3,9,10-tetraene (II).** In an Elenmeyer flask fitted with a full-condensation distillation head and a thermometer was loaded 24.2 g (0.13 mol) of bis(trimethylsilyl)carbodiimide and 50.0 ml of benzene, and then 27.9 g (0.13 mol) of bis(dimethylchlorosilyl)ethane in 50.0 ml of benzene was poured by portions. A few drops of SnCl<sub>4</sub> were added, and the reaction mixture was then heated for 16 h until trimethylchlorosilane release ceased. After evacuation for 1 h at 1 mm Hg 11 g (89%) of viscous liquid was isolated (compound II).

The IR spectrum,  $\nu$ , cm<sup>-1</sup>: 2200 (C=N). Found, %: C 45.15, H 8.67, N 15.12. *M* 371. C<sub>14</sub>H<sub>32</sub>N<sub>4</sub>Si<sub>4</sub>. Calculated, %: C 45.10, H 8.70, N 15.20.

**1,1,5,5,8,8,12,12-Octamethyl-1,5,8,12-tetrasil-2,4,9,11-tetraazacyclotetradeca-2,3,6,9,10,11-hexaene (III).** In a conical flask fitted with a nozzle of full of condensation and a thermometer was loaded 16.4 g (0.09 mol) of bis(trimethylsilyl)carbodiimide and 50.0 ml of benzene, and then was added portionwise 18.8 g (0.09 mol) of bis(dimethylchlorosilyl)ethylene in 50.0 ml of benzene. A few drops of SnCl<sub>4</sub> was added, and the reaction mixture was then heated for 19 h. After evacuation for 1 h at 1 mm Hg was isolated 10.6 g (96%) g of a viscous liquid (compound III). The IR spectrum,  $\nu$ , cm<sup>-1</sup>: 2200 (C=N). Found, %: C 46.12, H 7.76, N 15.34. *M* 368. C<sub>14</sub>H<sub>28</sub>N<sub>4</sub>Si<sub>4</sub>. Calculated, %: C 46.10, H 7.74, N 15.36.

#### REFERENCES

1. Kirilin, A.D. and Gavrilova, A.V., *Zh. Obshch. Khim.*, 2009, vol. 79, no. 11, p. 1932.
2. Kirilin, A.D., Gavrilova, A.V., Sheludyakov, V.D., Shamina, M.G., and Storozhenko, P.A., *Plastmassy*, 2010, no. 11, p. 36.