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1,3-THIASILACYCLOPENTANE AND 1,3- AND 1,4-THIASILACYCLOHEXANE DERIVATIVES

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Summary

The reaction of dimethyl(chloromethyl)alkenylsilanes, $ClCH_2Me_2Si(CH_2)_n$-$CH{=}CH_2$ ($n = 0$, 1) with alcoholic KSH solution or thiourea followed by hydrolysis in the latter case leads to the corresponding dimethyl(mercaptomethyl)alkenylsilanes which are unstable compounds readily undergoing polymerization or cyclization. Intramolecular ring closure of dimethyl(mercaptomethyl)allylsilane affords 3,3,5-trimethyl-1-thia-3-silacyclopentane or 3,3-dimethyl-1-thia-3-silacyclohexane, depending on the reaction conditions. Photochemical addition of gaseous H_2S to the above dimethyl(chloromethyl)alkenylsilanes yields 3,3-dimethyl-1-thia-3-silacyclopentane and 3,3-dimethyl-1-thia-3-silacyclohexane, respectively. H_2S adds to dimethyldivinylsilane upon UV-irradiation to give a mixture of isomeric 2,3,3-trimethyl-1-thia-3-silacyclopentane and 4,4-dimethyl-1-thia-4-silacyclohexane. Thiasilacyclopentane and thiasilacyclohexane derivatives are readily converted to the corresponding sulfonium salts by CH_3I. In contrast, 3,3-dimethyl-1-thia-3-silacyclobutane undergoes ring opening to form sulphonium dimethyl(iodomethyl)silylmethiodide, $[ICH_2Me_2SiCH_2\overset{+}{S}(CH_3)_2]I^-$.

Results and discussion

Previously, we reported a convenient route to 1,3-thiasilacyclobutane, the simplest silicon- and sulfur-containing four-membered ring [1,2]. This study is concerned with the synthesis of a series of new heterocyclic systems in which the silicon and sulfur atoms are separated by one or two carbon bridges. The reported method for the preparation of 1-thia-3-silacyclopentane derivatives is a multi-step one [3]. The reaction of α,ω-dihaloalkanes with Na_2S, widely used in organic synthesis [4], proved to be inapplicable to 1-thia-3-silacyclopentane and 1-thia-4-silacyclohexane derivatives due to instability of the starting (2-haloethyl)haloalkylsilanes [5].

Thiacycloalkanes with different ring sizes are readily obtained by intramolecular cyclization of the corresponding mercaptoalkenes [6,7]. We attempted to use a

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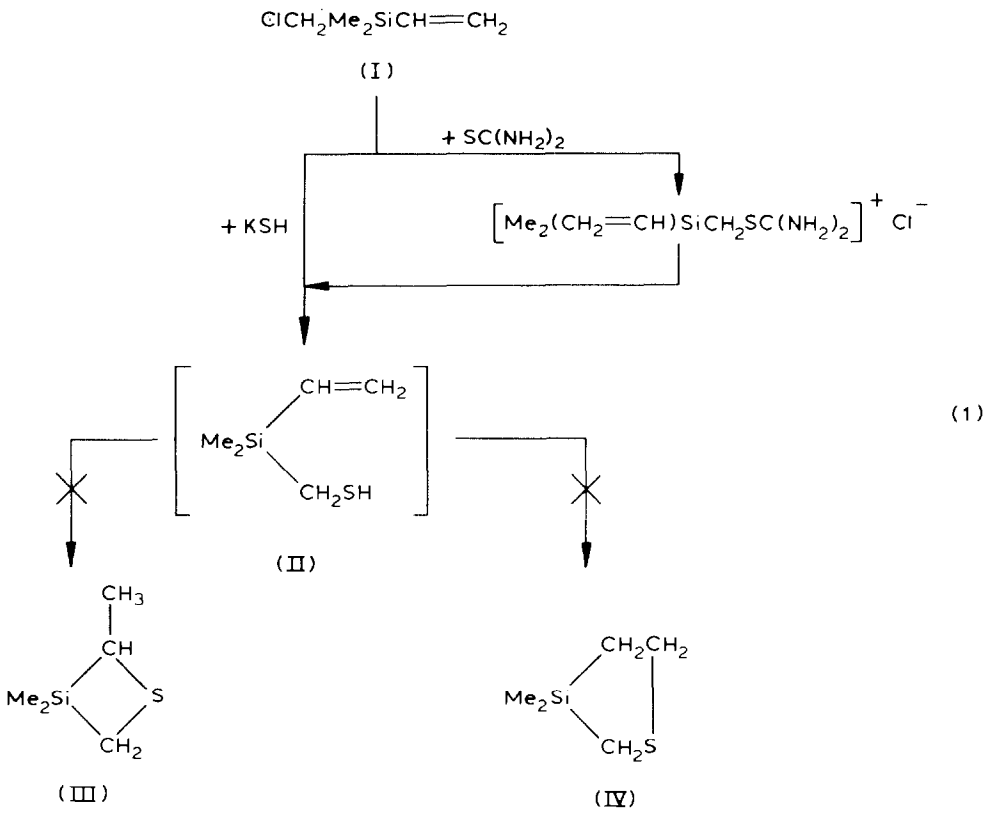
similar reaction as a synthetic route to 1,3- and 1,4-thiasilacycloalkanes. The starting dialkyl(mercaptomethyl)alkenylsilanes were prepared in two ways. Treatment of dimethyl(chloromethyl)vinylsilane (I) with potassium hydrosulfide in ethanol [8] led to dimethyl(mercaptomethyl)vinylsilane (II). The same compound (II) is obtained by the reaction of the initial silane I with thiourea followed by base hydrolysis of the isothiuronium salt thus formed.

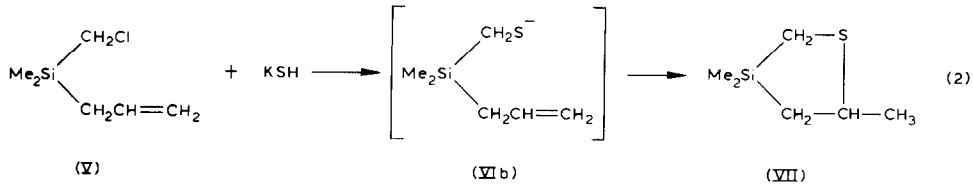


(1)

We failed to isolate thiol II in the pure form due to its instability. When dissolved in pentane in the presence of hydroquinone, thiol II can be kept in the dark at 0–5°C for a long time (about a month). Upon UV-irradiation (5 h) of a pentane solution of II none of the desired ring compounds (III and IV) were observed. Under these conditions thiol II affords only polyaddition products. Such behavior distinguishes thiol II from butene-3-thiol-1 which readily converts to thiacyclopentane (with traces of 2-methylthiacyclobutane) [6a]. The intramolecular cyclization of thiol II, or rather an intermediate thyil radical, $^{\cdot}SCH_2SiMe_2CH{=}CH_2$, into heterocycle IV seems to be hindered by steric factors. Nevertheless, compound IV was readily prepared in another way as described below. It seems that thermodynamic factors are not important in forming the 1,3-thiasilacyclopentane skeleton.

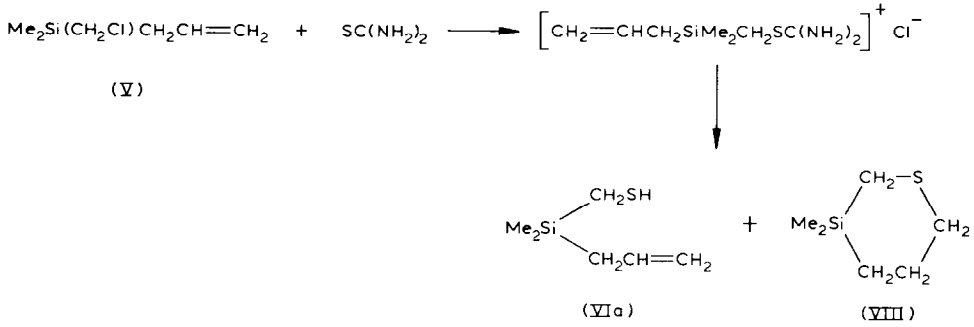
The reaction of dimethyl(chloromethyl)allylsilane with alcoholic KSH solution does not afford the desired thiol VIa. Unexpectedly, the only reaction product is 3,3,5-trimethyl-1-thia-3-silacyclopentane (VII, 60% yield).

Nevertheless we have prepared dimethyl(mercaptomethyl)allylsilane (VIa) under



(V) (VIb) (VII)

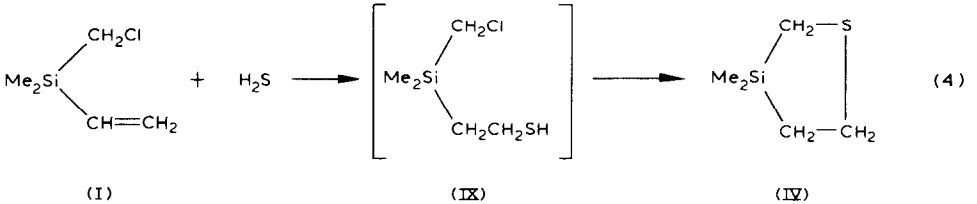
milder conditions by treatment of compound V with thiourea followed by hydrolysis of the isothiuronium salt formed.



(V) (VIa) (VIII)

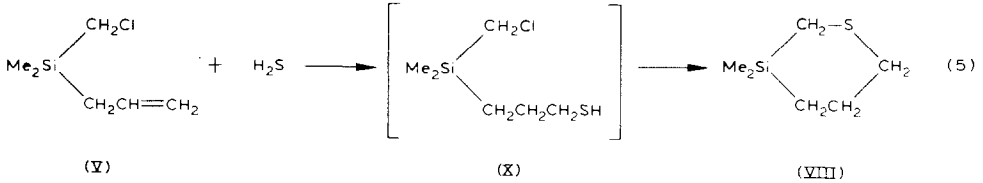
The structure of thiol VIa is confirmed by ^1H NMR spectroscopy. However, pure thiol VIa could not be isolated. In reaction 3 cyclization is observed to give 3,3-dimethyl-1-thia-3-silacyclohexane (VIII). The isomeric thiasilacycloalkanes VII and VIII seem to be formed by different mechanisms. In strongly basic media, nucleophilic addition of the thiolate anion at the allyl double bond occurs to lead to the five-membered ring compound VII. The order of addition agrees with the double bond polarization in allylsilanes [9,10]. The six-membered cyclic compound VIII results from the intramolecular ring closure of the thiyl radical. Both reactions 2 and 3 of dimethyl(mercaptomethyl)allylsilane occur selectively. In contrast, the ionic and free-radical cyclization of pentene-4-thiol-1 leads to a mixture of five- and six-membered thiacycloalkanes in ratios governed by the reaction conditions [6a,7a,b]. The assumption that the five-membered heterocycle VII is formed due to preceding isomerization of the allyl group into a propenyl one in either the starting compound V or intermediate VI should be rejected since no migration of the double bond occurs upon long heating of trimethylallylsilane with alcoholic potassium hydrosulfide.

As another route to thiasilacycloalkanes we decided to use the intramolecular cyclization of dimethyl(chloromethyl)mercaptoalkylsilanes. We tried to prepare the latter compounds by the photochemical reaction of gaseous hydrogen sulfide to the corresponding triorganylalkenylsilanes [11]. However, photoinitiated addition of gaseous H_2S to dimethyl(chloromethyl)vinylsilane (I) gave 3,3-dimethyl-1-thia-3-silacyclopentane (IV).



(I) (IX) (IV)

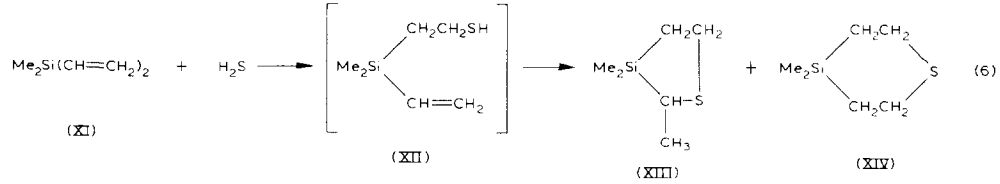
Analogously, H_2S adds to dimethyl(chloromethyl)allylsilane (V) to form 3,3-dimethyl-1-thia-3-silacyclohexane (VIII).



(V) (X) (VIII) (5)

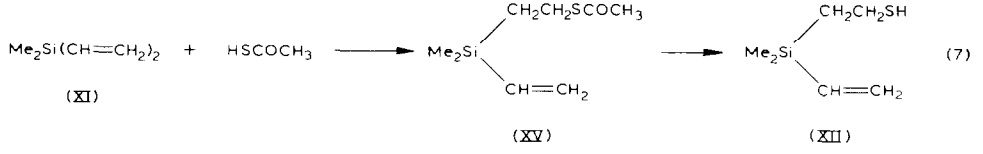
Compound V reacts with H_2S more slowly than compound I does, i.e. after 5 hours the conversions of the starting compounds (I and V) are 50–60 and 30–35%, respectively.

The intermediate formation of dimethyl(chloromethyl)mercaptoalkylsilanes IX and X by the above two reactions (eqs. 4 and 5) is supported by the absence of photochemical interaction of hydrogen sulfide with the chloromethyl group of Me_3SiCH_2Cl. Reactions 4 and 5 are the first examples of intramolecular interaction of $-SH$ and $-SiCH_2Cl$ groups which occur in the absence of HCl acceptors. In contrast, the cyclization of α,ω-chloroalkanethiols requires a basic medium [12]. Thus, the photochemical reactions of gaseous H_2S with dialkyl-chloromethylalkenylsilanes offer a convenient synthetic route to 3,3-dialkyl-1-thia-3-silacycloalkanes. Photochemical addition of liquid H_2S to dimethyldiallylsilane yields 5,5-dimethyl-1-thia-5-silacyclooctane [13]. We found that the photoinitiated reaction of gaseous H_2S with dimethyldivinylsilane (XI) leads to a mixture of 2,3,3-trimethyl-1-thia-3-silacyclopentane (XIII) and 4,4-dimethyl-1-thia-4-silacyclohexane (XIV) in a 1 : 2 ratio. The latter compound is the first example of derivatives of 1,4-thiasilacyclohexane.



(XI) (XII) (XIII) (XIV) (6)

The intermediate of the above reaction is dimethylvinyl(2-mercaptoethyl)silane (XII), the intramolecular cyclization of which takes place in two directions to give a mixture of XIII and XIV. Thiol XII has been prepared independently by photo-initiated addition of thiolacetic acid to dimethyldivinylsilane *, followed by hydrolysis of the thioacetate formed.



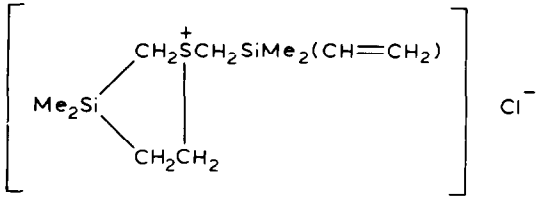
(XI) (XV) (XII) (7)

Photolysis of XII gave the same compounds XIII and XIV. Similarly, pentene-4-thiol-1 undergoes ring closure to give a mixture of five- and six-membered thia-

* Apart from the desired 2-dimethylvinylsilylethyl thioacetate (XV), an isomeric 1-dimethylvinylsilylethyl thioacetate (XVI) is formed. The XV : XVI ratio is 6 : 1.

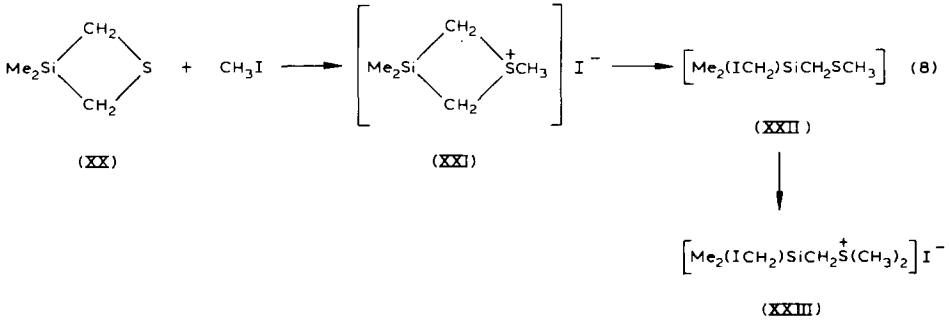
cycloalkanes with the six-membered ring predominant [6a,7a,14].

The mode of the reaction of thiasilacycloalkanes with electrophilic reagents depends on the ring size. The thiasilacyclopentane and thiasilacyclohexane derivatives (IV and XIV) form complexes with $HgCl_2$ in an alcoholic solution. In contrast, 3,3-dimethyl-1-thia-3-silacyclobutane undergoes cleavage at the endocyclic Si–C bond under similar conditions [2]. Heterocyclic compounds (IV, VII, VIII and XIV) are readily converted to the corresponding sulfonium salts when treated with organic and organosilicon halide derivatives. These salts are formed even on storage of the crude heterocyclic compounds containing traces of the starting chloromethylsilane. We have, for example, isolated a sulfonium salt of the following structure



(XVII)

The reaction of IV and XIV with CH_3I affords the corresponding sulfonium salts, $[Me_2Si(CH_2)_n(CH_2CH_2)S(CH_3)]^+I^-$ (XVIII, $n = 1$, XIX, $n = 2$). At the same time, treatment of 3,3-dimethyl-1-thia-3-silacyclobutane (XX) with CH_3I leads to sulfonium dimethyl(iodomethyl)silylmethiodide.



(XX) (XXI) (XXII) (8)

(XXIII)

In this case, like that of thietane [15], an intramolecular rearrangement of the initially formed methiodide XXI leading to compound XXII seems to occur. With CH_3I the latter forms XXIII.

Experimental

In most cases reactions were carried out under a nitrogen atmosphere. The light source used in all the photolyses was a DRT-400 mercury lamp. Analytical gas liquid chromatography (GLC) was carried out on a PACHV LCHM-8 MD instrument with a catharometer having a stainless steel 2 m × 3 mm column packed with 10% Lukopren G-1000 on 45–60 mesh Chromaton N-AW. A PACHV-07 gas chromatograph was used for GLC separation (5.0 m × 10 mm column packed with 10% Polyethyleneglycol-20 M on Chromaton AW-HMDS). The reaction mixtures were quantified using peak areas.

NMR spectra were recorded on a Tesla BS 487 B spectrometer (80 MHz). 10% solutions of the compounds in CCl_4 were used. Chemical shifts are given in ppm downfield from internal tetramethylsilane.

Dimethyl(mercaptomethyl)vinylsilane (II)

a) 4.12 g (0.03 mol) of dimethyl(chloromethyl)vinylsilane (I) was added dropwise to a potassium hydrosulfide solution (prepared by saturation with H_2S of 100 ml dry ethanol containing 3.42 g (0.06 mol) of potassium hydroxide). The mixture was stirred and refluxed for 0.5–1 h, then water was added and the product was extracted with pentane. The pentane solution was dried over $MgSO_4$.

b) A solution of 5.30 g (0.039 mol) of dimethyl(chloromethyl)vinylsilane (I) and 2.83 g (0.37 mol) of thiourea in 20 ml of ethanol was refluxed for 2 hours. On cooling there was obtained 7.80 g of the isothiuronium chloride as white crystals. 7.50 g of the salt was hydrolyzed with 20% aqueous KOH for 1.5 h at room temperature. After the organic layer was separated, the aqueous phase was acidified with dilute HCl and extracted with pentane. The combined organic phases were dried over $MgSO_4$. The identity of the above pentane solutions was determined by GLC. GLC analysis also shows the absence of the initial compound I and the presence of a volatile component. In both cases the solvent was removed under vacuum (140 mmHg) to give a residue the NMR spectra of which show the expected SH (t, 0.94 ppm) and vinyl (m, 5.94 ppm) groups. However, compound II could not be isolated by either distillation or preparative GLC. Heating of crude thiol II leads to polymerization. A pentane solution of thiol II was prepared by distillation of the reaction mixtures under vacuum (5 mmHg) without heating.

3,3,5-Trimethyl-1-thia-3-silacyclopentane (VII)

5.00 g (0.034 mol) of dimethyl(chloromethyl)allylsilane (V) was added to a solution of KSH obtained from 3.78 g (0.068 mol) of KOH in 100 ml EtOH. After the addition was completed the reaction mixture was stirred for 3 h at room temperature and worked-up as described above. There was obtained 3.00 g of VII (60% yield), b.p. 65–67°C (23 mm Hg), n_D^{20} 1.4855. Anal. Found: C, 50.87; H, 10.24; Si, 19.79; S, 20.10. $C_6H_{14}SiS$ calcd.: C, 49.31; H, 9.58; Si, 19.18; S, 21.81%. ^1H NMR (δ, ppm): 0.15 (s, CH_3Si); 0.24 (s, CH_3Si); 0.65 (d,d, SiCHC, $^2J_{(HH)} = 15$ Hz, $^3J_{(HH)} = 9$ Hz); 1.22 (d,d, SiCHC, $^2J_{(HH)} = 15$ Hz, $^3J_{(HH)} = 5$ Hz); 1.29 (d, CCH_3, $^3J_{(HH)} = 6$ Hz); 1.78 (s, $SiCH_2S$); 3.23 (m, CH). The presence of the chiral ring carbon atom causes inequivalence of chemical shifts of the methyl groups at the silicon and α-CH_2-group protons.

Dimethyl(mercaptomethyl)allylsilane (VIa) and 3,3-dimethyl-1-thia-3-silacyclohexane (VIII)

A mixture of 5.00 g (0.034 mol) of dimethyl(chloromethyl)allylsilane (V) and 2.82 g (0.037 mol) of thiourea in 10 ml of dry ethanol was refluxed for 3 h. After cooling and removal of the solvent there was obtained a crystalline isothiuronium salt which was hydrolyzed with 20 ml of 20% aqueous solution of KOH. The reaction mixture was stirred for 1.5 h, then the organic layer was separated, the aqueous phase was neutralized with dilute HCl and extracted with pentane. The combined organic phases were dried over $MgSO_4$. After removal of solvent GLC of the residue showed two peaks in a ratio of 1 : 1.5, one of which disappeared slowly

on standing *. Vacuum distillation of the residue yielded 2.50 g of a fraction with b.p. 40–41°C (5 mmHg), which contains dimethyl(mercaptomethyl)allylsilane (VI) and 3,3-dimethyl-1-thia-3-silacyclohexane (VIII) in a ratio of 1 : 1, identified by NMR. VI, ^{1}H NMR spectrum (δ, ppm): 0.08 (s, CH_3Si); 0.63 (m, $SiCH_2C$); 0.98 (d, SH); 1.59 (d, CH_2S and d, $SiCH_2$); 4.76 (m, $C{=}CH_2$); 5.96 (m, $CH{=}$).

The structure of VIII was confirmed by comparison of spectral data and GLC retention time with those of an authentic sample prepared as described below.

2.90 g (0.013 mol) of dimethylchloromethyl(3-bromopropyl)silane was added to 100 ml ethanolic solution of KSH obtained from 4.0 g (0.07 mol) KOH. After stirring at room temperature for 24 h, the reaction mixture was worked-up in the usual manner. Vacuum distillation gave 1.69 g of crude VIII (purity 70%, yield 62%), b.p. 60–70°C (14 mmHg). Compound VIII isolated by preparative GLC had n_D^{25} 1.4972 (n_D^{25} 1.4978 [16]). Anal. Found: C, 50.08; H, 9.77; Si, 18.49; S, 22.08. $C_6H_{14}SiS$ calcd.: C, 49.31; H, 9.58; Si, 19.18; S, 21.91%. ^{1}H NMR spectrum (δ, ppm): 0.14 (s, CH_3Si); 0.69 (m, CH_2Si); 1.63 (s, $SiCH_2S$); 2.04 (m, CCH_2C); 2.39 (m, CCH_2S).

3,3-Dimethyl-1-thia-3-silacyclopentane (IV)

10.00 g (0.09 mol) of dimethyl(chloromethyl)vinylsilane (I) was placed in a quartz flask equipped with a dry ice-acetone reflux condenser and a gas inlet tube. H_2S was bubbled through I while it was irradiated with a mercury lamp for 9 h at 70–80°C. 7.20 g of a fraction was obtained (b.p. 94–95°C (100 mmHg) containing 71% of IV (60% yield). Compound IV was further purified by GLC and had n_D^{20} 1.4978. Anal. Found: C, 45.46; H, 9.01; Si, 21.30; S, 23.45. $C_5H_{12}SiS$ calcd.: C, 45.40; H, 9.09; Si, 21.20; S, 24.21%. ^{1}H NMR spectrum (δ, ppm): 0.18 (s, CH_3Si); 0.94 (t, CH_2S); 2.19 (s, $SiCH_2S$), 2.69 (t, SCH_2C). $C_5H_{12}SiS \cdot HgCl_2$, m.p. 129–130°C. Anal. Found: C, 14.90; H, 3.31; Si, 7.08; S, 7.11; Cl, 17.01; Hg, 49.31. $C_5H_{12}SiSCl_2Hg$ calcd.: C, 14.89; H, 2.98; Si, 6.94; Cl, 17.60; Hg, 49.61%. Methiodide XVIII, m.p. 159–160°C. Anal. Found: S, 12.22; I, 44.82. $C_6H_{15}SiSI$ calcd.: S, 11.69; I. 46.27%. Chloride XVII, m.p. 37–38.5°C. Anal. Found: S, 11.45; Cl, 14.55. $C_{10}H_{23}Si_2SCl$ calcd.: S, 12.00; Cl, 13.32%.

3,3-Dimethyl-1-thia-3-silacyclohexane (VIII)

H_2S was bubbled through 5.00 g (0.034 mol) of dimethyl(chloromethyl)allylsilane (V) under simultaneous illumination with a mercury lamp. Photolysis was continued for 9 h. From time-controlled GLC analysis the reaction mixture contains, besides the starting compound V, heterocycle VIII and a third main component, the proportion of which increases with time. After 3 and 9 h the ratio of the above two components is 4 : 1 and 8 : 1, respectively. After distillation, however, the fraction obtained (2.67 g, b.p. 99–100°C (20 mmHg) contains 80% of VIII (43% yield) and only 2% of the third component. It is reasonable to assume that the latter is the thermally unstable thiol X. An analytical sample of VIII was obtained by GLC and its physical and spectral properties were in agreement with those described above.

* After standing for one month GLC showed only one peak, the fractional distillation of which gave 0.9 g of VIII (98% purity), b.p. 60–63°C (14 mmHg), n_D^{20} 1.4956.

Addition of H_2S to dimethyldivinylsilane (XI) *

H_2S was bubbled through 3.00 g (0.028 mol) of XI for 1.5 h under irradiation. The reaction mixture contains dimethylvinyl(2-mercaptoethyl)silane (XII), 2,3,3-trimethyl-1-thia-3-silacyclopentane (XIII) and 4,4-dimethyl-1-thia-4-silacyclohexane (XIV) in a ratio of 1 : 9 : 17. Vacuum distillation gave 1.50 g of a fraction, b.p. 60–65°C (20 mmHg), containing 3% of XII (yield 1%), 34% of XIII (13% yield) and 63% of XIV (25% yield). Pure compounds XIII and XIV were isolated by GLC and had the following characteristics. XIII, n_D^{20} 1.4950. Anal. Found: C, 50.06; H, 9.10; Si, 19.03; S, 22.54. $C_6H_{14}SiS$ calcd.: C, 49.31; H, 9.58; Si, 19.18; S, 21.91%. ^1H NMR spectrum (δ, ppm): 0.11 (s, CH_3Si); 0.99 (t, CCH_2Si); 1.22 (d, CH_3); 2.06 (q, CH); 2.71 (m, CCH_2S). XIV, n_D^{20} 1.4981. Anal. Found: C, 50.06; H, 9.47; Si, 20.23; S, 22.10. $C_6H_{14}SiS$ calcd.: C, 49.31; H, 9.58; Si, 19.18; S, 22.10%. ^1H NMR spectrum (δ, ppm): 0.05 (s, CH_3Si); 0.90 (m, CCH_2Si); 2.71 (m, CCH_2S). $C_6H_{14}SiS \cdot HgCl_2$, m.p. 163–164°C. Anal. Found: C, 17.34; H, 3.76; Si, 6.65; S, 7.42; Cl, 17.10; Hg, 48.65. $C_6H_{14}SiSCl_2Hg$ calcd.: C, 17.25; H, 3.36; Si, 6.71; S, 7.67; Cl, 17.00; Hg, 48.00%. Methiodide XIX, m.p. 178–180°C. Anal. Found: S, 12.02; I, 43.47. $C_7H_{17}SiSI$ calcd.: S, 11.15; I, 44.09%.

The retention time of compound XII is identical with that of an authentic sample prepared as described below.

Addition of thiolacetic acid to dimethyldivinylsilane (XI)

3.40 g (0.045 mol) of thiolacetic acid was added dropwise to 5.00 g (0.045 mol) of XI. The reaction mixture was irradiated for 1.5 h. Distillation afforded 3.27 g of a mixture of 2-(dimethylvinylsilyl)ethyl thioacetate (XV) and 1-(dimethylvinylsilyl)ethyl thioacetate (XVI) in a ratio of 6 : 1. Anal. Found: C, 50.92; H, 8.46; Si, 14.68; S, 17.06. $C_8H_{16}SiSO$ calcd.: C, 51.06; H, 8.51; Si, 14.89; S, 17.02%. XV, ^1H NMR spectrum (δ, ppm): 0.06 (s, $SiCH_3$); 0.86 (m, $SiCH_2C$); 2.25 (s, CH_3CO); 2.83 (m, CCH_2S); 5.96 (m, $CH{=}CH_2$). IR: 1590 (C=C); 1695 (CO) cm^{-1}. The ^1H NMR spectrum displays a resonance signal of the CH_3CH-group of compound XVI. The CH-proton resonance signal is overlapped by a multiplet arising from the CH_2S group of compound XV. This is confirmed by homonuclear INDOR. The XV : XVI ratio is 6 : 1. 2.50 g (0.013 mol) of the above mixture of XV and XVI was hydrolyzed by refluxing with 20% ethanolic KOH for 1 h. The cooled solution was neutralized with dilute acetic acid. After the usual work-up the organic phase contained dimethylvinyl(2-mercaptoethyl)silane (XII) and dimethylvinyl(1-mercaptoethyl)silane (XIIa). in a 10 : 1 ratio. XII, ^1H NMR spectrum (δ, ppm): 0.06 (s, CH_3Si); 0.95 (m, $SiCH_2C$); 1.31 (t, SH); 2.59 (m, CH_2S); 5.85 (m, $CH{=}CH_2$). The compound XIIa could not be identified by ^1H NMR spectroscopy due to its low concentration in the reaction mixture and overlapping in the spectrum.

Sulfonium dimethyl(iodomethyl)silylmethyliodide (XXIII)

A mixture of 0.3 g (0.0025 mol) of 3,3-dimethyl-1-thia-3-silacyclobutane and 0.36 g (0.0025 mol) of CH_3I was allowed to stand for 15 h at room temperature. The precipitate formed was washed with dry ether and dried under vacuum. 0.4 g (40% yield) of XXIII was obtained as white crystals, m.p. 100–101°C. Anal. Found: I,

* The reaction is shown to be catalyzed by YCl_3 [17].

65.64. $C_6H_{16}SiSI_2$ calcd.: I, 63.18%. ^1H NMR spectrum (δ, ppm): 0.40 (s, CH_3Si); 2.25 (s, CH_2I); 2.79 (s, CH_2S); 2.91 (s, SCH_3).

Acknowledgement

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