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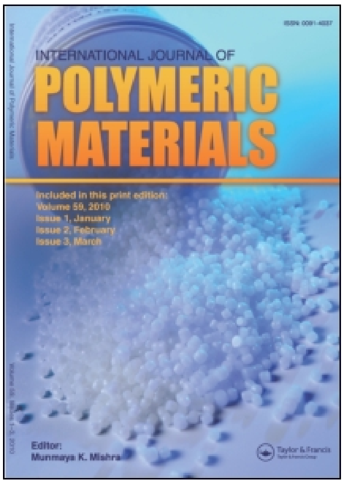

International Journal of Polymeric Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

Synthesis and Properties of *N*-Organosililmethyllactams

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To cite this Article Janiashvili, Lili K. , Khananashvili, Lotari M. , Akhobadze, Dali Sh. , Manjgaladze, Nana G. and Surmava, Antonina A.(1994) 'Synthesis and Properties of *N*-Organosililmethyllactams', International Journal of Polymeric Materials, 24: 1, 141 — 149

To link to this Article: DOI: 10.1080/00914039408028560

URL: http://dx.doi.org/10.1080/00914039408028560

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Intern. J. Polymeric Mater., 1994, Vol. 24, pp. 141–149
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Printed in Malaysia

Synthesis and Properties of *N*-Organosililmethyllactams

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The heterofunctional condensation of lactams with chloromethyl derivatives of organosilanes in the presence of catalyst—acceptor of anhydrous K_2CO_3 or Na_2CO_3 has been investigated. The hydrolytic stability of *N*-dialkyl(aryl)silylmethyllactams in neutral, acid and alkaline media has been studied. The biological activity of synthesized silicon-containing lactams has been investigated. The hydrolytic polycondensation of *N*-(methyldimethoxysilylmethyl) lactams in the presence of a catalytic quantity of concentrated hydrochloric acid at room temperature has been conducted.

KEY WORDS *N*-Organosililmethyllactams, synthesis, hydrolytic stability, biological activity

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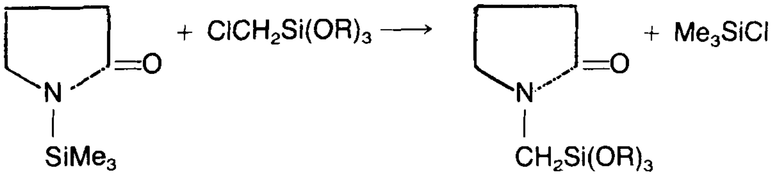
INTRODUCTION

The silylized lactams containing the bonds $\equiv$Si—N$=$[1–12] are described in the literature.

These lactams are biologically active, but hydrolytically unstable.

In Reference 13 the synthesis of *N*-trialkoxysilylmethyl-2-pyrrolidones is reported to be obtained by the interaction of *N*-trimethylsilyl-2-pyrrolidone with chloromethyltrialkoxysilanes by the following scheme:



However, this method is rather laborious.

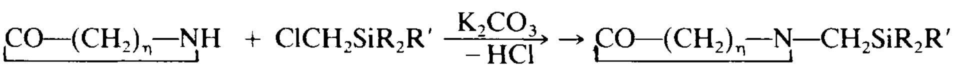
EXPERIMENTAL PART

Lactams and triorganochloromethylsilanes were used as the original substances for the synthesis of silicon-containing lactams.

The purity of the original substances was controlled on the gas-liquid chromatograph "LKhM-8MD," phase SKTF-100 (10%) on chromosorb NAW, gas-carrier helium, on a 2 M column. PMR spectra were obtained on the spectrometer "Bs-497" of Tesla company (Brno, Chekho-Slovakia) with an operating frequency of 90 MHz in a solution of $CDCl_3$ with the internal standard, TMC.

RESULTS AND DISCUSSION

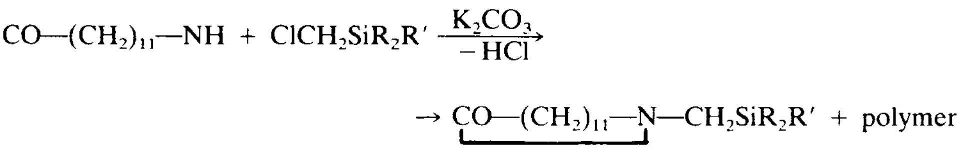
The reaction of 2-pyrrolidone and ω-caprolactam with chloromethyltriorganosilanes was conducted at the temperature of 110°C in the presence of anhydrous K_2CO_3 or Na_2CO_3 with a molar ratio of reacting substances I:I during 4–6 hours in dried toluene. The reaction proceeded in one stage by the following scheme:



$$\underbrace{CO{-}(CH_2)_\eta{-}NH} + ClCH_2SiR_2R' \xrightarrow[-HCl]{K_2CO_3} \to \underbrace{CO{-}(CH_2)_\eta{-}N}{-}CH_2SiR_2R'$$

where at η = 3: R = CH_3, R' = C_2H_5 (I); C_4H_9 (II) i—C_4H_9 (III); C_6H_5 (IV); R = R'=OCH_3 (V); OC_2H_5 (VI); OC_4H_9 (VII); R' = CH_3, R = OCH_3 (VIII) at η = 5 R = CH_3, R' = C_2H_5 (IX); C_3H_7 (X); C_4H_9 (XI); C_6H_5 (XII). R = C_6H_5, R' = CH_3 (XIII); R = R'=OCH_3 (XIV); OC_2H_5 (XV); OC_4H_9 (XVI); R' = CH_3, R = OCH_3 (XVII).

In the above mentioned conditions the reaction of ω-dodecalactam with chloromethyltriorganosilanes does not take place. However, by changing the reaction conditions, that is by conducting the reaction between ω-dodecalactam and chloromethyltriorganosilanes at a higher temperature (180–200°C) and in the absence of solvent we succeeded in isolating *N*-triorganosilylmethyl-ω-dodecalactams with satisfactory yields (34–45%) by the following scheme:



$$CO{-}(CH_2)_{11}{-}NH + ClCH_2SiR_2R' \xrightarrow[-HCl]{K_2CO_3}$$

$$\to \underbrace{CO{-}(CH_2)_{11}{-}N}{-}CH_2SiR_2R' + \text{polymer}$$

where R = CH_3, R' = C_2H_5 (XVIII); C_4H_9 (XIX); C_6H_5 (XX); R = R'=OCH_3 (XXI); R' = OCH_3; R = OCH_3 (XXII).

In this reaction, along with *N*-triorganosilylmethyl)-ω-dodecalactams, in all cases a rubbery polymer of poly-ω-dodecane amide is formed, containing no silicon atoms in the chain.

Synthesized silicon-containing lactams are colourless, odourless viscous liquids, except *N*-(trimethoxysililmethyl)-ω-dodecalactam, which is a very viscous substance and crystallizes after a few days. All the synthesized products are quite soluble in various organic solvents.

In Table I some physical chemical characteristics, elemental analysis and yield of synthesized *N*-triorganosililmethyllactams are given.

The structure of the synthesized *N*-triorganosililmethyl derivative lactams was established with the help of infrared and PMR spectra.

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TABLE I

Physico-chemical data, elemental analysis and *N*-triorganosililmethyllactam output

Compound, *N*:	Output, %	T_{boil}, °C, (*P*, mmHg)	n_D^{20}	d_4^{20}	MR_D		Found, %				Formula	Calculated, %				MM	
					Found	Calculated	C	H	N	Si		C	H	N	Si	Found	Calculated
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
I	45	71–73	1.4460	0.8356	57	55.1	57.85	10.77	7.36	15.43	$C_9H_{19}Si_1N_1O_1$	58.38	10.27	7.57	15.14	179	185
II	40	102–103	1.4640	0.8996	64	64.4	61.59	10.69	7.03	13.20	$C_{11}H_{23}Si_1N_1O_1$	61.97	10.80	6.57	13.15	210	213
III	39	93–95	1.4620	0.8986	65	64.4	61.81	9.56	6.90	12.86	$C_{11}H_{23}Si_1N_1O_1$	61.97	10.80	6.57	13.15	217	213
IV	47	130–134	1.4849	0.9636	69	70.4	67.12	7.83	6.43	12.52	$C_{13}H_{19}Si_1N_1O_1$	66.95	8.15	6.01	12.01	230	233
V	45	104/1	1.4620	1.1601	52	52.9	44.20	7.64	6.21	12.56	$C_8H_{17}Si_1N_1O_4$	43.84	6.76	6.39	12.79	219	206
VI	40	100/0.5	1.4545	1.0510	67	66.9	51.19	8.91	5.43	10.86	$C_{11}H_{23}Si_1N_1O_4$	50.57	8.81	5.36	10.73	258	261
VII	38	196–198	1.4300	0.9480	94	94.8	58.90	10.56	4.61	8.56	$C_{17}H_{35}Si_1N_1O_4$	59.13	10.14	4.06	8.12	340	345
VIII	36	97/1	1.4596	1.1701	47.5	47.1	47.99	8.02	7.10	14.22	$C_8H_{17}Si_1N_1O_3$	47.29	8.37	6.90	13.80	197	203
IX	43	78/1	1.4655	0.9301	63.5	64.4	62.15	11.00	6.51	13.00	$C_{11}H_{23}Si_1N_1O_1$	61.97	10.80	6.57	13.15	205	213
X	41	125/2	1.4670	0.8077	69	69.1	63.91	11.19	6.72	12.01	$C_{12}H_{25}Si_1N_1O_1$	63.45	11.01	6.12	12.33	200	227
XI	38	108/1	1.4570	0.8666	74.5	73.7	65.02	11.54	5.75	11.93	$C_{13}H_{23}Si_1N_1O_1$	64.72	11.20	5.81	11.62	237	241
XII	46	170/1	1.4720	0.9475	79.2	79.7	69.56	8.31	5.02	11.33	$C_{15}H_{23}Si_1N_1O_1$	68.97	8.81	5.36	10.73	208	261
XIII	20	190/1	1.4530	0.9060	95.8	96.2	74.75	7.65	4.11	8.09	$C_{20}H_{25}Si_1N_1O_4$	74.30	7.74	4.33	8.67	321	323
XIV	37	116/1	1.4709	1.0905	63.3	62.2	48.79	8.87	5.51	10.94	$C_{20}H_{21}Si_1N_1O_4$	48.58	8.50	5.67	11.34	239	247
XV	39	127/1.5	1.4565	0.9815	77.4	76.2	54.12	9.66	4.96	9.10	$C_{13}H_{23}Si_1N_1O_4$	53.98	9.34	4.84	9.69	279	289
XVI	41	158–160	1.4440	0.9377	104.8	104.1	61.57	11.07	3.51	7.92	$C_{19}H_{39}Si_1N_1O_4$	61.13	10.46	3.75	7.51	370	373
XVII	42	170/3	1.4530	1.0625	60.8	61.4	52.25	9.30	6.51	11.93	$C_{10}H_{21}Si_1N_1O_3$	51.95	9.09	6.06	12.12	239	231
XVIII	39	75/2	1.4160	0.7815	93.1	92.3	69.50	11.24	5.01	9.60	$C_{17}H_{35}Si_1N_1O_1$	68.69	11.78	4.71	9.43	290	297
XIX	37	175/1	1.4400	0.7865	100.6	101.6	69.45	11.22	4.02	8.02	$C_{19}H_{39}Si_1N_1O_1$	70.15	11.02	4.30	8.61	320	325
XX	45	91 $2\ 10^{-2}$	1.5280	0.7785	93.5	92.3	63.00	9.61	4.00	7.79	$C_{21}H_{35}Si_1N_1O_1$	73.04	10.14	4.05	8.11	340	345
XXI	38	197 $1\ 10^{-2}$	—	—	—	—	58.45	10.02	4.57	8.12	$C_{16}H_{33}Si_1N_1O_4$	58.01	9.97	4.23	8.46	328	331
XXII	40	170 $2\ 10^{-2}$	1.4595	0.9564	90.1	89.3	61.22	10.86	4.00	8.46	$C_{10}H_{33}Si_1N_1O_3$	60.95	10.48	4.44	8.89	320	315

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In the infrared spectra of all the synthesized compounds the absorption bands for the valence vibrations of the C—Cl bond in the region of 780 sm^{-1} are absent. At the same time, in all the spectra distinctive absorption bands for the deformation vibrations of Si—CH_3 groups (1260–1250 sm^{-1}), antisymmetrical and symmetrical valence vibrations of the Si—CH_2 bonds in the region of 820–760 and 700–650 sm^{-1}, respectively, are developed. For CO groups, being coupled with a nitrogen atom, distinctive absorption bands are found in the region of 1720 sm^{-1} (for *N*-triorganosililmethyl-2-pyrrolidones), 1680 sm^{-1} (for *N*-triorganosililmethyl-ε-caprolactams) and 1650 sm^{-1} (for *N*-triorganosililmethyl-ω-dodecalactams). In addition, in the spectra of I, IX and XVIII compounds absorption bands are observed in the region of 1235–1220 sm^{-1} (Si—C_2H_5), in compounds II, XI and XIX; at 1190 sm^{-1} (Si—C_4H_9), in compounds IV, XII, XIII and XX; at 1429 sm^{-1} (Si—C_6H_5), in compound X; and at 1230 sm^{-1} (Si—C_3H_7). Moreover, in compounds VIII, XVII and XXII the absorption bands are observed in the region of 1280 sm^{-1} (Si—CH_3) and 1163–1149 sm^{-1} (Si—O—C).

Data from the PMR spectra of the synthesized *N*-triorganosilillactams are given in Table II.

To determine the hydrolytic stability of *N*-dialkyl(aryl)silylmethyllactams, namely, *N*-dimethyl(phenyl)silylmethyl-2-pyrrolidone (IV), *N*-dimethyl(phenyl)-silylmethyl-ε-caprolactam (XII) and *N*-dimethyl(phenyl)sililmethyl-ω-dodecalactam (XX), they are held at 100°C for 5 hours in neutral, acid and alkaline media. The data on hydrolytic stability are given in Table III. These data indicate that sililized lactams with $\equiv Si-\overset{|}{\underset{|}{C}}-N=$ bonds are hydrolytically stable compounds.

The hydrolytic polycondensation of *N*-alkyldialkoxysililmethyllactams was also studied. The investigation of hydrolytic polycondensation of *N*-(methyldimethoxysililmethyl)lactams showed that, depending on the reaction condition, this process proceeds ambiguously. So, when this reaction is conducted in methyl alcohol in the presence of a catalytic quantity of concentrated hydrochloric acid and at room

TABLE II

Chemical shifts of proton signals in PMR spectra of triorganosililmethyllactams

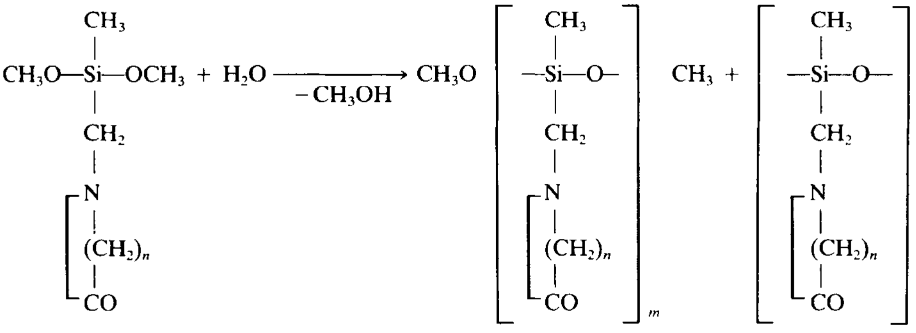
N.	Code	PMR Spectrum Solvent $CDCl_3$
1.	I	0.01–0.02 S (Si(CH_3)$_2$), 2.2τ (SiCH_2), 2,6τ (cycle protons), 0.2 M (SiC_2H_5), 2.56–2.58 S (Si—CH_2—N)
2.	II	0.01–0.02 S (Si(CH_3)$_2$), 2.9–3.4τ (Si—C_4H_9), 2.5τ (Si—CH_2), 2.5–2.6 S (Si—CH_2—N), 2.9τ (cycle protons)
3.	IV	0.01–0.02 S (Si(CH_3)$_2$), 6.9 M (Si—C_6H_5), 2.57 S (Si—CH_2—N), 2.85τ (cycle protons)
4.	V	0.015–0.02 S (Si(CH_3)$_2$), 2.6 S (Si—CH_2—N), 0.2–0.26 M (Si—C_2H_5), 2.95τ (cycle protons)
5.	VI	0.015–0.02 S (Si(CH_3)$_2$), 2.0–2.18τ (Si—C_3H_7), 2.65 S (Si—CH_2—N) 2.85τ (cycle protons)
6.	VIII	0.015–0.02 S (Si(CH_3)$_2$), 7.3 M (SiC_6H_5), 2.3τ (Si—CH_2), 2.85τ (cycle protons)
7.	XVIII	0.02 S (Si(CH_3)$_2$), 0.2–0.3 M (Si—C_2H_5), 2.3–2.4τ (Si—CH_2), 2.75–2.85τ (cycle protons)
8.	XIX	0.02 S (Si(CH_3)$_2$), 3.0τ (Si—C_4H_9), 2.35τ (Si—CH_2), 2.8τ (cycle protons)
9.	XX	0.018 S (Si(CH_3)$_2$), 2.35τ (SiCH_2), 7.1 M (Si—C_6H_5), 2.5–2.7τ (cycle protons)

TABLE III

Hydrolytic stability of *N*-dialkylarylmethyllactams in neutral, acid and alkaline medium (100°C, 5 hours)

Compound N.	Solvent system	Medium: Neutral — Before boiling g(%)	Neutral — After boiling g(%)	Acid — Before boiling g(%)	Acid — After boiling g(%)	Alkaline — Before boiling g(%)	Alkaline — After boiling g(%)
IV	Toluol-water	1 (100)	0.99 (99)	1 (100)	0.978 (97.8)	1 (100)	0.967 (96.7)
	Dioxan-water	1 (100)	0.98 (98)	1 (100)	0.983 (98.3)	1 (100)	0.973 (97.3)
XIII	Toluol-water	1 (100)	0.998 (99.8)	1 (100)	0.985 (98.5)	1 (100)	0.983 (98.3)
	Dioxan-water	1 (100)	0.976 (97.6)	1 (100)	0.979 (97.9)	1 (100)	0.987 (98.7)
XX	Toluol-water	1 (100)	0.993 (99.3)	1 (100)	0.995 (99.5)	1 (100)	0.991 (99.1)
	Dioxan-water	1 (100)	0.98 (98)	1 (100)	0.987 (98.7)	1 (100)	0.987 (98.7)

temperature the mixture of oligosiloxanes of the linear (73–84%) or cyclic (11–21%) structure is formed. In this case the process proceeds by the scheme:



where at

$n = 3$: $m = 9$ (XXIII), $p = 3$ (XXVI)

$n = 5$: $m = 11$ (XXIV), $p = 4$ (XXVII)

$n = 11$: $m = 15$ (XXV), $p = 4$ (XXVIII)

The lowering of the reaction temperature to −3 to −5°C leads to an increase in the line or oligosiloxane yield to 90%.

When the hydrolytic polycondensation of *N*-(methyl-dimethoxysililmethyl)lactams is conducted in ethyl alcohol in the presence of a catalytic quantity of concentrated hydrochloric acid and at room temperature only six- and eight-unit cyclosiloxanes are formed.

The course of the reaction was controlled by an increase in the specific viscosity

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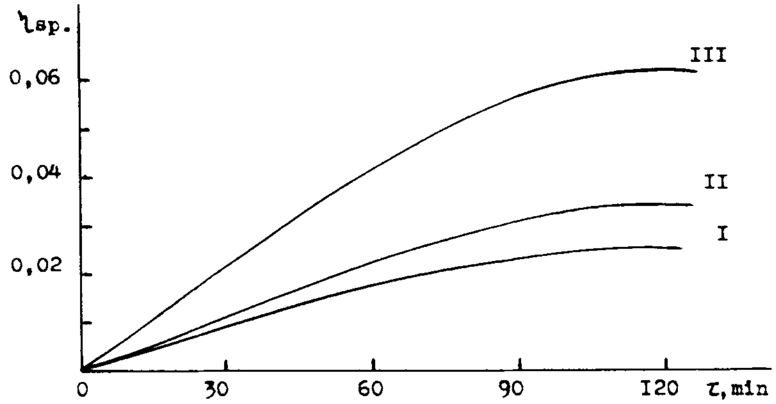

FIGURE 1 Relationship between η_{specific} variation and time under condensation of *N*-dimethoxymethylsililmethyl-2-pyrrolidone (I), *N*-dimethoxymethylsililmethyl-ε-caprolactam (II) and *N*-dimethoxymethylsililmethyl-ω-dodecalactam (III).

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over time (Figure 1). As is seen from the figure, the hydrolytic polycondensation of *N*-(methyldimethoxysililmethyl)-ω-dodecalactam proceeds more rapidly than that of *N*-(methyldimethoxysililmethyl)-ε-caprolactam and *N*-(methyldimethoxysililmethyl)-2-pyrrolidone and in 90 minutes oligo-dimensional product with maximum specific viscosity (η_{sp} = 0.06) is formed. The specific viscosities of the products of hydrolytic polycondensation of *N*-(methyldimethoxysililmethyl)-ε-caprolactam and *N*-(methyldimethoxysililmethyl)-2-pyrrolidone are not higher than 0.035 and 0.028, respectively.

Methyl-lactyl containing organocyclosiloxanes (XXVI–XXVIII) are colourless transparent liquids, completely soluble in water and chloroform, but oligosiloxanes of linear structure are resin-like transparent products, quite soluble in standard organic solvents.

Synthesized products are identified on the basis of elemental analysis, molecular mass and infrared spectral data.

In the infrared spectra of the synthesized compounds (XXIII–XXVIII) the intense absorption bands of the Si—O—Si bonds are observed in the region of 1030–1010 sm^{-1} for noncyclic organosiloxanes and in the region of 1020–1015 sm^{-1} and 1085–1080 sm^{-1}, being typical of organocyclotri- and organocyclotetrasiloxanes. Characteristic absorption bands of the lactam ring are completely retained. In so doing, as differentiated from the spectra of the initial substances (VIII, XVII and XXII) one can observe a significant decrease in the intensity (for XXIII–XXV) and a complete disappearance of the band (for XXVI–XXVIII) in the region of 960 cm^{-1} being related to the asymmetrical valence vibrations of the Si—O—C bond, as evidenced for complete consumption of the methoxy groups.

We've also studied the hydrolytic polycondensation of *N*-(methyldiethoxysililethylene)- and *N*-(methyldibutoxysililethyl)-2-pyrrolidones having been obtained by hydrosililizing of *N*-vinylpyrrolidone by methyldialkoxysilanes. This process was conducted at room temperature in ethyl alcohol in the presence of catalytic quantities of concentrated hydrochloric acid. The reaction proceeded easily with a spon-

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TABLE IV

Physico-chemical characteristics, elemental analysis, and output of lactyl-containing oligosiloxanes and cyclosiloxanes

Compound N.	Output, %	Specific	MM		Found				Formula	Calculated, %			
			Found	Calculated	C	H	Si	N		C	H	Si	N
XXIII	73	0.028	1565	1559	46.02	8.85	17.61	7.01	$C_{56}H_{105}Si_{19}N_9O_{19}$	46.06	8.69	17.27	8.64
XXIV	77	0.035	2069	2081	52.25	8.44	14.53	7.61	$C_{90}H_{171}Si_{11}N_{11}O_{29}$	51.90	8.22	14.80	7.40
XXV	84	0.06	4120	4081	62.73	10.25	10.53	4.93	$C_{212}H_{411}Si_{15}N_{15}O_{31}$	62.33	10.07	10.29	5.15
XXVI	21	—	500	471	45.49	7.35	18.02	8.71	$C_{18}H_{33}Si_3N_3O_6$	45.86	7.01	17.83	8.92
XXVII	14	—	729	740	52.21	8.45	15.33	7.32	$C_{32}H_{60}Si_4N_4O_8$	51.89	8.11	15.14	7.56
XXVIII	11	—	1089	1076	62.00	10.51	10.09	5.37	$C_{56}H_{108}Si_4N_4O_8$	62.45	10.04	10.42	5.20
XXIX	83	—	703	684	49.51	7.89	16.05	8.47	$C_{28}H_{52}Si_4N_4O_8$	49.12	7.60	16.37	8.19

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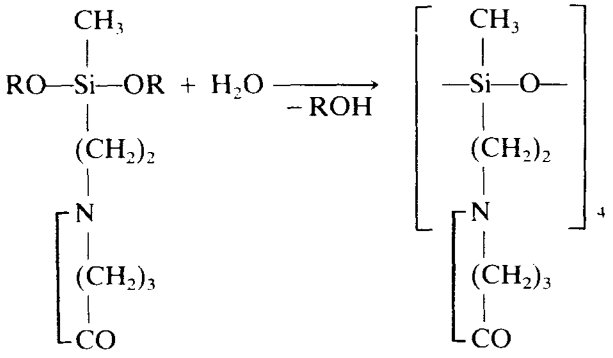
TABLE V

The effect of *N*-organosililimethyllactams on the growth of some phytopathogenic bacteria

		Compound			
	Control	II	IV	XI	XII
Microorganisms	Zone of depression of microorganism growth				
Bacterium tumefaciens	0	5.0	6.0	6.0	5.0
Xanthomonas campestris	0	4.0	5.5	8.0	1.0
Pectobacterium aroideae	0	4.0	5.5	4.0	—

taneous temperature increase up to 60–70°C. In both cases 8-unit organocyclosiloxane is formed by the scheme:



where R = C_2H_5, C_4H_9 (XXIX).

After distilling off the solvent and reprecipitating XXIX from the toluene solution by hexane addition, resin-like product was obtained, that was quite soluble in water and chloroform.

In the infrared spectra of the synthesized compound one can observe the complete disappearance of the absorption band within the region of 960 cm^{-1}, related to the asymmetrical valence vibrations of the Si—O—C bond. In addition there is a band within the region of 3600–3400 sm^{-1} that is not characteristic of OH group absorption and also a characteristic absorption band for the bonds of the pyrrolidone ring is completely retained (1720 sm^{-1} for CO).

Physical chemical data for the synthesized compounds are given in Table IV.

The biological activity of the synthesized silicon containing lactams-*N*-dimethylbutyl-silil-methyl- and *N*-dimethyl-phenylsilil-methyl-lactams was investigated.

As test objects the following strains of microorganisms were used: xanthomonas campestris (responsible for the white cabbage bacteriosis), Bacterium tumefaciens (responsible for the vine cancer disease) and Pectobacterium aroideae (responsible for the white cabbage mucous bacteriosis). The results of the investigations are given in Table V.

Analysis of the information made it possible to conclude that the substances being tested to various degrees inhibit the growth of microorganisms and have inhibitory properties.

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