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INFRARED SPECTROSCOPIC STUDY OF VERY LOW PRESSURE PYROLYSIS PRODUCTS OF CYCLOSILTHIANES AND 3,3-DIMETHYL-3-SILATHIETANE ISOLATED IN ARGON MATRICES. A NEW SOURCE OF 1,1-DIMETHYL-1-SILAETHYLENE ($Me_2Si{=}CH_2$), THIOFORMALDEHYDE ($H_2C{=}S$) AND DIMETHYLSILANTHIONE ($Me_2Si{=}S$)

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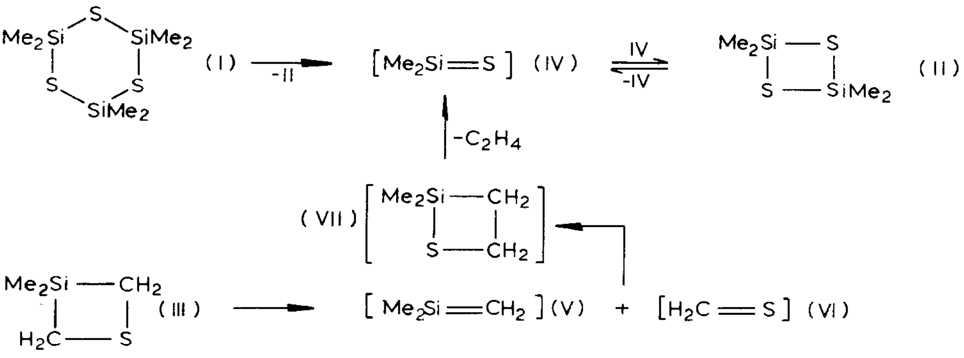
Summary

The products of very low pressure pyrolysis (VLPP) of hexamethylcyclotrisilthiane (I), tetramethylcyclodisilthiane (II) and 3,3-dimethyl-3-silathietane (III) were isolated in Ar matrices and were studied by IR spectroscopy. The only pyrolysis product of I was cyclosilthiane II, a dimer of transient dimethylsilanthione ($Me_2Si{=}S$) (IV). The starting material was recovered on pyrolysis of II. Thermal decomposition of III involves three intermediate unsaturated compounds: dimethylsilaethylene ($Me_2Si{=}CH_2$) (V) and thioformaldehyde ($H_2C{=}S$) (VI), both isolated in Ar matrix at 10 K, as well as silanthione IV fixed in the matrix in a form of the cyclic dimer II. The latter was also observed in the study of copyrolysis of 1,1-dimethyl-1-silacyclobutane and thietane, being authentic sources of intermediates V and VI. IR spectra of starting compounds I, II and III isolated in Ar matrices were obtained. The theoretical structure of IV and force constant F(Si=S) were determined by the CNDO/2 method. With regard to CNDO/2 errors, Si=S bond distance and F(Si=S) are equal to 1.993 Å and 4.72 mdyn/Å, respectively. Calculation of normal vibrations resulted in the following values of vibrational frequencies of dimethylsilanthione (cm^{-1}): 884 $\nu_s(SiC_2)$ (A_1), 735 $\nu_{as}(SiC_2)$ (B_2), 626 ν_s(Si=S) (A_1), 200 < CSiC (A_1).

Introduction

Hexamethylcyclotrisilthiane (I) is known to be transformed into tetramethylcyclodisilthiane (II) under liquid phase thermolysis (200°C) [1–3]. The latter compound is also formed in the pyrolysis of 3,3-dimethyl-3-silathietane (III) [4,5]. The formation of II in both cases is believed to result from cyclodimerization of dimethylsilanthione (IV) (an unstable intermediate with a silicon–sulphur double bond). Dimethylsilanthione is produced in the pyrolysis of III as a result of the consecutive reactions of [4 → 2 + 2]-cyclodecomposition and [2 + 2]-cycloaddition of dimethylsilaethylene (V) and thioformaldehyde (VI) via intermediate 2,2-dimethyl-2-silathietane (VII) [5].



SCHEME 1

In order to study the mechanism of thermal decomposition of I, II and III in more detail, we carried out an IR investigation of the starting compounds and their very low pressure pyrolysis products isolated in Ar matrices.

Experimental

Starting compounds were prepared according to the procedures described elsewhere: tetramethylcyclodisilthiane [6]; hexamethylcyclotrisilthiane [1]; 3,3-dimethyl-3-silathietane [7].

Pyrolysis and matrix isolation techniques were described in our previous publication [8].

IR spectra were recorded on a Unicam SP-1200 spectrophotometer; the IR spectrum of gaseous 3,3-dimethyl-3-silathietane was recorded on a Bruker IFS-113 spectrometer with a resolution 0.5 cm^{-1}.

Raman spectrum of liquid 3,3-dimethyl-3-silathietane was obtained on a Ramanor HG2-S spectrometer.

Results

1. Spectra of original compounds

A. Hexamethylcyclotrisilthiane and tetramethylcyclodisilthiane. In Figs. 1 and 2 are shown IR spectra of I and II isolated in Ar matrices at 10 K. IR spectra of crystalline samples of I and II prepared by condensation of the substances on a KBr cryostat target cooled to 77 K are given in Figs. 3 and 4. In Table 1 the frequencies thus measured are compared with IR spectra of the compounds in CS_2 [1]. Assignments of the observed bands to different vibrational modes are given for I according to ref. 1. Assignments for II are based on normal coordinate analysis. These are in

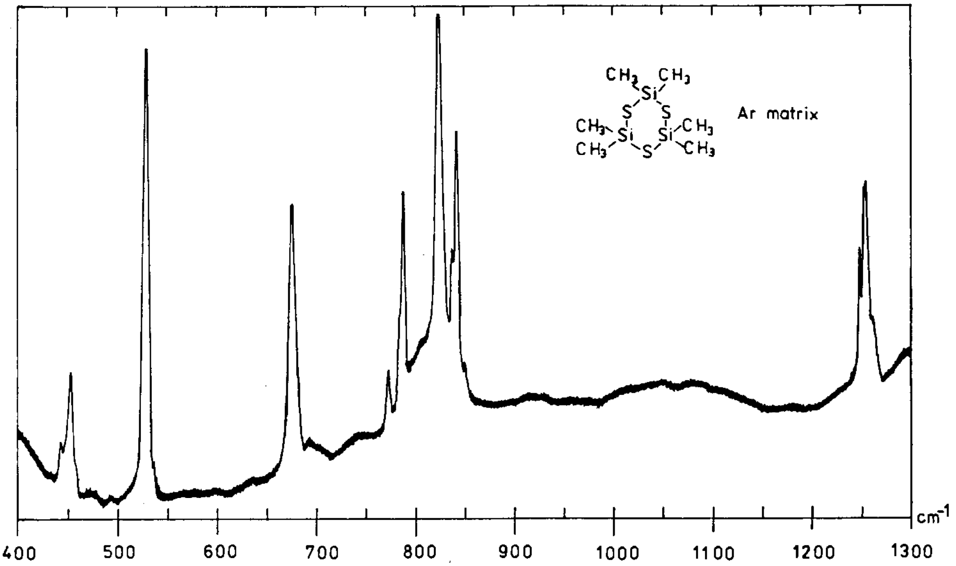


Fig. 1. Infrared spectrum of hexamethylcyclotrisilthiane in Ar matrix at 10 K.

agreement with the reported data [1]. X-ray structural data [9] were used for calculation of normal vibrations of molecules II.

Comparison of results of this work with the reported data [1] shows the following. In the spectra of I and II, the same bands are observed as before except for some weak bands and the bands in the 1000–1100 cm^{-1} region. The latter appear in the

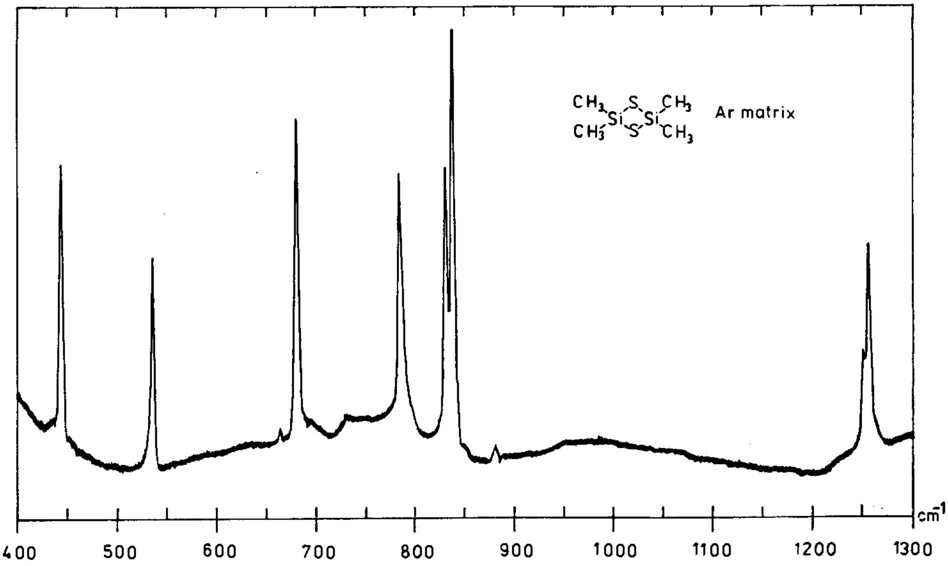


Fig. 2. Infrared spectrum of tetramethylcyclodisilthiane in Ar matrix at 10 K.

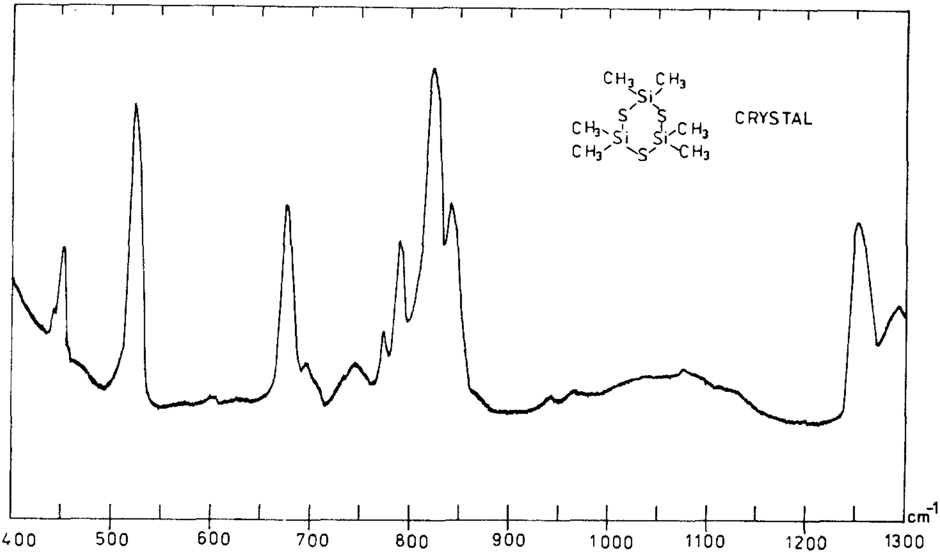


Fig. 3. Infrared spectrum of crystalline hexamethylcyclotrisilthiane at 77 K.

IR spectra of crystalline I and II. However, their relative intensities is less than reported [1] and depends on the way of preparation of sample. Therefore we assume that the bands in the region 1000–1100cm^{-1} do not belong to cyclosiltianes I and II but to the products of their partial hydrolysis.

The bands of crystalline I and II are wide and their identification in the mixture is difficult because of superposition. Due to the narrowness of the bands, small

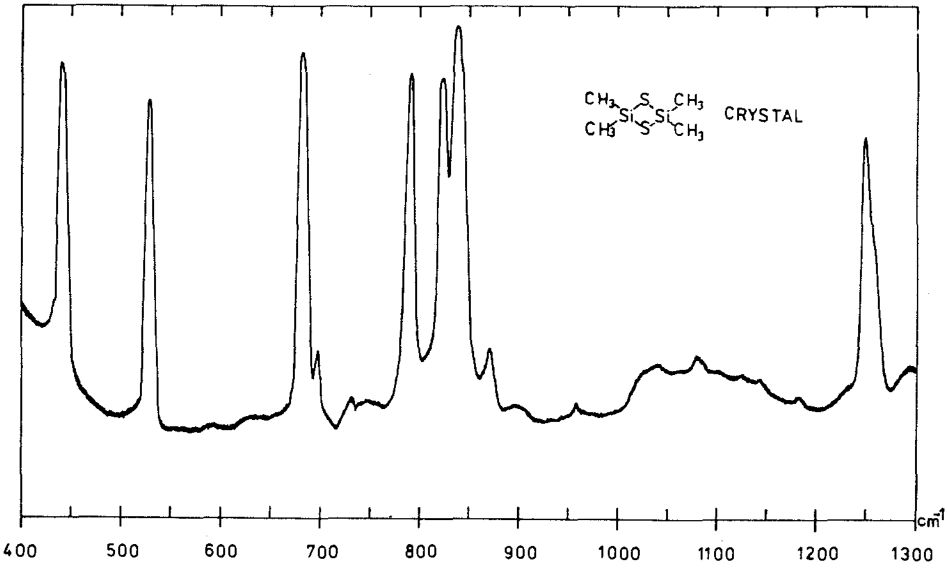


Fig. 4. Infrared spectrum of crystalline tetramethylcyclodisilthiane at 77 K.

TABLE 1

INFRARED SPECTRA OF TETRAMETHYLCYCLODISILTHIANE AND HEXAMETHYLCYCLOTRISILTHIANE (cm^{-1})[a]

$((CH_3)_2SiS)_2$				$((CH_3)_2SiS)_3$			
In CS_2 [1]	crystal (77 K)	Ar matrix	Assignments of frequencies	In CS_2 [1]	crystal (77 K)	Ar matrix	Assignments of frequencies
440s	439s	443s	ring stretch B_{2u}	449s	451w	452m	$\nu_s(SSi_2)$
473vw				473vw			
511(sh)				486vw			
528s	527s	536m	ring stretch B_{3u}	521vs	523vs	529vs	$\nu_{as}(SSI_2)$
				580vw			
				598vw			
673s	682vs	680vs	$\nu(SiC_2)$ B_{3u}	670s	675m	676s	$\nu_s(SiC_2)$
				694w		693vw	$\nu_s(SiC_2)$
	741vw	739vw		750w	745w	743vw	
				776(sh)	773w	773w	$\nu_{as}(SiC_2)$
787s	790s	784s	$\nu(SiC_2)$ B_{1u}	790w	790m	778m	$\nu_{as}(SiC_2)$
835s	823s	830m	< (HSiC)	821vs	822s	823s	< (HSiC)
	837vs	837vs		840s	841vs	842s	
				1018m			
1043m				1049m			
1080(sh)							
1250s	1249s	1251w	< (HCH)	1253s	1253m	1253m	< (HCH)
		1256m	< (HSiC)			1255(sh)	< (HSiC)
		1263vw				1262(sh)	

[a] s = strong, m = medium, w = weak, v = very, (sh) = shoulder.

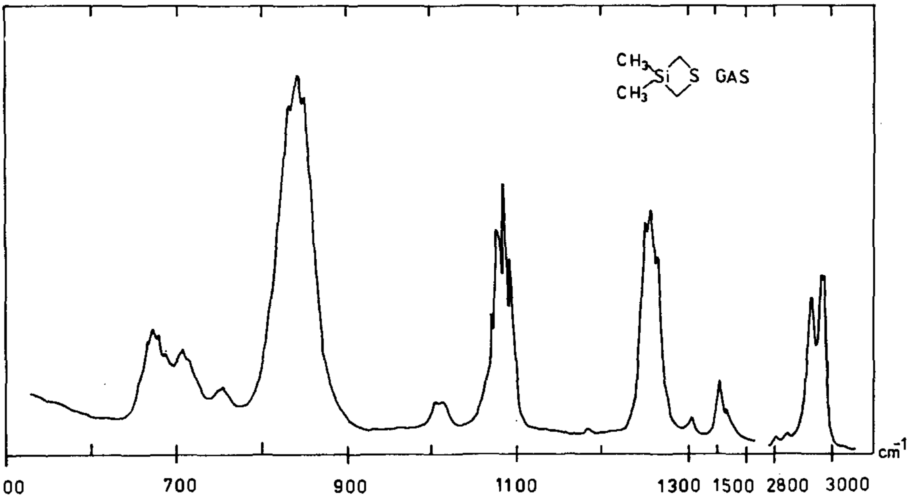


Fig. 5. Infrared spectrum of gaseous 3,3-dimethyl-3-silathietane.

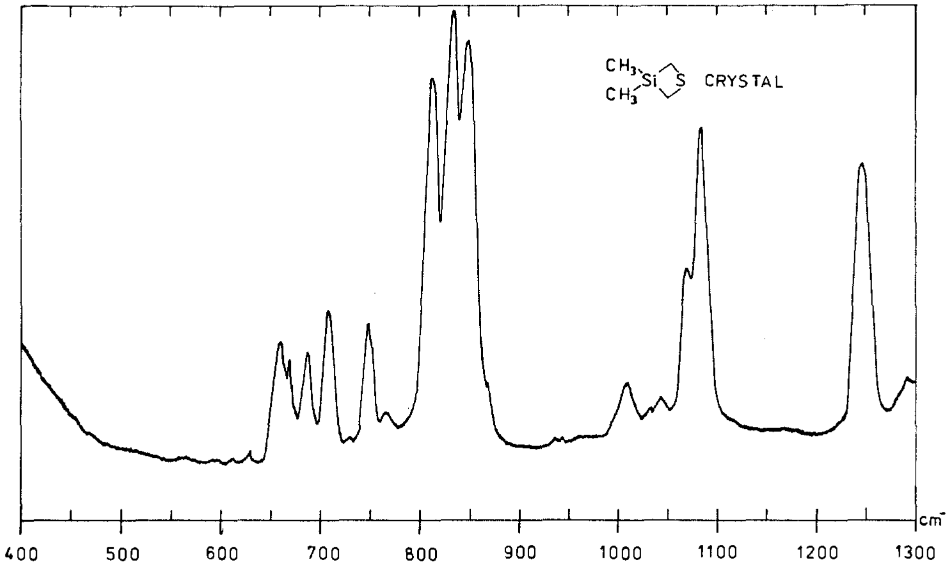


Fig. 6a. Infrared spectrum of crystalline 3,3-dimethyl-3-silathietane at 77 K.

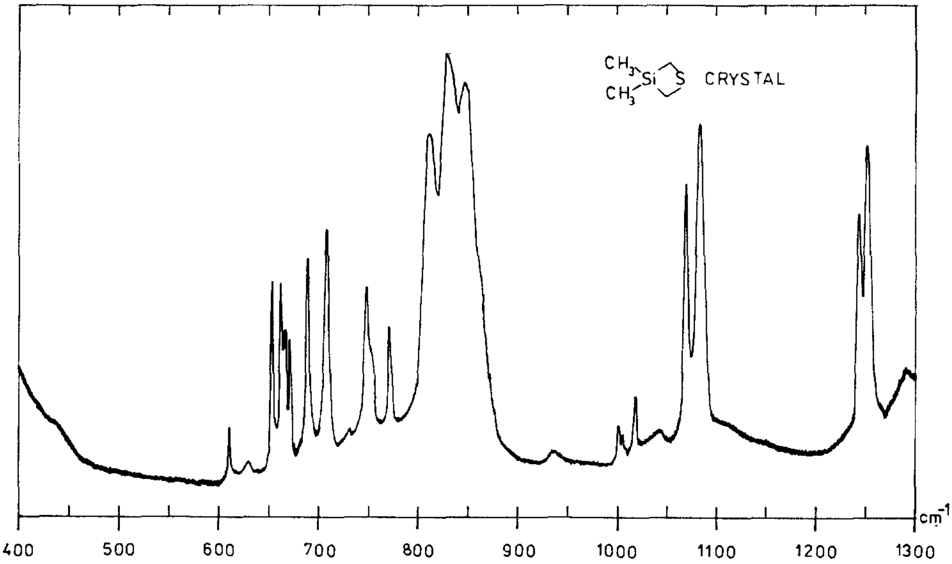


Fig. 6b. Infrared spectrum of crystalline 3,3-dimethyl-3-silathietane at 77 K (after annealing).

impurities of I in II and vice versa are observed in matrix spectra though some vibrational frequencies of I are close to that of II. Thus in the spectra of I besides its own bands, weak bands at 443, 536 and 784 cm^{-1} belonging to II are observed. It seems that a partial conversion I into II cannot be avoided when condensing I onto the target. The reverse process (transformation of II into I) occurs in the condensed phase upon melting of II. Any bands which are not characteristic of I or II and might be assigned to IV or to other products were not observed in the spectra.

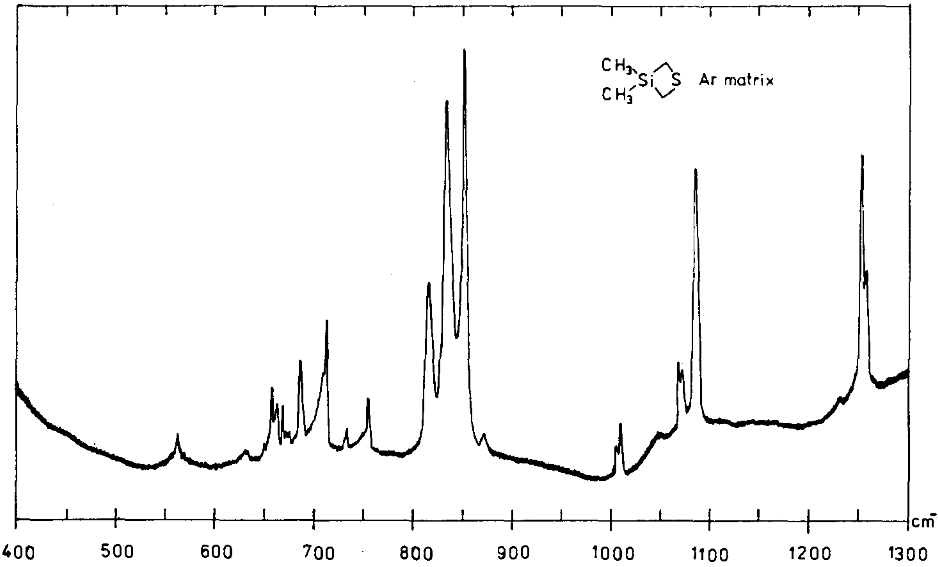


Fig. 7. Infrared spectrum of 3,3-dimethyl-3-silathietane in Ar matrix at 10 K.

B. 3,3-Dimethyl-3-silathietane. IR spectra of gaseous and crystalline samples as well as Ar matrix isolated of III are shown in Figs. 5-7. The Raman spectrum of liquid III is given in Fig. 8. The values of vibrational frequencies of III are listed in Table 2. Assignments of the observed frequencies to basic vibration modes are presented in Table 3.

The selection rules for 42 normal vibrations and their activity in the IR and Raman spectra will be as follows: $13A_1$ (IR, Raman), $8A_2$(Raman), $11B_1$(IR,

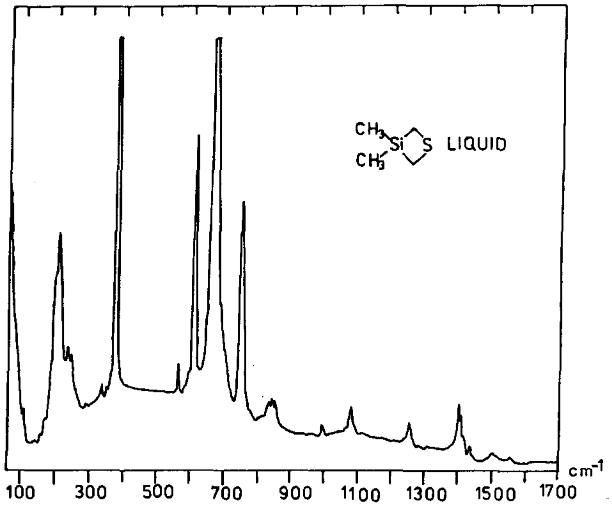


Fig. 8. Raman spectrum of liquid 3,3-dimethyl-3-silathietane.

TABLE 2

INFRARED AND RAMAN SPECTRA OF 3,3-DIMETHYL-3-SILATHIETANE (cm^{-1})

IR, gas	IR, crystal[a]		IR, Ar matrix	Raman, liquid, (depolarisation ratio)
	i	ii		
				198m
				209w (0.69)
				238w
				343vw
				378vs (0.11)
			562w	567w (0.15)
	610vw	610w		614s (0.07)
	630vw	630vw	630vw	
	656w	652s	649vw	
	658m		657w	
	661m	662s	662w	
669.8m	669m	666m		
		671m	674vw	671vs (0.22)
678.6w	678vw			
683.5w	686m	688s	685m	
706.3w	708m	708s	709w	
712.5w			712m	
750.0vw	749m	748s	749vw	
	768vw	771m	752m	755m (0.15)
			807w	
	813vs	813vs	815s	
		828vs		
833.8vs	833vs	831vs	833vs	839vw
842.6vs				
850.0vs	849vs	847vs	850vs	849vw (0.41)
	870vs		872vw	
	936vw	936vw		
987.0m				
		1001w	1005w	
1008.9w	1009w	1006vw	1008w	
1016.8w		1012vw		
		1019w		
			1034vw	
	1044vw	1044vw		
			1057vw	
1069.6s	1070w	1069s	1068w	
1077.5s			1072w	
1084.5vs	1083vs	1084vs	1084vs	1086w (0.21)
1089.8s			1132w	
1141.7vw			1143vw	
			1146w	
	1157w	1157w		
			1162w	
	1173vw	1173vw		
1180.3vw				
	1224vw	1224vw		
			1231vw	
	1246vs	1244vs		
1252.4vs		1252vs	1252vs	
1256.0vs			1257s	1256vw (0.24)
1258.5vs				

TABLE 2 (continued)

IR, gas	IR, crystal [a]		IR, Ar matrix	Raman, liquid, (depolarisation ratio)
	i	ii		
1263.8m				
1308.7vw				
		1346w	1350w	
	1370vw	1370w	1370vw	
	1376wv			
		1385m	1382w	
	1393w	1393m	1396w	
		1400m		
1408m [b]	1407w	1407w	1407w	1407w (0.73)
1416m			1411vw	
1422w				
			1435vw	
1445w	1440vw	1440vw	1440w	
1455w		1452vw		
		1460vw	1460w	
2808vw	2800vw			
	2850m	2850w		
2854vw			2856w	
		2870w	2875m	
	2890m	2890m		
			2905w	
	2919vs	2919vs		
			2928m	
2935s			2935w(sh)	
		2950s		
	2960vs	2958s		
2966s				
2972s			2975w	
2998w(sh)			2995w	

[a] i-crystal at 77 K. ii-annealed crystal at 77 K. [b] Gasphase spectra in the region 1400–3000 cm^{-1} were recorded on the Unicam SP-1200 spectrophotometer. s = strong, m = medium, w = weak, v = very, sh = shoulder.

Raman), $10B_2$(IR, Raman) for the planar four-membered ring with C_{2v} symmetry and $24A'$(IR, Raman), $18A''$(IR, Raman) for the non-planar four-membered ring of C_s symmetry.

To analyze the IR bands envelopes in the spectrum of gaseous III, calculation of moments of inertia and rotational constants for the planar and non-planar models of molecules III was carried out with the use of structural parameters of silacyclobutane [10] and thietane [11] (four-membered rings containing the same heteroatoms). According to the calculation the molecule III appears to be a prolate asymmetrical top. The value of ΔPR [12] for the A band envelope is equal to 12.6 cm^{-1} and 11.6 cm^{-1} for the planar and non-planar molecules, respectively. For the C band envelopes, ΔPR is equal to 18.8 cm^{-1} for the planar molecule and to 17.8 cm^{-1} for the non-planar one.

In the IR spectrum of gaseous III, as far as its medium region is concerned, the only band at 1084.5 cm^{-1} has a distinct envelope with ΔPR = 12.3 cm^{-1} which

TABLE 3

INFRARED AND RAMAN SPECTRA OF 3,3-DIMETHYL-3-SILATHIETANE (cm^{-1})

Frequencies (cm^{-1})	Types of symmetry	Assignments of frequencies
1407	$B_2{}^a$, $A''{}^b$	< (HCH), < (HCSi)
1256	A_1, A'	< (HCH), < (HCSi)
1086	A_1, A'	CH_2 wagging
849	B_2, A''	< (HCSi)
839	B_1 A'	< (HCSi)
813	A_2, A''	< (HCSi)
755	B_1, A'	CH_2 rocking
712	B_1, A'	$\nu_{as}(SiC_2)$
671	A_1, A'	ring stretching
649	B_2, A''	ring stretching
614	A_1, A'	$\nu_s(SiC_2)$
567	B_1, A'	ring stretching
378	A_1, A'	ring deformation
209	B_1, A'	< (CSiC)

[a] For the planar four membered ring. [b] For the non-planar four membered ring.

indicates that this band belongs to the A type. In the Raman spectrum, the intense polarized line at 1086 cm^{-1} corresponds to this bands. Therefore the band 1084.5 cm^{-1} was assigned to A_1 (A') wagging of CH_2 groups of the four-membered ring. The Raman polarized lines at 674 and 567 cm^{-1} were assigned to the A_1 (A') stretching of the ring whereas the line at 375cm^{-1} to the A_1 (A') deformation mode of the ring. The other assignments in Table 3 are similar to those of dimethylsilacyclobutane [13].

Comparison of the data of Table 2 shows the similarities of the spectra for crystalline and Ar matrix-isolated samples of III, whereas in the spectrum of gaseous III some bands disappear. Thus the symmetry of molecules III in the crystal cell is close to that in the matrix and lower than that in the gas phase. Additional bands observed both in the matrix and crystal spectra are assigned to the A_2 vibration modes forbidden for the molecule with a planar four-membered ring. This indicates the non-planar ring structure for the III both in the crystal and in the matrix. The absence of bands assigned to A_2 vibrational modes in the IR spectrum of gaseous III and the value of $\Delta PR = 12.3$ cm^{-1} for the 1084.5 cm^{-1} band, close to the theoretical value of 12.6 cm^{-1} calculated for the planar model of III, indicate either a planar ring structure of the molecule III in gas phase or a non-planar ring structure with a small dihedral angle. Similar structural changes were observed for 1,1-dimethyl-1-silacyclobutane [13].

II. Theoretical IR spectrum and molecular structure of dimethylsilanthione

Since the experimental frequencies for IV were unknown, we calculated the theoretical IR spectrum for this molecule. Structural parameters and the force constant F(Si=S) were determined using the CNDO/2 method. This method overestimates values of bond distances for elements of the third period. To make correction we calculated also the structure and F(Si=C) of 1,1-dimethyl-1-silaethylene (V) and compared them with ab initio data [14,15] and F(Si=C) obtained from

TABLE 4

COMPUTED STRUCTURE AND SPECTRAL PARAMETERS OF 1,1-DIMETHYL-1-SILAETHYLENE AND DIMETHYLSILANTHIONE

	$Me_2Si{=}CH_2$			$Me_2Si{=}S$		$Si{=}S$
	[14]	[15]	i	i	ii	[17]
r(Si=X) (Å) [a]	1.692	1.728	1.892	2.190	1.993	1.929
r(Si–C) (Å)	1.873	1.924	2.074	2.067	1.881	–
r(C–H) (Å)	1.087	1.082	1.12	1.12	1.09	–
< (CSiC) (°)	113.7	114.4	111.4	111.4	111.4	–
ν(Si=X) (cm^{-1})			1001	626	732 [b]	749.5
F(Si=X) (mdyn/Å)			5.77	6.13	4.72	4.94

[a] X = C, S. [b] Computed in diatomic approximation. i-computed in present work. ii-calculated values corrected for the CNDO/2 errors.

the experimental IR spectrum of $Me_2Si{=}CH_2$ [16]. The structural parameters for molecules IV and V and F(Si=X) (X = C, S) are presented in Table 4, where experimental bond distance and F(Si=S) for the diatomic Si=S molecule are also given [17]. It is seen that the CNDO/2 Si=S bond distance for IV and the value of F(Si=S) are in a good agreement with those of the diatomic molecule. The Si=S bond distance in IV is about 8% shorter than a single Si–S bond (2.156 Å, [9]). This is similar to the shortening of the Si=C bond with respect to the Si–C bond [14,15,18].

Calculation of normal vibrations of IV resulted in the following values of IR frequencies (cm^{-1}): 884 $\nu_s(SiC_2)(A_1)$, 735 $\nu_{as}(SiC_2)$ (B_2), 626 ν_s(Si=S) (A_1), 200 $< CSiC(A_1)$.

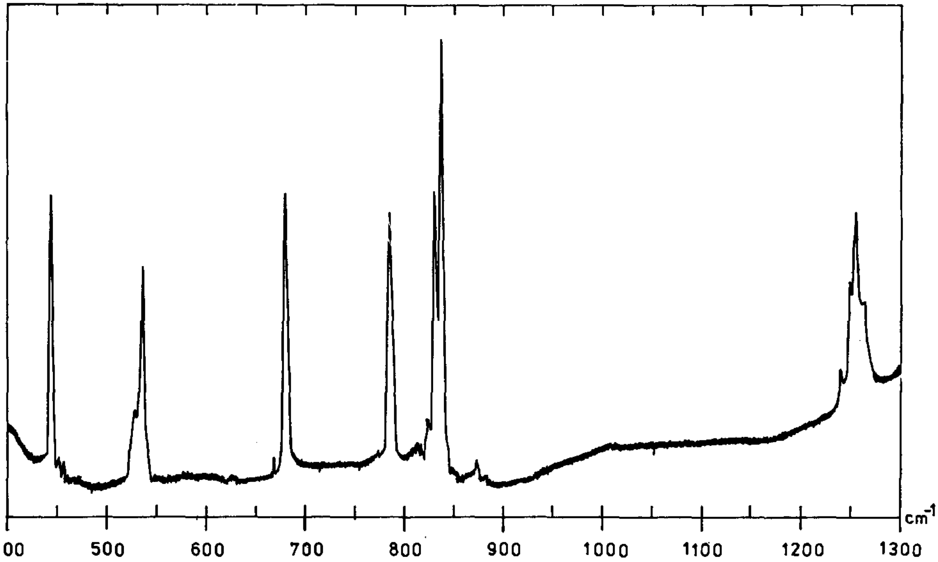


Fig. 9. Infrared spectrum of matrix-isolated VLPP products of hexamethylcyclotrisilthiane at 730°C, 4×10^{-3} Torr.

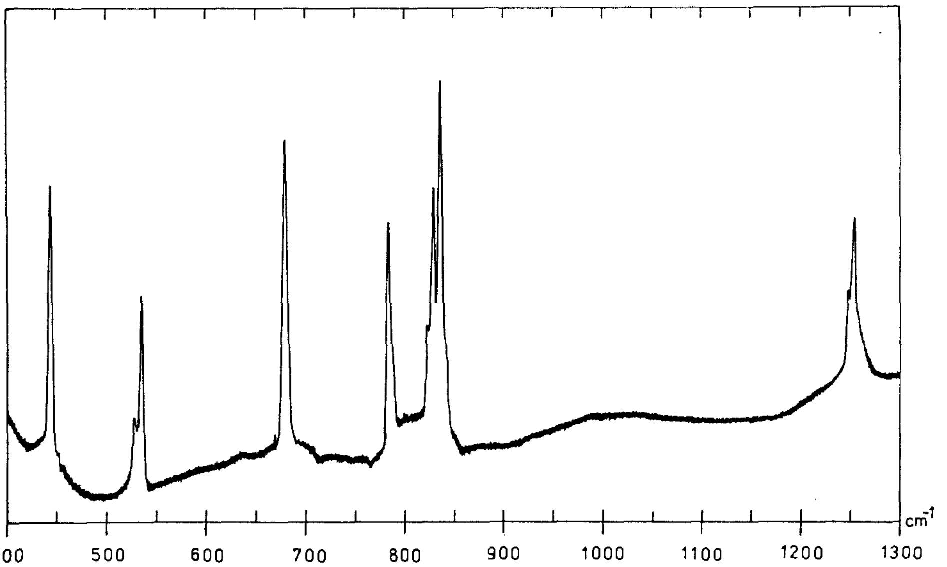


Fig. 10. Infrared spectrum of matrix-isolated VLPP products of tetramethylcyclodisilthiane at 730°C, 4×10^{-3} Torr.

III. Infrared spectra of VLPP products isolated in argon matrices

A. Pyrolysis of hexamethylcyclotrisilthiane and tetramethylcyclodisilthiane. The IR spectra of pyrolysis products of I and II are shown in Figs. 9 and 10. The bands at 443, 536, 680, 784, 830 and 837 cm^{-1} corresponding to II and very weak bands of I

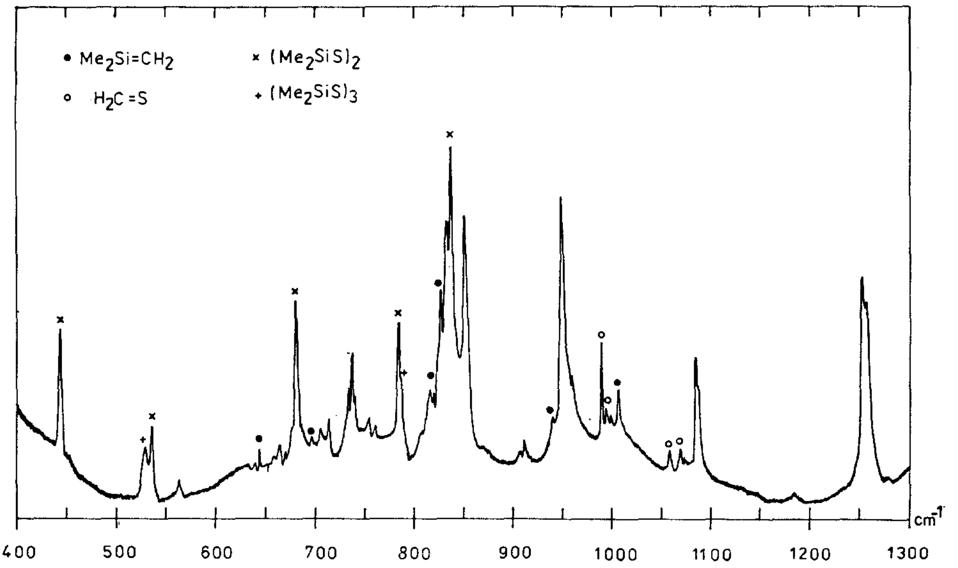


Fig. 11. Infrared spectrum of matrix-isolated VLPP products of 3,3-dimethyl-3-silathietane at 740°C, 1×10^{-2} Torr.

at 529 and 822 cm^{-1} are observed in the both spectra. That is, the pyrolysis of two different cyclosilthianes results in the same product viz., tetramethylcyclodisilthiane. No bands at 884, 735 and 626 cm^{-1} or near that expected for the intermediate IV were detected in the spectra.

B.Pyrolysis of 3,3-dimethyl-3-silathietane. The new bands at 1063, 1057, 1006, 994, 989, 949, 837, 826, 820, 784, 737, 695, 680, 644, 536, 529 and 443 cm^{-1} absent in the initial spectrum (Fig. 7) are observed in the IR spectrum of VLPP products of III (Fig. 11). The bands at 1006, 826, 820, 696 and 644 cm^{-1} were attributed to absorption by V [8], and the bands at 1063, 1057, 994, 989 cm^{-1} were ascribed to VI [19]. The remaining bands in the IR spectrum of the VLPP products of III correspond to ethylene (949 cm^{-1} [20]) acethylene (737 cm^{-1} [21]) to cyclosilthiane II (837, 784, 680, 536, 443 cm^{-1}) and I (788, 529 cm^{-1}), the latter being produced in very small quantities.

Discussion

In general the results of the IR spectroscopic studies on pyrolysis of cyclosilthianes and 3,3-dimethyl-3-silathietane are in a good agreement with Scheme 1. Both the intermediates dimethylsilaethylene V and thioformaldehyde VI have been identified in the pyrolysis products of III isolated in Ar matrix. On contrary we observed no dimethylsilanthione IV in IR spectra when pyrolysing both the cyclosilthianes and 3,3-dimethyl-3-silathietane. Only the dimeric form of IV, tetramethylcyclodisilthiane II, has been found. Moreover we carried out the copyrolysis of 1,1-dimethyl-1-silacyclobutane and thietane known to be authentic sources of V [8] and VI [22,23], respectively, and have found not IV but its dimer II in Ar matrix. The spectrum obtained is presented in Fig. 12. Comparison of this spectrum with the

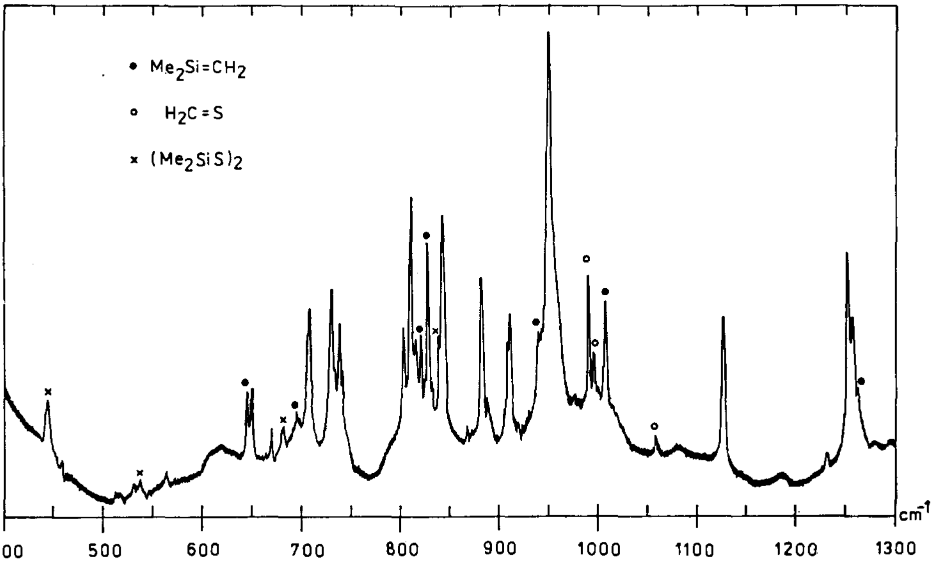
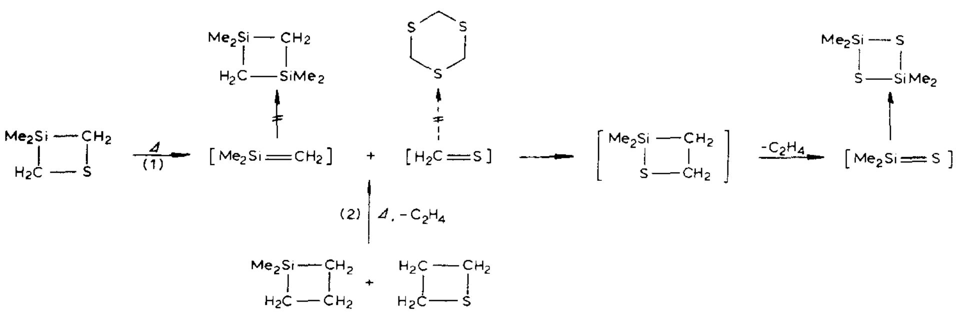


Fig. 12. Infrared spectrum of matrix-isolated products of copyrolysis of 1,1-dimethyl-1-silacyclobutane and thietane.



SCHEME 2

one for the VLPP products of III shows that intermediates V and VI and stable products (ethylene and II) are also present though the relative amount of II, compared with V and VI, formed by copyrolysis is considerably less than by the pyrolysis of III. In Scheme 2 are shown two ways of generation of V and VI from both pyrolysis (pathway 1) and copyrolysis (pathway 2).

Since disappearance of both intermediates occurs in bimolecular reactions of [2 + 2]-cycloaddition type, decreasing the pressure of pyrolysis makes possible direct spectroscopic observation of V and VI. Indeed no cyclodimerisation (1,1,3,3-tetramethyl-1,3-disilacyclobutane [24]) and cyclotrimerisation (trithioformaldehyde [25]) products were observed in spectra (Figs. 11, 12). However reaction of V and VI via [2 + 2]-cycloaddition is not affected as much by the pressure changes presumably due to its higher rate. It produces 2,2-dimethyl-2-silathietane which further decomposes resulting in ethylene and dimethylsilanthione. The cyclodimerisation of the latter is responsible for tetramethylcyclodisilthiane. An unstable 2-silathietane was suggested [26] as intermediate in copyrolysis of 1,1-dimethyl-1-silacyclobutane and thiobenzophenone.

The reduction in pressure of pyrolysis from 2×10^{-2} to 3×10^{-3} Torr led to a fall of the relative amount of II among with both VLPP products of III and copyrolysis of 1,1-dimethyl-1-silacyclobutane with thietane. Similar results were obtained when increasing dilution of the sample with matrix gas, suggesting dimerisation of IV occurs only on the wall whereas V and VI react in the gas state.

Thus, the experiments on the effects of variations of pressure and dilution confirm the intermediacy of VII, it being the source of ethylene and IV. The question whether thermally unstable VII decomposes completely or traces of it are present in VLPP products remains to be solved.

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