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SYNTHESIS OF CYCLIC POLYSULFIDES BY THE REACTION OF TRIMETHYLVINYLSILANE WITH S_8 IN THE PRESENCE OF $Fe_3(CO)_{12}$

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Summary

Reaction of trimethylvinylsilane with S_8 in the presence of $Fe_3(CO)_{12}$ yielded the following compounds: $S_2Fe_2(CO)_6$, $S_2Fe_3(CO)_9$, 1-trimethylsilyl-2,3,4,5,6-pentathiacycloheptane, 1,4-di(trimethylsilyl)-2,3,6-trithiacycloheptane, 1,4-di(trimethylsilyl)-2,3,6,7-tetrathiacyclooctane. An X-ray analysis was performed for the two latter polysulfides.

Introduction

The reactions of S_8 with olefins relate directly to rubber vulcanization and have been studied in detail. Their main reaction products are the longitudinally- and cross-linked polysulfides [1,2]. The reaction kinetics of olefins with sulfur has been studied generally for cyclohexene. Reaction of cyclohexene with S_8 starts only at 150°C and leads to a mixture of products: dicyclohexylsulfide, dicyclohexyldisulfide and saturated polymeric polysulfides [3]. Until this work, cyclic polysulfides could be isolated only with the use of cyclic olefins. Selective and stereospecific sulfuration of the olefins with S_8 in the presence of ammonia (110°C, DMF) giving rise to *exo*-3,4,5-trithiatricyclo[5,2,1,$0^{2,6}$]decanes [4] was the first example of a sulfuration of unsaturated hydrocarbons to trithia-hydrocarbons. The formation of cyclic polysulfides from elemental sulfur and cycloheptotriene-1,3,5 [5], norbornene and cyclohexene [6] and dicyclopentadiene [7] has also been described. In the most cases S_8 was activated with various nitrous bases or UV irradiation. Thermal reaction has been performed (140°C) only for dicyclopentadiene.

Unusual behaviour of a S_8-$Fe_3(CO)_{12}$ system in the presence of unsaturated

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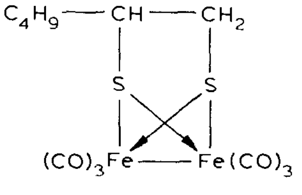
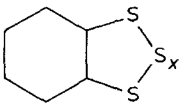
compounds has been revealed for hexene-1 [8] and cyclohexene [9]. The authors failed to obtain cyclic polysulfides from hexene-1 although they were generated in the course of this reaction: the main product was the respective dithiolane diironhexacarbonyl complex (I). Under the conditions described its formation could be due only to the reaction of di- or poly-sulfides with $Fe_3(CO)_{12}$ [8]. This has been established by special reactions of cyclic polysulfides using cyclohexene (II) and $Fe_3(CO)_{12}$ [10].



(I)



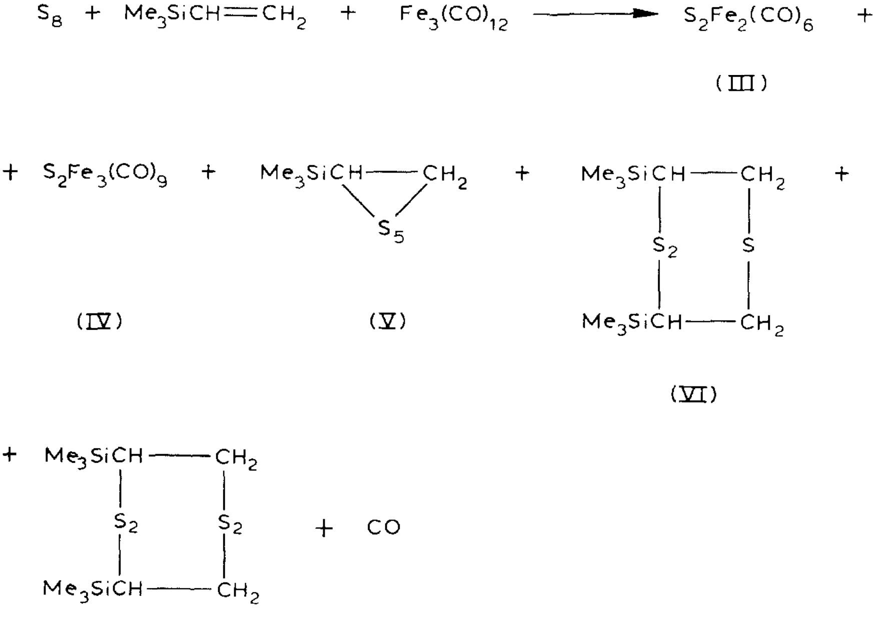
(x = 4.5)

(II)

Here we describe the synthesis of cyclic and cross-linked polysulfides from trimethylvinylsilane and S_8 in the presence of $Fe_3(CO)_{12}$.

Results and discussion

We found that elemental sulfur reacts with excess trimethylvinylsilane under mild conditions (55°C) in the presence of $Fe_3(CO)_{12}$ giving the following compounds: dithio-diiron hexacarbonyl (III), dithiotriiron nonacarbonyl (IV), 1-trimethylsilyl-2,3,4,5,6-pentathiacycloheptane (V), 1,4-di(trimethylsilyl)-2,3,6-trithiacycloheptane (VI) and 1,4-di(trimethylsilyl)-2,3,6,7-tetrathiacyclooctane (VII).



(III) (IV) (V) (VI) (VII)

The yields relative to initial sulfur were: III + IV, 5–10%, V, 3-3.7%; VI, VII, 1%. Besides these compounds the reaction gave negligible quantities of the dithiolanediiron hexacarbonyl complex 1-trimethylsilyl-μ-S,S'-ethylene dithiolato hexacarbonyldiiron and 1-methyl-3-trimethylsilyl-2,5-dithiacyclopentane. Physical-chemical constants and structure of these compounds will be described in our next communication. As one might expect, the elemental sulfur in the absence of $Fe_3(CO)_{12}$ does not react with trimethylvinylsilane under conditions described. It should be pointed out that the reaction occurs without evolution of H_2S. Synthesis of compound V is the first and only example of generation of a cyclic polysulfide from an acyclic olefin. We failed to isolate any products from sulfur-addition at the double bond from the carbon analog of trimethylvinylsilane (tert-butylethylene) and 1-trimethylsilyl-2-chloroethene under these and even more drastic conditions.

The compounds V–VII are white crystalline substances, well-soluble in hydrocarbon solvents, and with limited solubility in the polar solvents. They are stable as solids as well as in solution at 0°C. Polysulfide V releases its sulfur at room temperature with time transforming into a polymeric polysulfide.

The structure of polysulfides V-VII was established on the basis of mass, IR, UV and PMR spectra. For VII a linking of two trimethylvinylsilane molecules via one or three sulfur atoms is not excluded: its UV spectrum exhibits a wing-pattern with an inflection point at 250 nm and extending to 310 nm. To establish the structure of the polysulfides we performed an X-ray study of compounds V and VI.

For V: monoclinic crystals, $a = 9.757(2)$, $b = 10.122(3)$, $c = 12.296(3)$ Å, $\beta = 78.91(2)°$, $d_{calc.} = 1.45$ g/cm^3, $Z = 4$, space group $P2_1/c$. The structure was decoded by direct methods and iterated by the least squares method using 840 reflections to $R = 0.030$ ($R_\omega = 0.047$), atoms Si, S, C were iterated anisotropically while H atoms

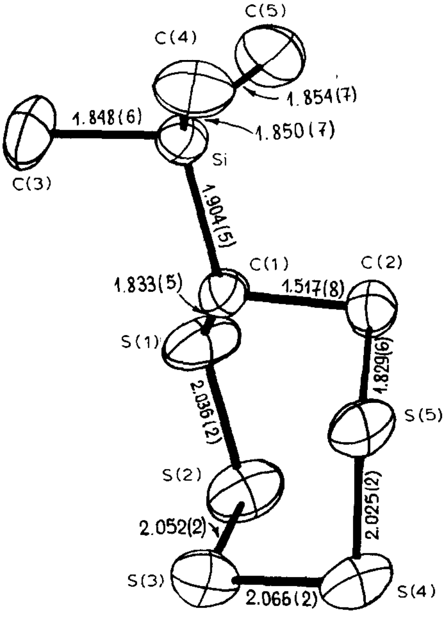


Fig. 1. Structure of compound V.

recovered from a difference series were found isotropically. In the structure of V (Fig. 1) the bond Si–C(1) between the trimethylsilyl substituent and the sevenmembered cycle is elongated to 1.904(5) A° with respect to the Si–C bonds in the substituent itself (1.851(7) Å, aver.) having a common length [11]. The bond angles

TABLE 1

ATOMIC COORDINATES ($\times 10^4$) AND PARAMETERS OF ANISOTROPIC TEMPERATURE FACTORS IN THE FORM $T = \exp[-1/4\,(B_{11}h^2a^{*2} + \ldots + 2\,B_{23}b^*c^*kl)]$ FOR COMPOUND V

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Si	3146(2)	2049(2)	8235(1)	2.70(7)	3.47(7)	3.42(7)	0.31(6)	−0.39(5)	0.49(6)
C(1)	1164(5)	1961(5)	8454(4)	2.9(2)	2.9(2)	3.0(2)	0.2(2)	−0.4(2)	0.2(2)
C(2)	678(6)	686(6)	8002(5)	3.6(3)	4.1(3)	5.3(3)	−0.4(2)	0.0(2)	−1.5(2)
C(3)	3626(7)	3706(6)	8670(5)	4.4(3)	4.2(3)	6.6(4)	−0.8(3)	2.3(3)	0.5(3)
C(4)	3831(7)	1823(7)	6738(5)	5.7(4)	6.5(4)	4.2(3)	0.8(3)	0.8(3)	0.7(3)
C(5)	3801(7)	724(6)	9040(5)	4.6(3)	4.8(3)	6.3(4)	1.1(3)	−1.1(3)	1.7(3)
S(1)	390(2)	2245(2)	9914(1)	3.62(7)	4.62(7)	2.88(6)	0.65(7)	0.40(5)	0.14(6)
S(2)	−1006(2)	3721(2)	9840(1)	3.69(7)	3.44(7)	4.91(7)	0.47(6)	−0.45(6)	−0.91(6)
S(3)	−2754(2)	2831(2)	9483(2)	2.91(6)	4.91(8)	6.18(9)	0.24(7)	0.04(6)	−0.47(8)
S(4)	−2100(2)	2096(2)	7902(1)	4.12(8)	5.39(9)	5.40(8)	−0.18(7)	−1.69(6)	−0.29(7)
S(5)	−1193(2)	357(2)	8168(2)	4.01(8)	3.59(7)	7.24(10)	−0.94(7)	−0.72(7)	−1.26(7)

TABLE 2

BOND LENGTHS IN COMPOUND V

Bond	d (Å)	Bond	d (Å)
Si–C(1)	1.904(5)	S(1)–C(1)	1.833(5)
		S(5)–C(2)	1.829(6)
Si–C(3)	1.848(6)	Average	1.832(6)
Si–C(4)	1.850(7)		
Si–C(5)	1.854(7)	S(1)–S(2)	2.036(2)
Average	1.851(7)	S(2)–S(3)	2.052(2)
		S(3)–S(4)	2.066(2)
C(1)–C(2)	1.517(8)	S(4)–S(5)	2.025(2)
		Average	2.045(2)

TABLE 3

BOND ANGLES ω IN COMPOUND V

Angle	ω (°)	Angle	ω (°)
C(1)SiC(3)	108.0(3)	SiC(1)C(2)	111.7(4)
C(3)SiC(4)	109.7(3)		
C(4)SiC(1)	107.4(3)	C(1)S(1)S(2)	103.0(2)
C(1)SiC(5)	109.6(3)	C(2)S(5)S(4)	106.4(2)
C(3)SiC(5)	111.6(3)	Average	104.7(2)
C(4)SiC(5)	110.3(3)		
Average	109.4(3)	S(1)S(2)S(3)	106.2(1)
		S(2)S(3)S(4)	104.0(1)
S(1)C(1)C(2)	113.2(4)	S(3)S(4)S(5)	103.1(1)
S(5)C(2)C(1)	119.4(4)	Average	104.4(1)
Average	116.3(4)	SiC(1)S(1)	110.4(3)

TABLE 4

TORSIONAL ANGLES ω IN SEVEN-MEMBERED CYCLE OF COMPOUND V

Bond	ω(°)
C(1)-C(2)	52.4
C(1)-S(1)	73.1
C(2)-S(5)	39.9
S(1)-S(2)	98.5
S(2)-S(3)	66.1
S(3)-S(4)	83.0
S(4)-S(5)	95.9

C-Si-C of the C(1) atom in the heterocycle are somewhat smaller than other C-Si-C angles (mean values are 108.3 and 110.5°, respectively). The C-C and S-C bond lengths in the heterocycle have common values (average values are 1.517(8) and 1.832(6) Å, respectively) which practically coincide with the sum of C and S covalent radii [12]. Atomic coordinates and anisotropic temperature factors are listed

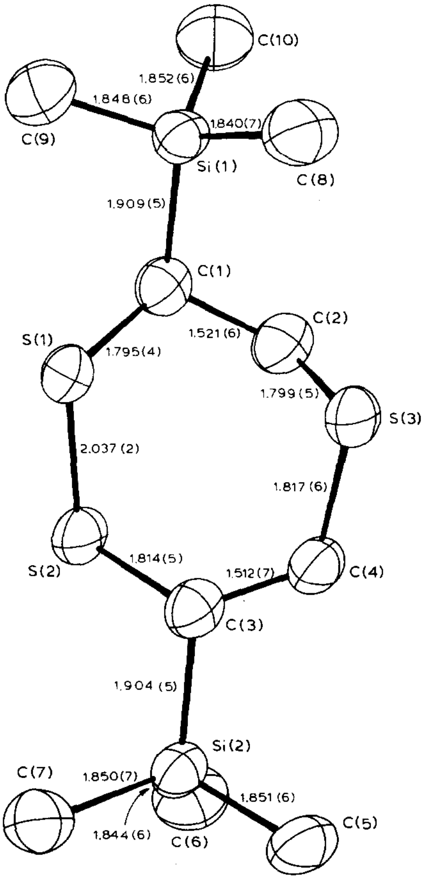


Fig. 2. Structure of compound VI.

in Table 1, bond lengths in Table 2 and bond angles in Table 3. The seven-membered heterocycle is highly distorted, its conformation described by torsional angles about the bonds is shown in Table 4. The bonds S(1)–S(2) and S(4)–S(5) are slightly shorter than the S(2)–S(3) and S(3)–S(4) bonds. The torsional angle most distorted from 90° (66.1°) refers to an elongated S(2)–S(3) bond. The heterocycle conformation may be described as a plane of C(2), S(5), S(2) atoms (with 0.05–0.07 Å atomic deviations) with S(3), S(4), S(5) inclined at 79°.

For VI: triclinic crystals, $a = 6.553(2)$, $b = 12.335(4)$, $c = 11.252(3)$ Å, $\alpha = 64.70(2)$, $\beta = 92.81(2)$, $\gamma = 94.47(2)°$, $Z = 2$, $d_{\text{calc.}} = 1.20$ g/cm^3, space group $P\bar{1}$. The structure was decoded by direct methods and iterated by the least squares method using 1956 reflections to $R = 0.045$ ($R_\omega = 0.050$), the Si, S, C atoms were iterated anisotropically, for H atoms recovered from a difference series only positional parameters were refined ($B = 5.0$ Å^2).

In the structure of VI (Fig. 2) the bond lengths Si(1)–C(1) and Si(2)–C(3) (1.906(5) Å, aver.) between trimethylsilyl substituents and the seven-membered heterocycle as in V are elongated with respect to the Si–C bonds in the substituents themselves (1.848(6) Å, aver.). Similar Si–C bond elongation with respect to trimethylsilyl groups has been found previously [13]. The bond angles, however, exhibit no such behaviour. The rigidity of the seven-membered heterocycle is responsible for the slight reduction in the exocyclic angles at the substituted C(1) and C(3) atoms, unlike the endocyclic angles. Atomic coordinates and anisotropic temperature factors for VI are listed in Table 5, bond lengths in Table 6, and bond angles in Table 7. The conformation of the seven-membered heterocycle is described by torsional angles shown in Table 8. Torsional angles about the bonds C(2)–S(3), S(3)–C(4) and S(1)–S(2) are least distorted from 90° with respect to other angles with substituted C(1) and C(3) atoms. The heterocycle conformation can be depicted most clearly as a plane of C(1), S(3), C(3), S(2) atoms with C(2) and C(4) deviating towards the

TABLE 5

NONHYDROGEN ATOMIC COORDINATES ($\times 10^4$) AND B_{ij} PARAMETERS OF ANISOTROPIC TEMPERATURE FACTORS IN THE FORM OF $T = \exp[-1/4(B_{11}h^2a^{\star 2} + \ldots + 2B_{23}b^\star c^\star kl)]$ FOR COMPOUND VI

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Si(1)	2223(2)	−2796(1)	5409(1)	4.07(5)	3.41(5)	3.26(5)	0.29(4)	0.31(4)	−1.34(4)
Si(2)	868(2)	2829(1)	−418(1)	4.04(4)	3.25(5)	3.34(5)	−0.02(4)	0.50(4)	−1.45(4)
S(1)	−155(2)	−690(1)	3547(1)	3.17(4)	3.76(5)	4.20(5)	0.06(4)	0.74(4)	−0.98(4)
S(2)	243(2)	1104(1)	2392(1)	5.29(6)	3.40(5)	3.90(5)	0.70(4)	1.59(4)	−1.28(4)
S(3)	4103(2)	−591(1)	2042(1)	5.64(6)	4.32(6)	4.51(6)	1.33(5)	1.82(5)	−1.40(5)
C(1)	2218(7)	−1096(4)	4443(4)	4.0(2)	3.3(2)	3.5(2)	0.1(1)	0.5(2)	−1.9(2)
C(2)	4120(7)	−550(4)	3621(5)	4.0(2)	4.2(2)	4.2(2)	−0.2(2)	0.5(2)	−1.4(2)
C(3)	1237(7)	1234(4)	857(4)	4.2(2)	3.4(2)	3.5(2)	−0.1(1)	0.7(2)	−1.7(2)
C(4)	3444(8)	922(4)	950(5)	5.4(2)	3.5(2)	3.9(2)	0.4(2)	1.9(2)	−1.0(2)
C(5)	1989(9)	3016(5)	−1978(5)	6.7(3)	4.6(2)	3.6(2)	0.5(2)	1.0(2)	−1.3(2)
C(6)	−1889(8)	3080(5)	−636(6)	5.1(3)	4.8(3)	4.7(3)	0.3(2)	0.1(2)	−1.7(2)
C(7)	2221(9)	3895(5)	154(6)	6.0(3)	3.6(2)	5.3(3)	−0.5(2)	0.3(2)	−1.9(2)
C(8)	3218(10)	−3499(5)	4425(6)	7.8(4)	4.4(3)	4.9(3)	1.3(3)	1.0(3)	−1.9(2)
C(9)	−394(8)	−3422(5)	5967(6)	5.5(3)	4.4(3)	6.2(3)	−0.4(2)	1.1(2)	−1.5(2)
C(10)	3923(9)	−3058(6)	6858(6)	5.9(3)	6.5(3)	4.7(3)	1.0(3)	−0.4(2)	−1.9(3)

TABLE 6

BOND LENGTHS IN COMPOUND VI

Bond	d (Å)	Bond	d (Å)
Si(1)–C(1)	1.909(5)	S(1)–S(2)	2.037(2)
Si(2)–C(3)	1.904(5)		
Average	1.906(5)	S(1)–C(1)	1.795(4)
		S(2)–C(3)	1.814(5)
Si(1)–C(8)	1.840(7)	S(3)–C(2)	1.799(5)
Si(1)–C(9)	1.848(6)	S(3)–C(4)	1.817(6)
Si(1)–C(10)	1.852(6)	Average	1.806(5)
Si(2)–C(5)	1.851(6)		
Si(2)–C(6)	1.844(6)	C(1)–C(2)	1.521(6)
Si(2)–C(7)	1.850(7)	C(3)–C(4)	1.512(7)
Average	1.848(6)	Average	1.516(7)

TABLE 7

BOND ANGLES ω IN COMPOUND VI

Angle	ω(°)	Angle	ω(°)
C(8)Si(1)C(9)	110.6(3)	Si(1)C(1)C(2)	113.1(3)
C(9)Si(1)C(10)	109.4(3)	Si(2)C(3)C(4)	111.6(3)
C(8)Si(1)C(10)	109.7(3)	Average	112.4(3)
C(5)Si(2)C(6)	110.8(3)		
C(6)Si(2)C(7)	110.1(3)	C(1)S(1)S(2)	105.1(2)
C(5)Si(2)C(7)	109.4(3)	C(3)S(2)S(1)	105.6(2)
Average	110.0(3)	Average	105.4(2)
C(1)Si(1)C(8)	111.5(3)		
C(1)Si(1)C(9)	109.7(3)	C(2)S(3)C(4)	101.7(2)
C(1)Si(1)C(10)	105.8(3)		
C(3)Si(2)C(5)	108.2(3)	S(1)C(1)C(2)	114.5(3)
C(3)Si(2)C(6)	109.2(3)	S(2)C(3)C(4)	113.9(3)
C(3)Si(2)C(7)	109.1(2)	S(3)C(2)C(1)	116.4(4)
Average	108.9(3)	S(3)C(4)C(3)	118.7(4)
		Average	115.9(4)
Si(1)C(1)S(1)	110.3(2)		
Si(2)C(3)S(2)	107.0(2)		
Average	108.6(2)		

TABLE 8

TORSIONAL ANGLES ABOUT THE BONDS IN THE SEVEN-MEMBERED HETEROCYCLE IN COMPOUND VI

Bond	ω(°)
C(1)–C(2)	135.6
C(2)–S(3)	99.8
S(3)–C(4)	102.4
C(4)–C(3)	117.8
C(3)–S(2)	106.8
S(1)–S(2)	90.7
S(1)–C(1)	43.6

same side and atom S(1) on the other side of the plane (by −0.83, −0.60 and 1.11 Å, respectively).

Experimental

The IR spectra were recorded on UR-20 spectrophotometer in KBr pellets. The mass spectra were recorded on an LKB 2091 instrument with a direct inlet to the ion source at 20 and 50°C and ionization energy of 70 eV. The PMR spectra were measured on a Varian T-60 spectrometer (60 MHz) with C_6H_6 as solvent and internal standard. The UV spectra were registered on a Specord UV-Vis in hexane. X-Ray analysis was performed on Nicolet R-3m automatic diffractometer (λ Mo, graphite monochromator, $2\,\theta \leqslant 48°$).

Reaction of trimethylvinylsilane with S_8 in the presence of $Fe_3(CO)_{12}$

A mixture of 19.2 g (0.075 mol) of S_8, 50 g (0.099 mol) of $Fe_3(CO)_{12}$ and 50 ml (0.34 mol) of silane in 200 ml of benzene was stirred at 55°C for 8 hours. The silane and benzene excess was removed in vacuum, the residue extracted with pentane in Soxhlet. After removal of pentane the residue was chromatographed on a silicagel column (hexane eluent). Isolated: first fraction (III), m.p. 45°C, second fraction (VI), m.p. 115°C (cf. [14]). Compounds V and VII were isolated as a mixture with the dithiolanediiron hexacarbonyl complex. They were separated by multiple recrystallization from ethanol. Polysulfide VI was eluted from the column with benzene.

For V: m.p. 58–59°C (ethanol). Mass spectrum (m/e): 260 (M^+), 228 ($M^+ - S$), 196 ($M^+ - 2S$), 164 ($M^+ - 3S$), 132 ($M^+ - 4S$), 117 ($M^+ - 4S - CH_3$), 90 ($[Me_2SiS])^+$, 73 ($[Me_3Si])^+$, 59 ($[C_2H_3S])^+$ IR spectrum (ν, cm^{-1}): 2952, 2930, 2894, 2857 (C–H), 840, 750 (Si–C), 668, 626 (C–S), 502, 494, 464, 425 (S–S). PMR spectrum (δ, ppm): 0.05 s ($3CH_3$), 2.87–3.69 m ($CH-CH_2$). UV spectrum (λ, nm, (log ε)): 233 (3.75), 281 (3.43). Found: C, 22.96; H, 4.60; Si, 10.82. $C_5H_{12}SiS_5$ Calcd.: C, 23.05; H, 4.64; Si, 10.78%.

For VI: m.p. 80°C (ethanol). Mass spectrum (m/e): 296 (M^+), 281 ($M^+ - CH_3$), 237 ($M^+ - C_2H_3S$), 205 ($M^+ - C_2H_3S_2$), 164 ($[Me_3SiC_2H_3S_2]^+$), 132 ($[Me_3SiC_2H_3S]^+$), 122 ($[Me_2SiS_2]^+$), 117 ($[Me_2SiC_2H_3S]^+$), 90 ($[Me_2SiS]^+$), 73 ($[Me_3Si]^+$), 59 ($[C_2H_3S]^+$). IR spectrum (ν, cm^{-1}): 2960, 2905, 2891, 2862 (C–H), 845, 755 (Si–C), 672, 631, 610 (C–S), 520 (S–S). PMR spectrum (δ, ppm): 0.10 s ($6CH_3$); 2.60 m (2CH); 3.10 m ($2CH_2$). UV spectrum (λ, nm, (log ε)): 223 (3.10), 250 (2.57). Found: C, 40.40; H, 8.22; Si, 19.30. $C_{10}H_{24}Si_2S_3$ calcd.: C, 40.49; H, 8.16; Si, 18.93%.

For VII: m.p. 137–138°C (ethanol). Mass spectrum (m/e): 328(M^+), 196 ($[Me_3SiC_2H_3S_3]^+$), 164 ($[Me_3SiC_2H_3S_2]^+$), 132 ($[Me_3SiC_2H_3S]^+$), 122 ($[Me_2SiS_2]^+$), 117 ($[Me_2SiC_2H_3S]^+$), 90 ($[Me_2SiS]^+$), 73 ($[Me_3Si]^+$), 59 ($[C_2H_3S]^+$). IR spectrum (ν, cm^{-1}): 2957, 2900, 2859 (C–H), 840, 748 (Si–C), 642, 610 (C–S), 432 (S–S). PMR spectrum (δ, ppm): 0.06 s ($6CH_3$), 2.54 m (2CH); 3.07 m ($2CH_2$), UV spectrum (λ, nm, (log ε)): 220 (3.11), 250 (2.78). Found: C, 36.68; H, 7.59; Si, 16.90. $C_{10}H_{24}Si_2S_4$ calcd.: C, 36.54; H, 7.36; Si, 17.09%.

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