THE NEW HETEROCYCLIC SYSTEMS 1-THIA-3-SILACYCLOBUTANE AND 1-THIA-3-SILACYCLOPENTANE

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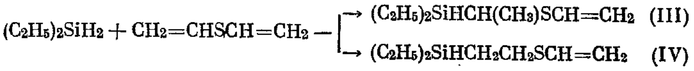
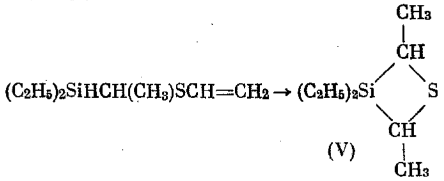
According to the data in [1], hydrosilanes are not added to the double bond in the α position in vinyl alkenyl sulfides with the general formula $CH_2=CHS(CH_2)_nCH-CH_2$. We found that in the presence of a Pt catalyst $(C_2H_5)_2SiH_2$ (I) reacts with divinyl sulfide (II) to form a mixture of the α (III) and β (IV) monoadducts with considerable predominance of III according to the scheme



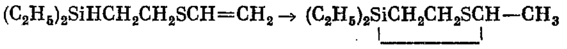
$$(C_2H_5)_2SiH_2 + CH_2=CHSCH=CH_2 - \begin{cases} \rightarrow (C_2H_5)_2SiHCH(CH_3)SCH=CH_2 & (III) \\ \rightarrow (C_2H_5)_2SiHCH_2CH_2SCH=CH_2 & (IV) \end{cases}$$

An unexpected product of the reaction was the four-membered heterocycle 2,4-dimethyl-3-diethyl-1-thia-3-silacyclobutane (V), which is the first compound containing a 1-thia-3-silethane ring. Its formation is due to the intramolecular hydrosilylation of the monadduct (III)



(V)

The ratio between the adducts (III) : (IV) : (V) is 4.5 : 2 : 1 (according to the data from gas — liquid chromatography and proton magnetic resonance). Compound V, which is also obtained by heating individual adduct III in the presence of H_2PtCl_6, is a 1 : 1 mixture of the cis and trans isomers. Under the same conditions, the intramolecular hydrosilylation of adduct IV proceeds with considerably more difficulty and results in the formation of a small amount of 2-methyl-3,3-diethyl-1-thia-3-silacyclopentane (VI), which is the first compound containing a 1-thia-3-silolane ring,



(VI)

$$(C_2H_5)_2SiHCH_2CH_2SCH=CH_2 \rightarrow (C_2H_5)_2SiCH_2CH_2SCH-CH_3$$

An equimolar mixture of I and II was heated in a sealed ampule in the presence of a Pt catalyst (60 h, 100°). Compound III (n_D^{20} 1.4850, d_4^{20} 0.8848) and a mixture of IV and V were isolated from the fraction boiling at 55-73° (3 mm Hg) by gel chromatography. The heating of adducts III and IV under the same conditions yielded compounds V (95% yield, n_D^{20} 1.4920) and VI (15% yield). The structure of all the compounds obtained was confirmed by the data from elemental analysis, IR spectroscopy, proton magnetic resonance, and mass spectrometry.

LITERATURE CITED

1. E. P. Plueddemann, Belgium Patent No. 628951 (1963); Chem. Abstrs., 60, 16103 (1964); USA Patent No. 3186965 (1965).

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