

LETTERS TO THE EDITOR

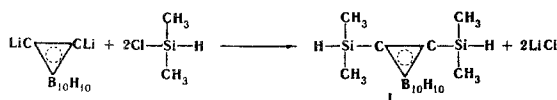
SYNTHESIS OF SOME *o*-CARBORANE DERIVATIVES

A. L. Klebanskii, V. F. Gridina, L. P. Dorofeenko, L. E. Krupnova, G. E. Zakharova, and N. I. Shkambarnaya

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 3, No. 3, p. 570, 1967

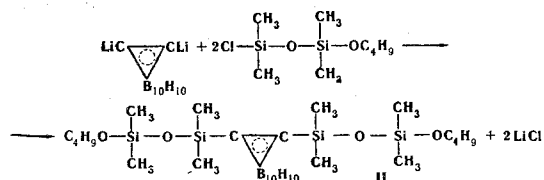
UDC 547.717'271:547.222'128

We have carried out work on the synthesis of some derivatives of *o*-carborane. Reaction of dilithium-*o*-carborane with dimethylchlorosilane has given, for the first time, 1,2-bis(dimethylsilylhydride)*o*-carborane:



The product I was a viscous oily colorless liquid, bp 121°–125° C (3 mm); d_4^{20} 0.9457; n_D^{20} 1.5360. Found: C 27.8; H 9.80; B 40.75; Si 20.90%. Calculated for $C_6H_{24}B_{10}Si_2$: C 27.7; H 9.23; B 41.50; Si 21.54%.

Reaction of dilithium-*o*-carborane with 1-chloro-3-butoxy-1,1,3,3-tetramethyldisiloxane also gave for the first time 1,2-bis(1-butoxy-1,1,3,3-tetramethyldisiloxane)*o*-carborane:



Compound II was a viscous oily colorless liquid, bp 110°–115° C (10 mm). Found: H 10.0; B 20.1; Si 20.4%. Calculated for $C_{16}H_{42}B_{10}O_4Si_4$: H 9.4; B 19.5; Si 20.2%.

A low-molecular dimethylsiloxane polymer, not containing boron, was obtained along with this product.

16 April 1966

Lebedev All-Union Scientific Research Institute for Synthetic Rubber, Leningrad

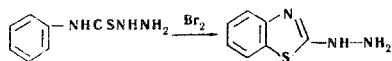
A NEW METHOD OF PREPARING 2-HYDRAZINOBENZO- AND NAPHTHOTHIAZOLES

I. L. Shegal, G. N. Tyurenkova, and I. Ya. Postovskii

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 3, No. 3, pp. 570–571, 1967

UDC 547.789.6

A paper recently published [1] stated that 4-phenylthiosemicarbazide cannot be cyclized to 2-hydroazinobenzothiazole by the method described in [2]. We have found that under certain conditions 2-hydrazinobenzothiazole can be obtained in 33% yield.



The method can also be used to prepare substituted 2-hydrazinobenzothiazoles, e.g. 6-ethoxy-2-hydrazinobenzothiazole. The appropriate naphthylthiosemicarbazides also give good yields of 2-hydrazinonaphtho-[1,2]- and -[2,1]thiazoles. In this way these hydrazines, difficultly accessible by other means, become readily accessible.

GENERAL METHOD OF PREPARING THE HYDRAZINES

A solution of 0.5 ml (0.01 mole) bromine in 15 ml $CHCl_3$ in small portions, was added with constant stirring to a suspension of 0.01 mole 4-arythiosemicarbazide in 30 ml $CHCl_3$. The whole was left for 30 min at room temperature, then heated for 40 min on a steam-bath, when there was vigorous evolution of HBr. Then the products were cooled, the precipitate of hydrazine hydrobromide filtered off, and washed with $CHCl_3$.

In the case of 2-hydrazinobenzothiazole, the solid was gently heated in 30 ml water, and SO_2 passed. The resultant tarry reaction product was filtered off, and the filtrate neutralized with alkali. The resultant pre-