

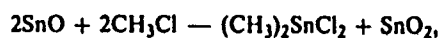
S/079/62/032/007/005/007
1032/1232

AUTHORS: ~~Andriyanov, K. A., Vasil'yeva T. V., Nudelman Z. N., Khananashvili, L. M., Kochetkova, A. C. and Cherednikova, A. G.~~

TITLE: Dimethyl-tin-dichloride; its synthesis and a study of its reaction with disodium salts of dimethyl-siloxanes.

PERIODICAL: Zhurnal obshchei khimii, v. 32, no. 7, 1962, 2307-2311

TEXT: Reaction of methyl chloride with stannous chloride in the presence of spongy metallic copper gives dimethyl-tin-dichloride (I) and methyl-tin-trichloride (II) according to the scheme



The reaction takes place between 250° and 350°C; the yield of the process and the ratio between I and II in the reaction product are temperature dependent. At 250°C mainly dimethyl-tin-dichloride is formed. The reaction of dimethyl tin-dichlorides and diethyl-tin-dichlorides with disodium salts of dimethyl-siloxanes gives polymer products the molecular weight of which exceeds 3000. There is 1 figure and 3 tables. The English-language reference [8] reads: E. Rochow, Smith, J. Am. Chem. Soc., 75, 4103 (1953).

SUBMITTED: July 5, 1961

Card 1/1

PHASE I BOOK EXPLOITATION

SOV/6118

Andrianov, Kuz'ma Andrianovich, and Vladimir Vladimirovich Skipetrov

Sinteticheskiye zhidkiye dielektriki (Synthetic Liquid Dielectrics). Moscow, Gosenergoizdat, 1962. 175 p. (Series: Polimery v elektroizolyatsionnoy tekhnike, vyp. 4) 8500 copies printed.

Editorial Board: K. A. Andrianov, Chief Ed.; K. I. Zabyrina, V. I. Kalitvyan-skiy, Yu. V. Koritskiy, A. V. Khval'kovskiy, and L. A. Epshteyn; Ed.: S. V. Shishkin; Tech. Ed.: V. V. Yemzhin.

PURPOSE: This book is intended for electrical insulation specialists, particularly technical personnel in plants and scientific research institutes.

COVERAGE: The book reviews synthetic dielectric fluids which are of interest in electrical and radio engineering. Such dielectric fluids are those possessing good electrical characteristics stable over a wide temperature range, low viscosity, and resistance to electric fields and high temperatures. The

Card 1/ 2

PHASE I BOOK EXPLOTTATION

SOV/6400

Andrianov, Kuz'ma Andrianovich.

Polimery s neorganicheskimi glavnyimi tsepyami molekul (Polymers with Inorganic Backbones in the Molecules) Moscow, Izd-vo AN SSSR, 1962. 326 p. Errata slip inserted. 4500 copies printed.

Sponsoring Agency: Akademiya nauk SSSR. Institut elemento-organicheskikh soyedineniy.

Resp. Ed.: V.V. Korshak, Corresponding Member Academy of Sciences USSR; Ed.: A.A. Zhdanov; Tech. Ed.: A.I. Ballod and I.N. Dorokhina.

PURPOSE: This book is intended for scientists, aspirants, and university teachers working in the field of polymer chemistry. It can also serve as a manual for students in this field.

COVERAGE: The book presents a systematic critical review of developments in the chemistry of polymers with inorganic

Card 1/5

2

15.8170

33267
S/062/62/000/001/006/015
B117/B101

11.2219
AUTHORS: Andrianov, K. A., Fromberg, M. B., Sorokina, L. I., and
Kirilenko, E. I.

TITLE: Polyorganoaluminoxanes and polyorganoaluminosiloxanes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh
nauk, no. 1, 1962, 78 - 86

TEXT: The composition, the structure, and properties of intermediates of polyorganoalumoxane synthesis, their conversion into polymers, and the possibility of producing compound polymers were investigated. Monomeric organoaluminum compounds were synthesized with azyloxy and chelate groups (Table 1). The solubility of the compounds obtained is largely affected by the nature of organic groups with aluminum. Aluminum isopropoxy dicaprylate and aluminum diisopropoxy caprylate are soluble, 8-hydroxyquinoline derivatives are poorly soluble in organic solvents. Dialkoxy derivatives, and above all aluminum dihalides are easily hydrolyzed by atmospheric moisture. Hydrolysis of benzoate aluminum dichloride always yields insoluble composite products, since the acyloxy group

Card 1/3

33267
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Polyorganoaluminoxanes ...

is separated. Aluminum dihydroxy caprylate and aluminum dihydroxy-8-oxyquinolate were obtained (with almost theoretical yields) by hydrolysis of aluminum diisopropoxy caprylate and aluminum diisopropoxy-8-oxyquinolate which takes place at the alkoxy groups only, and does not destroy the chelate bond nor split off the acyloxy group. These hydroxy derivatives are poorly soluble in the usual organic solvents. Their infrared spectra showed absorption bands (3600 and 3430cm^{-1}) corresponding to associated HO---X and $-\text{OH---X}$ hydroxyl groups. Experiments have shown that the synthesis of polyorganoaluminoxanes proceeds via hydroxyl derivatives which are condensed with alkoxy groups into polymers either directly or due to a reaction with hydroxyl groups bound with aluminum. The interaction of hydroxyl derivatives of organoaluminum compounds with alkoxy derivatives is a general one. This reaction takes place among organoaluminum monomers and among organosilicon and organoaluminum compounds. Isopropyl alcohol is separated, and a polymer is formed by polycondensation of aluminum diisopropoxy-8-oxyquinolate with hydroxyl derivatives of organosilicon compounds. Polycondensation of aluminum diisopropoxy caprylate with α, ω -dihydroxy-methyl-phenyl siloxanes

Card 2/13

Polyorganoaluminoxanes ...

33267
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B117/B101

yields linear polyorganoaluminosiloxanes. Such a polymer is elastic and well soluble in organic solvents. These properties are also preserved with continuous heating (200°C). Heterofunctional polycondensation of alkoxy derivatives of organoaluminum compounds also takes place with organosilicon compounds in which hydroxyl groups are replaced by other functional groups: In this process, caprylic acid is separated presumably due to the presence of HCl traces. Therefore, insoluble, steric polymers of compound structure are formed, but no linear molecules. There are 3 figures, 2 tables, and 7 references: 3 Soviet-bloc and 4 non-Soviet-bloc. The two references to the English-language publications read as follows: USA patent 2744074 (1956); English patent 783679 (1957). ✓

ASSOCIATION: Vsesoyuznyy elektrotekhnicheskiy institut im. V. I. Lenina
(All-Union Electrotechnical Institute imeni V. I. Lenin)

SUBMITTED: July 19, 1961

Legend to Table 1: (1) Compound; (2) melting point, °C.

Card 3/3

33268
S/062/62/000/001/007/015
B117/B101

S-3700

AUTHORS: Andrianov, K. A., and Volkova, L. M.

TITLE: Reactions of amines with bis-(chloro-methyl)-tetramethyl disiloxane and its derivatives

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 1, 1962, 87 - 90

TEXT: The interactions of 1,1,1,2,3,4,4,4-octamethyl-2,3-di-(chloro-methyl)-tetrasiloxane with hexamethylene diamine, and bis-(chloro-methyl)-tetramethyl disiloxane with trimethyl-(β -amino-ethoxy)-silane were studied. Both hydrogen atoms of the amino group were substituted. Therefore, to avoid cyclization, the reaction of trimethyl-(β -amino-ethoxy)-silane and chloro-methyl pentamethyl disiloxane was studied. In this reaction (8 hrs at 110 - 120°C), one hydrogen atom only was substituted and 2,2,4,4,10,10-hexamethyl-6-aza-3,9-dioxa-2,4,10-trisila-undecane was separated. Treatment of the reaction products with aqueous alkali yielded 4,4-tetramethyl-6-aza-3-oxa-2,4-disiloxane-8-ol. The interaction

Card 1/2

33978

S/062/62/000/002/003/013

B117/B138

15. P150

AUTHORS: Andrianov, K. A., Pichkhadze, Sh. V., and Komarova, V. V.

TITLE: Synthesis of polychelate titanosiloxane elastomers by the polycondensation method

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 2, 1962, 261-264

TEXT: Polychelate titanosiloxane elastomers were synthesized by polycondensation of organosilicon diols with monomers containing titanium. The monomers used were bis-(acetyl acetate)dibutoxy titanium (I) and bis-(8-oxyquinoline)dibutoxy titanium (II). Organosilicon diols were obtained by the method described in Ref. 3 (K. A. Andrianov and V. V. Severnyy, Dokl. AN SSSR 134, 1347 (1960)). The polycondensation took place in pure nitrogen atmosphere at 180°C and a residual pressure from 1 to 2 mm; the separating butyl alcohol was distilled off at the same time. Soluble red-brown elastic polymers were produced: poly-bis-(8-oxyquinoline)titanodimethyl siloxane elastomer ($C_{138}H_{372}O_{63}Si_{60}TiN_2$)_n (molecular weight 97,000, vitrification temperature at -110°C) and

Card 1/2

33978

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B117/B138

Synthesis of polychelate...

poly-bis-(acetyl acetate) titanodimethyl siloxane elastomer
($C_{130}H_{374}O_{65}Si_{60}Ti_n$) (molecular weight 58,000, vitrification temperature at $-75^{\circ}C$). The thermomechanical properties were examined by the method described in Ref. 4 (B. L. Tsetlin, V. I. Gavrilov, N. A. Velikovskaya, and V. V. Kochkin, *Zavodsk. laboratoriya* 22, 352 (1956)). The vitrification temperature was found to be influenced by the groups surrounding the titanium atom. Molecular weights were determined by the light diffusion method. There are 2 figures and 6 references: 5 Soviet and 1 non-Soviet. The reference to the English-language publication reads as follows: A. Yamamoto, S. Kambara, *J. Amer. Chem. Soc.* 79, 4344 (1957).

ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk SSSR (Institute of Elemental Organic Compounds of the Academy of Sciences USSR)

SUBMITTED: September 23, 1961

Card 2/2

33979

S/062/62/000/002/004/013
B117/B138

11.9.200
15.8.170

AUTHORS: Andrianov, K. A., and Volkova, Lora M.

TITLE: Synthesis of dimethyl cyclosiloxanes with functional groups at the silicon atom

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 2, 1962, 264-269

TEXT: Cyclic dimethyl siloxane compounds with functional groups at the silicon atom were synthesized. Highly reactive groups such as chloro, alkoxy, phenoxy, and amino groups may serve as functional groups in the conversion of polymers into different materials. The exchange reaction between 1,5-sodium oxyhexamethyl trisiloxane and methyl trichloro silane in the presence of excess methyl trisiloxane showed that the reaction could be influenced to yield low-molecular cyclic compounds. Heptamethyl chlorocyclotetrasiloxane ($C_7H_{21}O_4SiCl$, boiling point at 79-81°C (14 mm Hg); yield ~30%), and other compounds not distillable in vacuum were obtained in this manner. The substitution of methoxy and phenoxy groups for

Card 1/1

3

33979

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B117/B138

Synthesis of dimethyl cyclosiloxanes...

chlorine in heptamethyl chlorocyclotetrasiloxane is accompanied by secondary processes. In all cases polymeric substances, not distillable in vacuum, are formed besides heptamethyl methoxycyclotetrasiloxane (I) and heptamethyl phenoxy cyclotetrasiloxane (II). [Abstracter's note: Roman numerals refer to the table.] The substitution of amino and phenyl-amino groups for chlorine in heptamethyl chlorocyclotetrasiloxane has a smooth course and shows that cyclic compounds with different functional groups at the silicon atom can be obtained by this reaction. Heptamethyl amino cyclotetrasiloxane (v) (yield 60.4 %) and heptamethyl phenyl amino cyclotetrasiloxane (VI) (yield ~40 %) were synthesized in this manner. The joint hydrolysis of dimethyl dichloro silane with methyl butoxy dichloro silane and methyl ethoxy dichloro silane yielded, correspondingly, hexamethyl dibutoxy cyclotetrasiloxane (IV) and hexamethyl diethoxy cyclotetrasiloxane (III). All synthesized compounds are colorless, transparent liquids, well soluble in benzene, toluene, sulfuric ether, and acetone. Their structure was determined by both IR-spectra and ultimate analysis. Physical properties are indicated in the table. L. Tartakovskaya, a student at the Institute, who participated in the experimental work, is mentioned. There are 1 figure, 1 table, and

Card 2/4

3

33979

Synthesis of dimethyl cyclosiloxanes...

S/062/62/000/002/004/013
B117/B138

3 Soviet references.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.
M. V. Lomonosova (Moscow Institute of Fine Chemical
Technology imeni M. V. Lomonosov)

SUBMITTED: July 18, 1961

Table. Physical properties of synthesized compounds. K

Legend: (1) cyclosiloxane; (2) formula; (3) boiling point, °C (p, mm Hg);
(4) found; (5) calculated; (I) heptamethyl methoxy cyclotetrasiloxane;
(II) heptamethyl phenoxy cyclotetrasiloxane; (III) hexamethyl diethoxy
cyclotetrasiloxane; (IV) hexamethyl dibutoxy cylcotetrasiloxane; (V)
heptamethyl amino cyclotetrasiloxane; (VI) heptamethyl phenyl amino
cyclotetrasiloxane. +) Position of alkoxy groups not established.

Card 3/18 *g*

33980

S/O62/62/000/002/005/013
B117/B138

5.3700 11.9200

11.2219
AUTHORS:

Semenova, Ye. A., Zhinkin, D. Ya., and Andrianov, K. A.

TITLE:

Synthesis of alkyl hydride cyclosilazanes

PERIODICAL:

Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh
nauk, no. 2, 1962, 269-271

TEXT: Pure alkyl hydride cyclosilazanes were synthesized on the base of methyl and ethyl dichloro silane. Method applied: A determined amount of dry ammonia was sent through a solution of suitable alkyl hydride dichloro silane in benzene (15-20°C; 1.5 l/min, 2 hr). Ammonium chloride was then filtered off from the reaction mass and benzene was distilled off. At a residual pressure of 10 mm the resulting mixture was distilled off from the Claisen flask and the distillate was decomposed in a rectifying column with 15 or 17 trays. The ammonolysis of ethyl dichloro silane yielded triethyl cyclotrisilazane ($C_6H_{21}Si_3N_3$, boiling point 62°C, 0.5 mm Hg; n_D^{20} 1.4700; d_4^{20} 0.9596; molecular weight 215 (219); MR 63.55 (63.63)) and tetraethyl cyclotetrasilazane ($C_8H_{28}Si_4N_4$, boiling point 102.0°C,

Card 1/2

33980

S/062/62/000/002/005/013
B117/B138

Synthesis of alkyl hydride...

0.5 mm Hg; n_D^{20} 1.4810; d_4^{20} 0.9767; molecular weight 290.4 (292);
MR 84.97 (84.84)). The residue from rectification was a viscous product
(n_D^{20} 1.5000; molecular weight 620). The total yield of ethyl hydride
cyclosilazanes was 68 %. The ammonolysis of methyl dichloro silane yielded
(total yield 45-49 %): tetramethyl cyclotetrasilazane ($C_4H_{20}Si_4N_4$,
boiling point 54°C (1 mm Hg); n_D^{20} 1.4780; d_4^{20} 1.0069; molecular weight 234 ✓
(236); MR 66.34 (66.32)), and a polymer consisting of condensed rings
(($CH_3SiH_2N_2(NH)_3$), boiling point 116°C (1.5 mm Hg); n_D^{20} 1.4860;
 d_4^{20} 1.0371; molecular weight 325 (337); MR 93.36 (93.66)). The residue
was a polymer of molecular weight 1261, n_D^{20} 1.5020. There are 1 table
and 4 non-Soviet references. The two references to English-language
publications read as follows: S. D. Brewer, Ch. P. Haber, J. Amer. Chem.
Soc. 70, 3888 (1948). US Patent 2885, 370, May 5, 1959.

SUBMITTED: August 15, 1961
Card 2/2

Investigation of the heat...

S/062/62/000/003/007/014
B117/B144

resistance was investigated on three types of polymers which were synthesized according to the method described previously by K. P. Andrianov, and A. Zhdanov (Ref. 1: Dokl. AN SSSR 114, 1005 (1957)). The polyorgano siloxanes, polyorganophenylene siloxanes and polyelementorgano siloxanes used contained 2-4 % terminal hydroxyl groups. On the basis of the experimentally determined data for polyorgano siloxanes and polyorganophenylene siloxanes it could be assumed that endothermal effects observed at relatively low temperatures may be traced to reactions produced in the chains by hydroxyl groups. They take place at 70-133°C for polymethylphenylphenylene siloxane and at 89 and 133-153°C for polyphenylphenylene siloxane. The destruction of the main chains of molecules is very distinct at 540°C, thus at a higher temperature than in the case of a linear polymer, polymethylphenylsiloxane. From the thermograms of polyorganometallo siloxanes it results that the weight changes most intensively in the temperature range from ~300-600°C. It was found that the heat resistance of the polymer may be increased by introducing metal into the main chain of the macromolecule or by substituting the oxygen in the main chain by a phenyl group. The following decomposition temperatures could be ascertained: for polymethyltin siloxane 480°C, for polyethyltin siloxane

Card 2/3

Investigation of the heat...

S/062/62/000/003/007/014
B117/B144

462°C and polyphenyltin siloxane 460°C. There are 8 figures and 1 Soviet reference.

ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk SSSR (Institute of Elemental Organic Compounds of the Academy of Sciences USSR). Institut obshchey i neorganicheskoy khimii im. N. S. Kurnakova Akademii nauk SSSR (Institute of General and Inorganic Chemistry imeni N. S. Kurnakov of the Academy of Sciences USSR)

SUBMITTED: October 24, 1961

X

Card 3/3

35592
S/062/62/000/003/008/014
B117/B144

15.8170
AUTHORS: Andrianov, K. A., Zhdanov, A. A., and Kashutina, E. A.
TITLE: Synthesis of polyorganosiloxane-oligomers with terminal malonate groups
PERIODICAL: . Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 3, 1962, 454-456

TEXT: The joint hydrolysis of dimethyl-diethoxy silane and (diethyl-malonylmethyl)dimethyl-ethoxy silane was studied. Dimethylchloromethyl-ethoxy silane synthesized by the method described by K. A. Andrianov and M. A. Golubenko (Ref. 4: Dokl. AN SSSR 112, 257 (1957)), boiling point 131-133°C, dimethyl-diethoxy silane, boiling point 112-116°C and freshly distilled malonic ester were used. The reaction was shown to take a smooth course in slightly acid medium without cleavage of ethoxy groups in malonic ester rests. Polyorganosiloxane-oligomers with terminal malonate groups are formed here. Investigation of infrared spectra of the compounds produced showed that the malonic ester residue is not added to the oxygen atom, but to the carbon atom. The following compounds were synthesized: X

Card 1/3

S/062/62/000/003/008/014
B117/B144

Synthesis of polyorganosiloxane-oligomers...

(diethylmalonylmethyl) dimethylethoxysilane, $C_{10}H_{19}O_4Si$, yield 42 %, clear, colorless liquid, boiling point 120-123°C (5 mm Hg), n_D^{20} 1.4312, d_4^{20} 1.0058, MR 71.29; 1,3-bis-(diethylmalonylmethyl)tetramethylsiloxane, $C_{20}H_{38}O_9Si_2$, yield 78.4 %, clear, colorless liquid, boiling point 184-188°C (2 mm Hg), n_D^{20} 1.4425, d_4^{20} 1.0588, MR 119.94; 1,5-bis-(diethylmalonylmethyl) hexamethyltrisiloxane, $C_{22}H_{44}O_{10}Si_3$, heavy, colorless liquid, boiling point 232-234°C (7-8 mm Hg), n_D^{20} 1.4363, d_4^{20} 1.0425, MR 138.93; 1,7-bis-(diethylmalonylmethyl) octamethyltetrasiloxane, $C_{24}H_{50}O_{11}Si_4$, clear, colorless liquid, boiling point 242-255°C (8-10 mm Hg), n_D^{20} 1.4338, d_4^{20} 1.0375, MR 157.56; 1,17-bis-(diethylmalonylmethyl) octadecamethylnonasiloxane, $C_{34}H_{80}O_{16}Si_9$, n_D^{20} 1.4250, d_4^{20} 1.0190, MR 250.66; 1,37-bis-(diethylmalonylmethyl) octatricosamethylnonadecasiloxane, $C_{54}H_{140}O_{26}Si_{19}$, clear, viscous liquid, n_D^{20} 1.4165, d_4^{20} 1.0068, MR 434.48. N. S. Leznov and

Card 2/3

35593
S/062/62/000/003/009/014
B117/B144

15.8170

AUTHORS: Andrianov, K. A., and Kuznetsova, I. K.

TITLE: Substitution of chlorine in α -chloromethyl-methylalkoxy silanes by residues of diethyl-, dibutyl-dithiophosphoric- and diphenyl-dithiophosphinic acids

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 3, 1962, 456-460

TEXT: The substitution of chlorine in α -chloromethyl-methylethoxy silanes by residues of dialkyl-dithiophosphoric- and diphenyl-dithiophosphinic acids was studied. Heating of α -chloromethyldimethylethoxysilane with potassium salt of diethyl-dithiophosphoric acid for 8-10 hrs at 130-135°C produced only a 30 % yield of diethyldithiophosphoric dimethylethoxysilyl-methyl ester (I). It was possible to increase its yield to 94 % after 3-4 hrs by adding catalytic amounts of diethyl aniline. Compound (II) was obtained in a similar way with a yield of 80 %. Reactions of potassium salts of dibutyl-dithiophosphoric- and diphenyl-dithiophosphinic acids with α -chloromethyl-dimethylethoxy silane and α -chloromethyl-

Card 1/3

Substitution of chlorine in...

S/062/62/000/003/009/014
B117/B144

methyldiethoxy silane in the presence of diethyl aniline also produced almost quantitative yields (90 %) of dimethylethoxysilyl- and methyl-diethoxysilylmethyl esters of the corresponding acids (III), (IV), (V) and (VI). Tetramethylsiloxy-1,3-disilylmethyl ester of diethyl-dithio-phosphoric acid (VII), of dibutyl-dithiophosphoric acid (VIII) (yield 65 %) and of diphenyl-dithiophosphinic acid (IX) (yield 85 %) were obtained from reactions with 1,3-bis-chloromethyltetramethyl disiloxane in the presence of catalytic diethyl aniline amounts. The catalytic effect of diethyl aniline is probably connected with its participating in the formation of a transition complex with α -chloromethyl-methylethoxy silanes. Owing to ionization of the C - Cl bond, this favors the mobility of chlorine in the chloromethyl group. The properties of the products obtained are listed in a table. There are 1 table and 1 Soviet reference.

ASSOCIATION: Institut elementoorganicheskikh soedineniy Akademii nauk .
SSSR (Institute of Elemental Organic Compounds of the
Academy of Sciences USSR)

SUBMITTED: October 16, 1961

Card 2/3

Substitution of chlorine in...

S/062/62/000/003/009/014
B117/B144

Legend to the Table:

(1) Number of the compound;
(2) Formula;
(3) Boiling point °C, (p mm Hg); (4) Molecular refraction; (5) found; (6) calculated;
*) Temperatures of the bath.

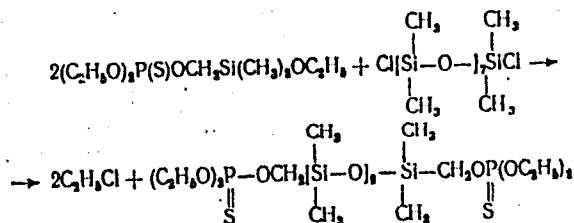
Card 3/3

Номер соединения	Формула (L)	Т. кип. °C (р. мм рт. ст.) (3)	n _D ²⁰	d ₄ ²⁰	Молекулярная рефракция (4)	
					найденная	вычисленная
I	$(CH_3)_2(C_2H_5O)Si - CH_2SP(S)(OC_2H_5)_2$	126 (1)	1,4863	1,0715	81,1	81,36
II	$CH_3(C_2H_5O)_2SiCH_2SP(S)(OC_2H_5)_2$	159-160 (6)	1,4815	1,0919	86,76	86,75
III	$(CH_3)_2(C_2H_5O) - SiCH_2SP(S)(OC_4H_9-n)_2$	166-168 (2-3)	1,4821	1,0267	99,82	99,81
IV	$CH_3(C_2H_5O)_2SiCH_2SP(S)(OC_4H_9-n)_2$	170-171 (2)	1,4770	0,0514	104,9	105,3
V	$(CH_3)_2(C_2H_5O)SiCH_2 - S - P(S)(C_6H_5)_2$	210-220 (1-10°)*	1,6056	1,1481	110,1	109,4
VI	$CH_3(C_2H_5O)_2SiCH_2SP(S)(C_6H_5)_2$	230-235 (1-10°)*	1,5848	1,1545	115,1	114,82
VII	$(CH_3)_2 - Si - O - Si - (CH_3)_2$ CH ₃ CH ₃	100 (1-10°)	1,4915	1,1134	138,4	139,2
VIII	$(C_2H_5O)P(S) - S - P(S)(OC_2H_5)_2$ (CH ₃) ₂ - Si - O - Si (CH ₃) ₂ CH ₃ CH ₃ S S	200-204 (1-10°)*	1,4996	1,0753	176,1	176,42
IX	$2(n-C_4H_9O)P(S) - P(S)(OC_4H_9-n)_2$ (CH ₃) ₂ - Si - O - Si - (CH ₃) ₂	T. пл. 126-127	-	-	-	-
	$(C_6H_5)_2(S)PSCCH_3 - CH_2 - S - P(S)(C_6H_5)_2$					

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B110/B138

Organosilicon compounds...

A liquid ($d_4^{20} = 1.0581$, $n_D^{20} = 1.4450$) boiling in vacuum (89°C , 15 mm Hg) without decomposition was obtained in good yield (52 %) in alcoholic medium. Condensation with α, ω -dichloro polydimethyl siloxanes takes place according to



where $\omega = 4, 5, 6$, or 7 . The most important English-language reference reads as follows: A. E. Canavan, C. Eaborn, J. Chem. Soc., no. 12, 3751 (1959).

Card 2/2

X

36635
S/062/62/000/004/004/013
B110/B101

15.9205

AUTHORS: Andrianov, K. A., and Zhdanov, A. A.

TITLE: Polyboron dimethyl siloxanes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye
khimicheskikh nauk, no. 4, 1962, 615-619

TEXT: Polyboron dimethyl siloxanes, which are of interest as elastomers (molecular weight 200,000-400,000), were produced by heterofunctional polycondensation as described by the authors (Dokl. AN SSSR 138, 361 (1961)). In the first stage, 326 g (1.1 moles) of octamethyl cyclotetrasiloxane was telomerized in the presence of 2.37 g (0.0184 moles) of dimethyl dichlorosilane in an autoclave for 4 hrs at 200°C. The resultant substance, 79 g of colorless, viscous α,ω -dichloro polydimethyl siloxane $(\text{ClSi}(\text{CH}_3)_2\text{O}[\text{Si}(\text{CH}_3)_2\text{O}]_{70}\text{Si}(\text{CH}_3)_2\text{Cl})$ was hydrolyzed in acetone and benzene by means of sodium bicarbonate, whereby 72 g of colorless, viscous α,ω -dioxy polydimethyl siloxane, $[(\text{CH}_3)_2\text{SiO}]_m$, was obtained. The molecular weight of the compound was calculated from

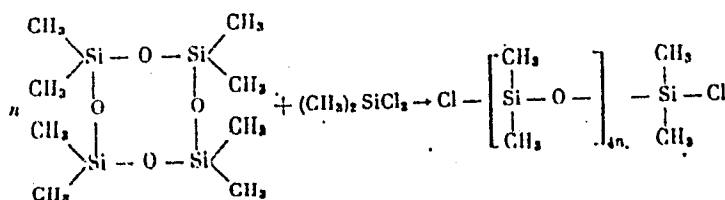
Card 1/4

X

S/062/62/000/004/004/013
B110/B101

Polyboron dimethyl siloxanes

$(\eta) = 2.15 \cdot 10^{-4} M^{0.65}$. In the second stage, 100 g of α, ω -dioxo polydimethyl siloxane (molecular weight 3300, specific viscosity of the 8% solution: 0.413) and 6.97 g of dibutoxy boron were polycondensed to a gel-like polymer, $[Si(CH_3)_2O]_{44}B(OC_4H_9)_2O$. The complete synthesis can be represented as follows:

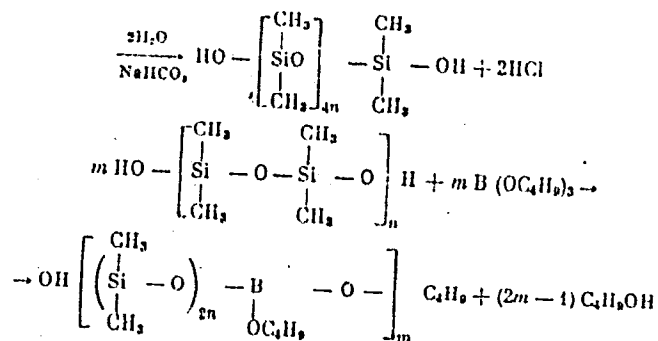


X

Card 2/5

Polyboron dimethyl siloxanes

S/062/62/000/004/004/013
B110/B101



In the two-stage process, the degree of polymerization of the oligomeric intermediate product is already so high as to render the formation of low-molecular cycles impossible. On heating, tributoxy boron reacts with the oligomer, whereas in the cold it reacts only by

Card 3/5

Polyboron dimethyl siloxanes

S/062/62/000/004/004/013
B110/B101

association. The characteristic viscosity of the reaction mixture of α,ω -dioxy polydimethyl siloxane and tributoxy boron does not increase at 20°C at ratios of 1:1 or 1:2. Measurements of viscosity showed that the removal of the resulting butanol with carbon dioxide accelerated the reaction. After 30-min heating at 200°C, the specific viscosity of the 8% solution increased to 0.946, butanol being obtained in a quantity of 43% of the theoretical amount. The gel-like, polymeric substance, which is soluble in polar and apolar solvents, was also formed after 30-40 min heating at 150°C in vacuo. Its vitrification temperature (-125°C) and yield point (-50°C) were established by thermomechanical investigations. It is elastic on sudden deformation, capable of springbacks on shocks, but flows under slow stress as in the case of polyaluminosiloxanes, coordination bonds are assumed to exist between oxygen and boron atoms in the siloxane chains:

Card 4/5

356L

S/062/62/000/004/010/013
B110/B101K-4170
AUTHORS:Andrianov, K. A., Pichkhadze, Sh. V., and Komarova, V. V.

TITLE:

Reactions of dimethyl cyclosiloxanes with butyl orthotitanate

PERIODICAL:

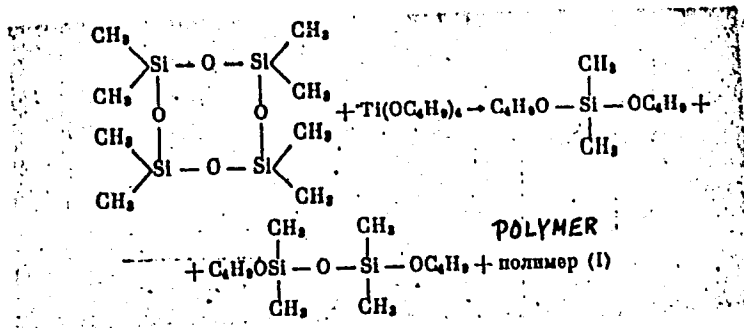
Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 4, 1962, 724-725

TEXT: In continuation of earlier papers by the authors (Vysokomolek. soyed. **3**, 577 (1961), *ibid.* **3**, 1321 (1961)) on the synthesis of polyorganotitanosiloxanes, the reaction of butyl orthotitanate with dimethyl cyclosiloxanes was investigated. It was established that the siloxane bond was split and that low-molecular organo-silicon compounds formed. 4 hrs' heating at 200°C of octamethyl cyclotetrasiloxane with butyl orthotitanate in a 1:1 molar ratio gives dimethyl dibutoxy silane (b.p. 186°C, $n_D^{20} = 1.4034$) and the hardly accessible 1,3-dibutoxy tetramethyl disiloxane ($C_{12}H_{30}O_3Si_2$, b. p. 98-100°C (10 mm Hg), $n_D^{20} = 1.4045$, $d_4^{20} = 0.866$):

Card 1/3

Reactions of dimethyl cyclosiloxanes ...

S/062/62/000/004/010/013
B110/B101



Hexamethyl cyclotrisiloxane with butyl orthotitanate (molar ratio 1:1), heated for 10 hrs at 180°C, gives dimethyl dibutoxy silane:

37402

S/062/62/000/005/004/008

B110/B101

5.3700
AUTHORS: Andrianov, K. A., Pichkhadze, Sh. V., Komarova, V. V., and Vardosanidze, Ts. N.

TITLE: The reaction of organocyclosiloxanes with butyl orthotitanate

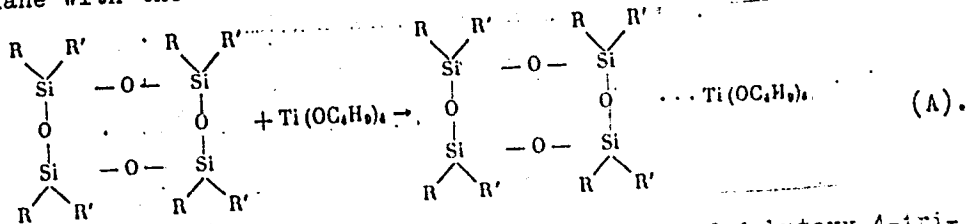
PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 5, 1962, 833 - 837

TEXT: The reaction of butyl orthotitanate with octamethyl cyclotetra-siloxane, tetramethyl tetravinyl cyclotetrasiloxane, and octaethyl cyclotetrasiloxane were examined. In the reaction of octamethyl cyclotetrasiloxane with butyl orthotitanate (5:1, 3:1), only two molecules of the cycle react with one molecule of butyl orthotitanate to form the following products: dimethyl dibutoxysilane ($n_D^{20} = 1.4055$), 1,3-dibutoxytetramethyl disiloxane ($d_4^{20} = 0.8700$; $n_D^{20} = 1.4040$), 1,5-dibutoxyhexamethyl trisiloxane (b.p. $96^\circ\text{C}/4$ mm Hg; $n_D^{20} = 1.4031$; $d_4^{20} = 0.8960$), 1,7-dibutoxyoctamethyl tetrasiloxane (b.p. $118^\circ\text{C}/4$ mm Hg; $n_D^{20} = 1.4049$; $d_4^{20} = 0.9060$), and a
Card 1/4

S/062/62/000/005/004/008
B110/B101

The reaction of organocyclosiloxanes ...

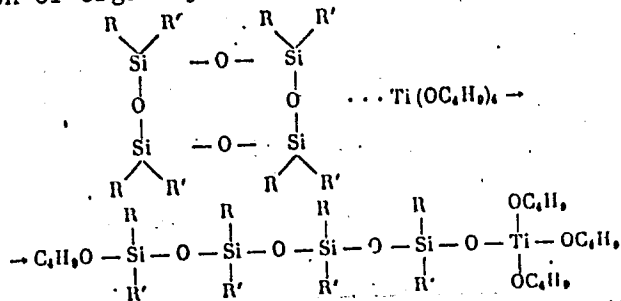
polymer of a chemically constant composition and the atomic ratio Si:Ti = 1:1. At 3:1 and 5:1 ratios of the initial components almost equal yields were obtained; however, at a 5:1 ratio, the part of non-reacting octamethyl cyclotetrasiloxane rose. At a 1:1 ratio, the yield of dimethyl dibutoxysilane, 1,3-dibutoxytetramethyl disiloxane, and of the polymer rose significantly. The polymers were readily soluble in benzene and toluene and had a vitrification temperature of ~100°C. The formation of ethers and polymers is explained by disproportionation of the initial reaction products. Coordination of one of the oxygen atoms of organocyclosiloxane with the titanium atom of butyl orthotitanate takes place first:



The SiO bond in the cycle is thereby weakened, opens, and 1-butoxy-4-tri-butoxyoctaalkyl tetrasiloxane is formed:
Card 2/4

S/062/62/000/005/004/008
B110/B101

The reaction of organocyclosiloxanes ...



(B).

The latter is disproportionated to dialkyl dibutoxysilane, 1,3-dibutoxy-tetraalkyl disiloxane, and the polymer. The following succession holds for the reactivity of organosiloxanes with butyl orthotitanate:
 $[(CH_3)_2SiO]_3 \gg [(CH_3)_2SiO]_4 > [CH_3(CH_2=CH)SiO]_4 > [(C_2H_5)_2SiO]_4$. The large organic radicals are steric hindrances making the reaction difficult. Disiloxane ethers are easy to prepare in the manner described. There are 2 figures and 4 tables.

Card 3/4

The reaction of organocyclosiloxanes ...

S/062/62/000/005/004/008
B110/B101

ASSOCIATION: Institut elementoorganicheskikh sovedineniy Akademii nauk
SSSR (Institute of Elemental Organic Compounds of the Academy
of Sciences USSR)

SUBMITTED: December 3, 1961

Card 4/4

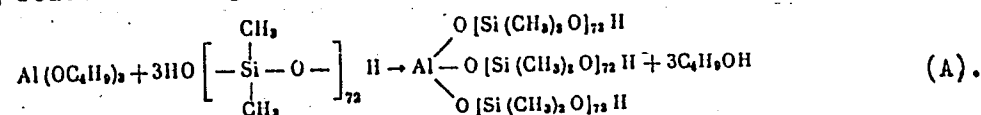
37403

S/062/62/000/005/005/008
B110/B1015.3700
15.9205AUTHORS: Andrianov, K. A., and Zhdanov, A. A.

TITLE: Synthesis of polyaluminosiloxanes with molecules of a periodic network structure

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 5, 1962, 837 - 840

TEXT: The polycondensation of α,ω -dioxypolydimethyl siloxanes (K. A. Andrianov et al. (Dokl. AN SSSR 134, 1347 (1961))) with aluminum butylate was investigated. When the components were in a proportion 3:1, the following reaction took place at 200°C:

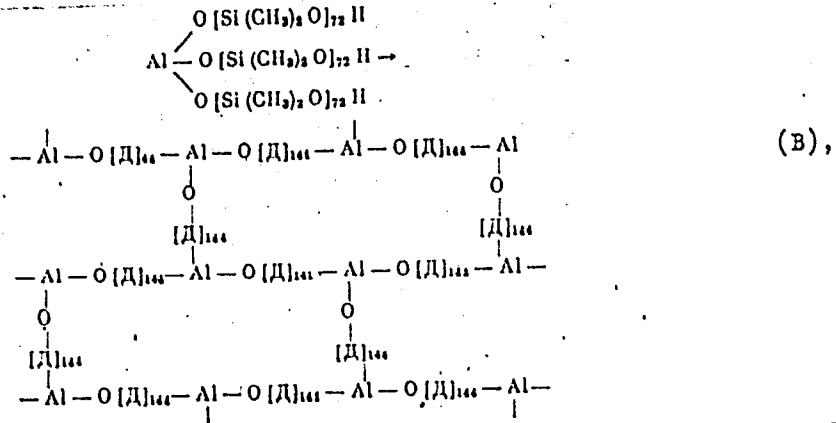


In the first stage of polycondensation, the intrinsic viscosity changed very slowly, increasing from 0.052 to 0.092 within 48 hrs. This indicates the formation of a branched oligomer with a central aluminum atom, whereby
Card 1/4

Synthesis of polyaluminosiloxanes...

S/062/62/000/005/005/008
B110/B101

viscosity is only slightly increased. A polymer with a periodic network structure, $C_{148}H_{441}O_{74}Si_{72}Al$, is obtained by further heating:

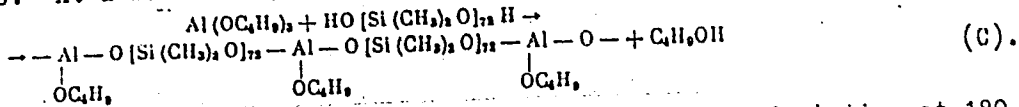


where $[D] = (CH_3)_2 SiO$. This assumption is confirmed by the substantial
Card 2/4

Synthesis of polyaluminosiloxanes ...

S/062/62/000/005/005/008
B110/B101

increase in viscosity from 0.092 to 0.263 during polycondensation for 32 hrs. At a ratio 1:1, linear polycondensation takes place:



This results from the change in viscosity during polycondensation at 180 and 200°C. At 200°C, viscosity rises from 0.092 to 0.164 after polycondensation for 14 hrs, but at a ratio 3:1 this value is only reached after 72 hrs. At room temperature, the 3:1 polymer with an intrinsic viscosity of 0.263 is very elastic, soluble in benzene, toluene, and ether, but becomes insoluble when kept on at a temperature of 200°C. It is elastic over a wide range and has a vitrification temperature of ~-60°C. The high elasticity of its periodic network structure is due to the large spacing of the Al atoms and the considerable flexibility of the polydimethyl siloxane chains. At room temperature, the linear molecule is elastic, readily soluble in organic solvents, and has a wider range of elasticity than the network polymer. There are 3 figures.

Card 3/4

Synthesis of polyaluminorganosiloxanes ...

S/062/62/000/005/005/008
B110/B101

ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk
SSSR (Institute of Elemental Organic Compounds of the Academy
of Sciences USSR)

SUBMITTED: December 19, 1961

Card 4/4

38065

S/191/62/000/006/006/016

B110/B138

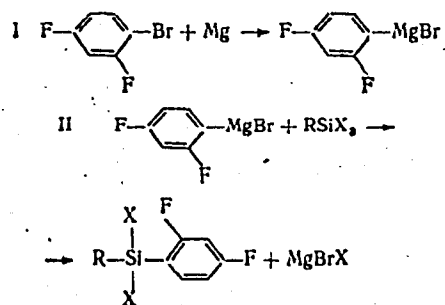
15.8150

AUTHORS: Shashkova, Z. S., Grinevich, K. P., Andrianov, K. A.

TITLE: Reactions of fluorophenyl magnesium bromides with alkyl chlorosilanes and alkyl ethoxysilanes

PERIODICAL: *Plasticheskiye massy*, no. 6, 1962, 18-19

TEXT: The reaction takes place as follows



Card 1/3

Reactions of fluorophenyl ...

S/191/62/000/006/006/016
B110/B138

reagent. The 1113 and 1159 cm^{-1} bands of $(p\text{-FC}_6\text{H}_4)\text{Si}(\text{CH}_3)(\text{OC}_2\text{H}_5)_2$ indicate a para-substituted group in the fluorophenyl radical, and with $(\text{F}_2\text{C}_6\text{H}_3)\text{Si}(\text{CH}_3)(\text{OC}_2\text{H}_5)_2$ the 1000-1200 cm^{-1} bands indicate an asymmetrical, trisubstituted radical. There is 1 table.

Card 3/3

38066

S/191/62/000/006/007/016

B110/B138

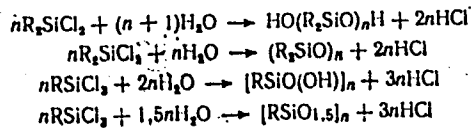
15.8170

AUTHORS: Kuznetsova, A. G., Andrianov, K. A., Zhinkin, D. Ya.

TITLE: Investigation of the hydrolytic condensation of diethyl dichlorosilane and phenyl trichlorosilane

PERIODICAL: *Plasticheskiye massy*, no. 6, 1962, 19-22

TEXT: The composition of hydrolytic condensation products of equimolecular quantities of diethyl dichlorosilane and phenyl trichlorosilane at organochlorosilane/water ratios of 1 : 1 : 2.5 and 1 : 1 : 1.25 was studied. The products formed are:



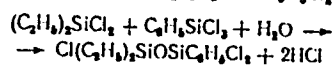
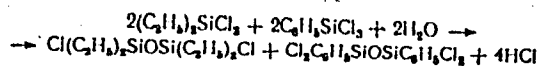
Polyorganosiloxanes and non-reactive diorganodichlorosilane are likewise

Card 1/4

Investigation of the ...

S/191/62/000/006/007/016
B110/B138

formed at ratios of water to hydrolyzable Cl < 0.5 : 1. The composition of the hydrolytic condensation products of dimethyl dichlorosilane, diphenyl dichlorosilane, and methyl phenyl dichlorosilane was investigated in a homogeneous solution with an insufficient quantity of water. The ratio of the components in the hydrolytic condensation of 5 moles/l solutions of phenyl trichlorosilane and diethyl dichlorosilane in dioxane (1 : 1 : 1.25) at 0-5°C was calculated by



Eight fractions containing 20.61-27.38% Si and 51.68-54.56% C were separated in yields of 0.8-21.8% by fractional distillation between 33 and 220°C. For the fractions between 74 and 185°C about 10% 1,3-diphenyl-1,1,3-tetrachlorodisiloxane was present, and in the high-boiling fractions (Si content, 22.0-22.04%) the ratio of diethylsiloxy groups to phenylsiloxy groups was ~0.3 : 1. In the hydrolytic condensation of homogeneous solutions with an insufficient quantity of water, the hydrolysis of

Card 2/4

Investigation of the ...

S/191/62/000/006/007/016
B110/B138

phenyl trichlorosilane occurs more rapidly than that of diethyl dichlorosilane. Non-reactive diethyl dichlorosilane and copolymers with a high content of phenylsiloxy groups are thus formed. In the hydrolytic condensation of 6.5 M solutions of diethyl dichlorosilane and phenyl trichlorosilane in dioxane (1 : 1 : 2.5), at $\sim 25^\circ\text{C}$, nine fractions were obtained with a content of polymer between 3.7 and 20.5%, of Si between 22.62 and 25.44%, and of OH between 1.56 and 3.03%, and with molecular weights between 834 and 1215. This proves the formation of copolymers with a variable ratio between diethylsiloxy and phenylsiloxy groups. The absence of a continuous decrease of the molecular weights is due to the varying solubility of the polymers, which depends not only on the molecular weight but also on the chemical composition. The content of OH groups indicates the presence of $\text{C}_6\text{H}_5\text{SiO}_{1.5}$ and $\text{C}_6\text{H}_5(\text{OH})\text{SiO}$ groups. For the ratio 1 : 1 : 2.5 the condensation products are:

$[(\text{C}_2\text{H}_5)_2\text{SiO}]_x [\text{C}_6\text{H}_5\text{SiO}_{1.5}]_y [\text{C}_6\text{H}_5\text{SiO}(\text{OH})]_z$, where $x + y + z$ is the number of structural units in the copolymer molecule; $x : (y + z)$ is the ratio of diethylsiloxy to phenylsiloxy groups in the copolymer molecule; $z : y$ is the ratio of phenylsiloxy to phenyl-(hydroxy)-siloxy groups

Card 3/4

S/062/62/000/006/004/008
B117/B101

AUTHORS: Morozova, L. P., Golubtsov, S. A., Andrianov, K. A., Trofimova, I. V., and Morozov, N. G.

TITLE: Formation of alkyl (aryl) chlorosilanes in direct reaction of alkyl (aryl) chlorides with silicon. Communication 1. Selectivity of silicon and copper catalysts, and formation of methyl dichlorosilane

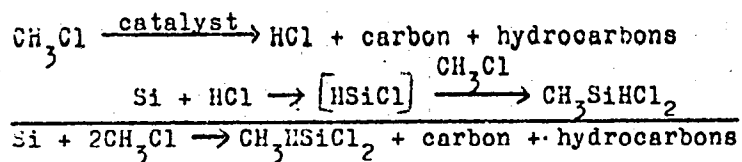
PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 6, 1962, 1005 - 1011

TEXT: Production conditions, precipitating agents, and promoters affecting the selective activity of silicon and copper hydroxide catalysts in the formation of methyl dichlorosilane and dimethyl dichlorosilane were studied. Sufficiently active catalysts can be obtained by using copper chloride and copper nitrate, but copper sulfate gives completely passive catalysts. Simultaneous precipitation of copper hydroxide and zinc hydroxide (~2% by weight) increases the selectivity of the catalyst. Sodium hydroxide (in the formation of methyl dichlorosilane) and NH_4OH or Na_2CO_3 (in the forma-
Card 1/3

Formation of alkyl ...

S/062/62/000/006/004/008
B117/B101

tion of dimethyl dichlorosilane) were found to be precipitating agents favoring the selectivity. The greatest effect on the selectivity of the catalyst is that exercised by promoters after the precipitation of hydroxides: Thus after introducing $ZnCl_2$ the yield of dimethyl dichlorosilane reaches 60% and after introducing Na_2SiO_3 that of methyl dichlorosilane reaches 45%. Thermal decomposition of methyl chloride on copper catalysts at 360-380°C (contact time 6-10 sec) was also studied. The hydrogen chloride separated in this reaction considerably affected the formation of methyl dichlorosilane. The following reaction course was suggested for the formation of methyl dichlorosilane:



The optimum temperature for synthesizing methyl dichlorosilane was found to be 350-380°C. At higher and lower temperatures, silicon tetrachloride,

Card 2/3

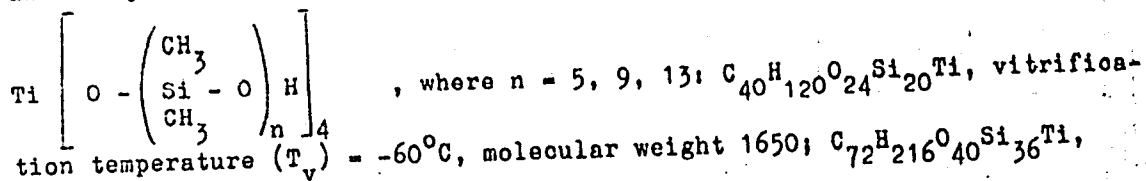
S/062/62/000/006/005/008
B117/B101

AUTHORS: Andrianov, K. A., and Kurasheva, N. A.

TITLE: Synthesis of cruciform titanodimethyl siloxane oligomers

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 6, 1962, 1011 - 1014

TEXT: Tetrafunctional, cruciform titanodimethyl siloxane oligomers with hydroxyl groups at the ends of the branchings were synthesized by the action of titanium tetrachloride on α, ω -dihydroxy dimethyl siloxanes. The reaction conducted at $>35^{\circ}\text{C}$ in C_6H_6 and in the presence of diethyl aniline yielded oligomers having the common formula :



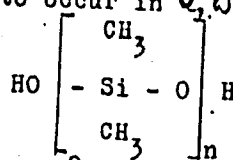
Card 1/2

30

Synthesis of cruciform...

S/062/62/000/006/005/008
B117/B101

$T_v = -110^\circ\text{C}$, molecular weight 3000; $\text{C}_{104}\text{H}_{312}\text{O}_{56}\text{Si}_{52}\text{Ti}$, $T_v = -120^\circ\text{C}$,
molecular weight 3540. Thus; increasing content of OH group lessens the
extent that T_v is reduced by increasing molecular weight. A similar in-
fluence of the OH groups on the T_v was found to occur in α, ω -dihydroxy
dimethyl siloxanes having the common formula



where $n = 9, 50, 150$; $T_v = -105^\circ\text{C}$ at $n = 9$; $T_v = -137^\circ\text{C}$ at $n = 50$; $T_v = -132^\circ\text{C}$
at $n = 150$. Conclusion: The association of molecules is greatly affected
by the hydroxyl groups at the ends of the branchings, probably owing to for-
mation of hydrogen bonds. There are 2 tables.

ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk SSSR
(Institute of Elemental Organic Compounds of the Academy of
Sciences USSR)
SUBMITTED: December 19, 1961
Card 2/2

S/062/62/000/006/006/008
B117/B101

AUTHORS: Tsitsishvili, G. V., Bagratishvili, G. D., Andrianov, K. A.,
Khananashvili, L. M., and Kantariya, N. L.

TITLE: Study of infrared spectra of cyclic organosiloxanes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh
nauk, no. 6, 1962, 1014 - 1019

TEXT: Infrared spectra of octamethyl cyclotetrasiloxane (I), trimethyl triphenyl cyclotrisiloxane (III), tetramethyl tetraphenyl cyclotetrasiloxane (IV), and 8 cycloorganotetrasiloxanes with methyl, ethyl, ethoxyl, butoxyl, phenyl, vinyl, and nitrile groups were investigated. The infrared spectra of (I), (III), and (IV) agreed with those described in the literature. The spectra of the other 8 cycloorganotetrasiloxanes were obtained for the first time. Stretching vibrations of the Si-O-Si group were determined for all organotetrasiloxanes in the form of broad, very intense 1080-1089 cm^{-1} bands; the positions of these were constant and scarcely effected by the character and number of the substituents. The corresponding band of the trimers appears at 1020 cm^{-1} and is less intense. The
Card 1/3

Study of infrared spectra ...

S/062/62/000/006/006/008
B117/B101

bands corresponding to the stretching vibrations of the $\text{CH}=\text{CH}_2$ group were found for compounds with 1 to 3 vinyl groups at 1596 cm^{-1} ; they became more intense with increasing number of these groups. The lower frequency of stretching vibrations of the C-C bond is due to the strong effect of the Si atom on the vinyl group. This effect is greater than that of the conjugate phenyl ring, and is commensurable with the effect of conjugate C-C or C-O bonds. The bands of the vinyl group found at 959 and 1006 cm^{-1} originate in uneven deformation vibrations of the CH bond in $-\text{CH}_2$ and $-\text{CH}$.

The intensity of these bands grows proportionally with the number of vinyl groups. Bands corresponding to stretching vibrations of the $\text{Si}-\text{C}_6\text{H}_5$ group were found at 1434 cm^{-1} for organocyclosiloxanes with phenyl groups. The 1034 cm^{-1} band ascribed to the $\text{Si}-\text{C}_6\text{H}_5$ group by L. Spialter, D. S. Priest, C. W. Harris (J. Amer. Chem. Soc. 77, 6227 (1955)) is masked by the vibrations of the Si-O-Si group; it appears distinctly in trimers only. Stretching vibrations of the $\text{Si}-\text{CH}_3$ and $\text{Si}(\text{CH}_3)_2$ groups were observed in all cyclo-organosiloxanes in the form of broad bands at $1258-1263\text{ cm}^{-1}$. Bands at 960 and 1010 cm^{-1} were found for the ethyl radical bound to silicon correspond-
Card 2/3

Study of infrared spectra ...

S/062/62/000/006/006/008
B117/B101

ponding to those reported by C. W. Joung et al. (J. Amer. Chem. Soc. 70, 3758 (1948)). Stretching vibrations of the methyl and methylene groups appear at 2885 - 2974 and 2923 cm^{-1} as in carbon compounds. There are 1 figure and 1 table.

ASSOCIATION: Institut khimii im. P. G. Melikishvili Akademii nauk GruzSSR (Institute of Chemistry imeni P. G. Melikishvili of the Academy of Sciences GSSR). Institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Institute of Fine Chemical Technology imeni M. V. Lomonosov)

SUBMITTED: January 13, 1962

Card 3/3

38718

S/191/62/000/007/005/011
B124/B144

15.8170

AUTHORS: Andrianov, K. A., Zhdanov, A. A.

TITLE: Some peculiarities in the structure of polyorganosiloxanes and polyorganometal siloxanes

PERIODICAL: Plasticheskiye massy, no. 7, 1962, 24-27

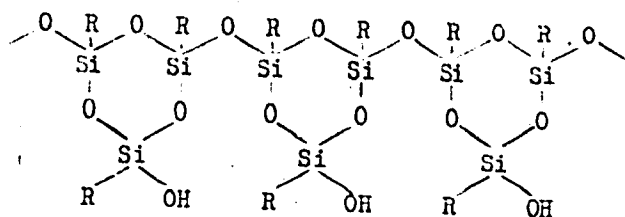
TEXT: Problems concerning the structure of polyorganosiloxanes and polyorganometal siloxanes are discussed on the basis of earlier papers by the authors and in publications from the USA. The flexibility of Si-O-Si bonds surrounded by organic groups is proved by the formation of cyclic compounds (e.g. octamethyl cyclotetrasiloxane) in the hydrolysis of aqueous dimethyl dichlorosilane solutions, the size of the resulting ring being dependent on the type of organic groups on the Si atom. Cyclic polymers of the structure

Card 1/6

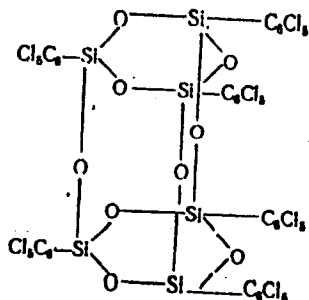
X

Some peculiarities in the structure ...

S/191/62/000/007/005/011
B124/B144



were also formed in the hydrolysis and cohydrolysis of trifunctional organosilicon monomers (phenyl trichlorosilane) in aqueous solution. A polymer having the structure

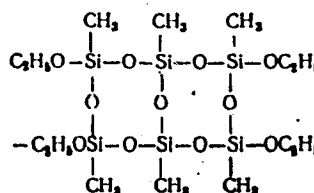
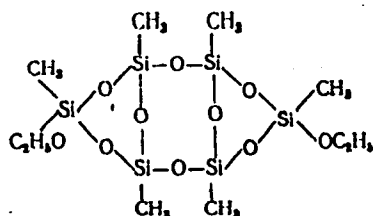


Card 2/6

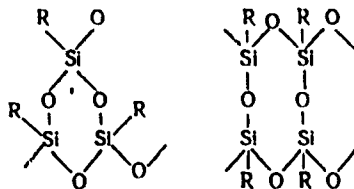
Some peculiarities in the structure ...

S/191/62/000/007/005/011
B124/B144

is formed in the hydrolysis of phenyl trichlorosilane, whereas compounds with the structure



are formed in the hydrolysis of ethyl triethoxy silane and methyl triethoxy silane. Rings of the composition

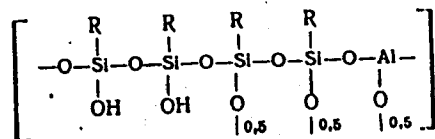


Card 3/6

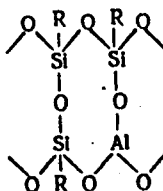
Some peculiarities in the structure ...

S/191/62/000/007/005/011
B124/B144

are formed in the synthesis of polymers with inorganic chain molecules by hydrolysis and condensation. Polyalumophenyl- or polyalumoethyl siloxanes:



result from the cohydrolysis of phenyl trichlorosilane or ethyl trichlorosilane with $AlCl_3$, as well as from reactions of $Cl_3H_5Si(OH)_2ONa$ with $Al_2(SO_4)_3$; they are readily soluble in organic solvents and do not melt at $700^\circ C$; their structure may be stated as follows:



Card 4/6

Some peculiarities in the structure ...

S/191/62/000/007/005/011
B124/B144

form cycloliner polymers, but steric hindrances caused by organic radicals on the Si atom prevent branching of molecular chains. There is 1 table. The most important English-language references are: M. M. Sprung, F. O. Guenther, J. Am. Chem. Soc. 77, 6045 (1955); *ibid.* 77, 3996 (1955); B. L. Chamberland, A. G. McDiarmid, *ibid.*, 83, 549 (1961); J. F. Brown et al., *ibid.* 82, 6194 (1960); K. A. Andrianov, J. Polymer Sci. 52, 257 (1961).

X

Card 6/6

TSITSISHVILI, G.V.; BAGRATISHVILI, G.D.; ANDRIANOV, K.A.; KHANANASHVILI, L.M.;
KANTARIYA, M.L.

Infrared spectra of cyclic organosilazanes. Izv.AN SSSR.Otd.khim.
nauk no.7:1197-1198 JI '62. (MIRA 15:7)

1. Institut khimii im. P.G.Milikishvili AN Gruzinskoy SSR i
Institut tonkoy khimicheskoy tekhnologii im. M.V.Lomonosova.
(Silazanes—Spectra)

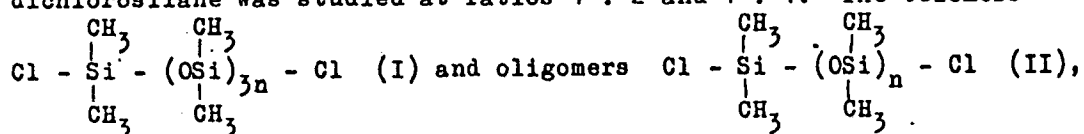
S/062/62/000/007/007/013
B117/B180

AUTHORS: Andrianov, K. A., and Severnyy, V. V.

TITLE: Splitting methyl-phenyl cyclosiloxanes with dimethyl dichlorosilane

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 7, 1962, 1237 - 1242

TEXT: The reaction of trimethyl-triphenyl cyclotrisiloxane with dimethyl dichlorosilane was studied at ratios 1 : 2 and 1 : 1. The telomers



with $n = 2 - 10$ were obtained. Despite the change in component ratio, the products are not dominating individual substances, but always mixtures of homologs. Reduction of the dimethyl dichlorosilane content reduced the yield of lowest homologs considerably and increased that of highest homologs (distillation residue). In the reaction of tetramethyl-tetra-

Card 1/2

S/062/62/000/007/011/013
B117/B180

AUTHORS: Andrianov, K. A., and Rumba, G. Ya.

TITLE: Rearrangements of dimethyl cyclosilanes and synthesis of tricyclotridecamethyl heptasilasane

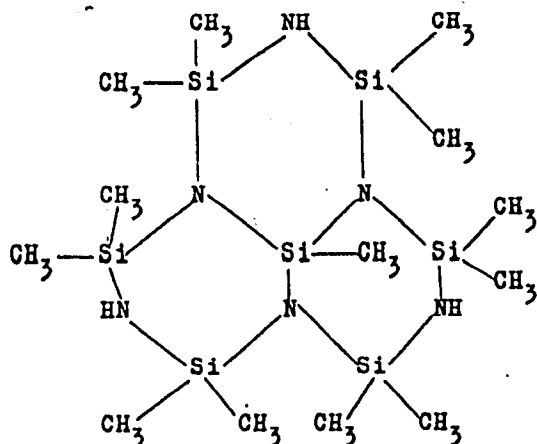
PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 7, 1962, 1313

TEXT: A new rearrangement has been found in dimethyl cyclosilanes, which changes the silicon - nitrogen skeleton of the molecules. Tricyclo-tridecamethyl heptasilasane was synthesized by this reaction at 160-260°C by heating octamethyl cyclotetrasilasane or hexamethyl cyclotrisilasane in the presence of catalytic amounts of caustic alkali. The material has molecular weight 482 - 488; melting point 165 - 167°C, refractive indices of $n_g = 1.551$, $n_p = 1.535$ extinction angle of $\sim 90^\circ$, and the structure: ✓

Card 1/2

Rearrangements of dimethyl...

S/062/62/000/007/011/013
B117/B180



ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk
SSSR (Institute of Elemental Organic Compounds of the Academy
of Sciences USSR)

SUBMITTED: May 16, 1962
Card 2/2

S/062/62/000/008/007/016
B117/B180

AUTHORS: Andrianov, K. A., and Sipyagina, M. A.

TITLE: Synthesis of α,ω -dihydroxy-methyl-phenyl siloxanes and their reactions with silicon tetrachloride and tetrabutoxy titanium

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 8, 1962, 1392-1395

TEXT: α,ω -dihydroxy-methyl-phenyl siloxanes were synthesized by hydrolyzing pure α,ω -dichloro-methyl-phenyl siloxanes. At -5°C , the following were obtained from an ethereal solution of α,ω -dichloro-methyl-phenyl siloxanes with 4% aqueous caustic soda: α,ω -dihydroxy-1,3-dimethyl-1,3-diphenyl disiloxane, m.p. 77°C , 84.7% yield; α,ω -dihydroxy-1,3,5-trimethyl-1,3,5-triphenyl trisiloxane, 76.1% yield; α,ω -dihydroxy-1,3,5,7-tetramethyl-1,3,5,7-tetraphenyl tetrasiloxane, 74.5% yield. Cross-shaped compounds with a silicon atom at the center and hydroxyl groups at the ends of the arms were obtained by reacting (excess) α,ω -dihydroxy-methyl-phenyl siloxanes with silicon tetrachloride at 23°C in the presence of pyridine: α,ω -dihydroxy-1,3-dimethyl-1,3-diphenyl disiloxane gave tetracid-(3,5-di-
Card 1/2

✓

S/062/62/000/008/008/016
B117/B180

AUTHORS: Andrianov, K. A. and Yakushkina, S. Ye.

TITLE: Synthesis and polymerization of some organocyclosilyl-dimethylene siloxanes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 8, 1962, 1396-1400

TEXT: The following six-membered cyclic compounds were synthesized by reacting bis-chloro-methyl-tetramethyl siloxane and aryl-(alkyl)-dichloro silane with metallic sodium in toluene at 108-109°C:

$(\text{CH}_3)_2\text{Si} - \text{O} - \text{Si}(\text{CH}_3)_2 - \text{CH}_2 - \text{Si}(\text{CH}_3)_2\text{CH}_2$, b.p. 90-93°C (45 mm Hg),

$n_D^{20} 1.4385, d_4^{20} 0.8846; (\text{CH}_3)_2\text{Si} - \text{O} - \text{Si}(\text{CH}_3)_2 - \text{CH}_2 - \text{Si}(\text{C}_2\text{H}_5)_2\text{CH}_2$,

b.p. 97-100°C (15 mm Hg), $n_D^{20} 1.4524, d_4^{20} 0.8930;$

$(\text{CH}_3)_2\text{Si} - \text{O} - \text{Si}(\text{CH}_3)_2 - \text{CH}_2 - \text{Si}(\text{CH}_3)\text{C}_6\text{H}_5\text{CH}_2$, b.p. 134-138°C (8 mm Hg),

Card 1/3

Synthesis and polymerization ...

S/062/62/000/008/008/016
B117/B180

n_D^{20} 1.5112, d_4^{20} 0.9607. In the presence of caustic soda and aluminum chloride, these compounds were found to polymerize less readily at 120°C than six and eight-membered dimethyl cyclosilanes. A polymer of molecular weight 169,800 was obtained from hexamethyl-cyclo trisiloxane after 4 hrs in the presence of KOH (46.3% yield). Under identical conditions it took 33 hrs to produce a viscous polymer, n_D^{20} 1.4400, from $(CH_3)_2Si - O - Si(CH_3)_2 - CH_2 - Si(CH_3)_2CH_2$ (18.2% yield). After

substituting a phenyl group for the methyl group, a viscous polymer (6%), molecular weight 1436, n_D^{20} 1.4950, was obtained from

$(CH_3)_2Si - O - Si(CH_3)_2 - CH_2 - Si(CH_3)_2C_6H_5CH_2$ after 3 hrs. When

$(CH_3)_2Si - O - Si(CH_3)_2 - CH_2 - Si(CH_3)_2CH_2$ with $AlEt_3$ at 125°C was heated for 20 hrs, ~70% of the monomer was recovered unchanged and ~30% remained as undistilled residue with n_D^{20} 1.4420. There are 3 figures and 1 table.

Card 2/3

Synthesis and polymerization ...

S/062/62/000/008/008/016
B117/B180

ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk
SSSR (Institute of Elemental Organic Compounds of the
Academy of Sciences USSR)

SUBMITTED: February 14, 1962

Card 3/3

S/062/62/000/008/012/016
B117/B180

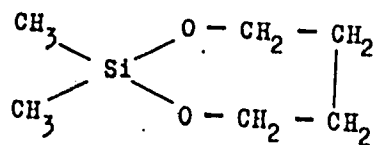
AUTHORS: Andrianov, K. A., Astakhin, V. V., and Sukhanova, I. V.
TITLE: Reaction of alkyl-phenyl-amino silanes with boric acid,
phosphoric acid and glycols
PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh
nauk, no. 8, 1962, 1478-1479

TEXT: Trialkyl-phenyl-amino silanes were found to react easily with boric and ortho-phosphoric acid, giving organo-silicon esters. With ortho-phosphoric acid and boric acid respectively the following were obtained: tris-(triethyl-silyl) phosphate, b.p. 180°C (4 mm Hg);
 n_D^{20} 1.4400; d_4^{20} 0.9700; 70% yield; tris-borate, b.p. 157-160°C (5 mm Hg);
 n_D^{20} 1.4372; d_4^{20} 0.8946; 95% yield. It was also found that dialkyl-diphenyl-amino silanes give cyclic dialkyl silane diole esters with glycols. 2,2-dimethyl-1,3-dioxa-2-sila cycloheptane: ✓

Card 1/2

Reaction of alkyl-phenyl-amino ...

S/062/62/000/008/012/016
B117/B180



b.p. 140°C (760 mm Hg); n_D^{20} 1.4252; d_4^{20} 0.9652; was synthesized from dimethyl-diphenyl-amino silane and 1,4-butanediene in 54% yield. ✓

ASSOCIATION: Institut elementoorganicheskikh soyedineniy Akademii nauk SSSR (Institute of Elemental Organic Compounds of the Academy of Sciences USSR). Vsesoyuznyy elektrotekhnicheskii institut im. V. I. Lenina (All-Union Electrotechnical Institute imeni V. I. Lenin)

SUBMITTED: February 14, 1962

Card 2/2

40728

15.8170

S/062/62/000/009/004/009
B179/B101AUTHORS: Andrianov, K. A., Dabagova, A. K., and Syrtsova, Zh. S.

TITLE: Heterofunctional condensation of methyl(phenyl) acetoxy silanes with organosilicon compounds containing an ethoxy-Si bond

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 9, 1962, 1572 - 1577

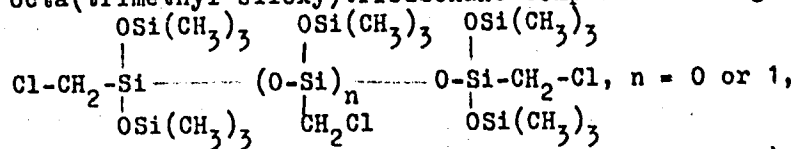
TEXT: Si-organic compounds with ethoxy-Si bond easily enter into condensation reactions with trimethyl-, phenyl methyl-, diphenyl methyl- and triphenyl acetoxy silanes if HCl is present. Tetra(trimethyl siloxy)-silane is formed by way of $\text{Si}(\text{OC}_2\text{H}_5)_4 + 4\text{CH}_3\text{COOSi}(\text{CH}_3)_3 \rightarrow \text{Si}[\text{OSi}(\text{CH}_3)_3]_4 + 4\text{CH}_3\text{COOC}_2\text{H}_5$ with 80% yield, whereas the reaction of SiCl_4 with Na-trimethyl silanolate yields only 18%. Hexa(trimethyl siloxy)-disiloxane is formed according to $(\text{C}_2\text{H}_5\text{O})_3\text{Si}-\text{O}-\text{Si}(\text{OC}_2\text{H}_5)_3 + 6\text{CH}_3\text{COOSi}(\text{CH}_3)_3 \rightarrow [(\text{CH}_3)_3\text{SiO}]_3\text{SiOSi}[\text{OSi}(\text{CH}_3)_3]_3 + 6\text{CH}_3\text{COOC}_2\text{H}_5$. In similar fashion

Card 1/3

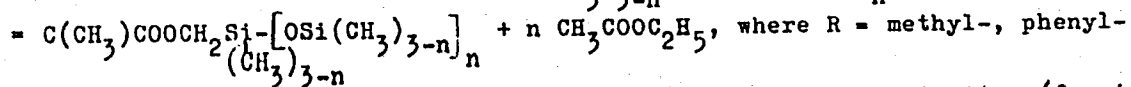
Heterofunctional condensation...

S/062/62/000/009/004/009
B179/B101

octa(trimethyl siloxy)trisiloxane compounds having the general formula



and 1,3-di(chloromethyl)tetra(phenyl methyl siloxy)disiloxane were obtained. Methyl methacrylate ethoxysilanes, with trimethyl-, phenyl dimethyl-, diphenyl methyl- and triphenyl acetoxy silane, react according to the general equation $\text{CH}_2=\text{C}(\text{CH}_3)\text{COOCH}_2\text{-Si}(\text{OC}_2\text{H}_5)_n + n \text{CH}_3\text{COOSi}(\text{CH}_3)_{3-n}\text{-CH}_2$



and $n = 1, 2, 3$. Certain compounds enter into radical polymerization (forming benzoyl peroxide), these being 1-methyl methacrylate-3-phenyl tetramethyl disiloxane, 3-methyl methacrylate-1,5-diphenyl pentamethyl disiloxane, 1-methyl methacrylate-3,3-diphenyl trimethyl disiloxane and 1-methyl methacrylate-3,3,3-triphenyl dimethyl disiloxane as well as other metha-
Card 2/3

ANDRIANOV, K.A.; YERMAKOVA, M.N.

Polycondensation reaction of α, ω -dihydroxydimethylsiloxane oligomers with phenylaminomethylmethyldiethoxysilane. Izv. AN SSSR. Otd. khim. nauk no. 9: 1577-1580 S '62. (MIRA 15:10)

1. Institut elementoorganicheskikh soedineniy AN SSSR.
(Siloxanes) (Silane)

RAFIKOV, S.R.; ANDRIANOV, K.A.; PAVLOVA, S.A.; TVERDOKHLEBOVA, I.I.

Polyorganotitanosiloxanes in solutions. Izv. AN SSSR. Otd. khim. nauk
no.9:1581-1584 S '62. (MIRA 15:10)

1. Institut elementoorganicheskikh soyedineniy AN SSSR.
(Titanium organic compounds) (Siloxanes)

ANDRIANOV, K.A.; DABAGOVA, A.K.; KUZNETSOVA, I.K.

Synthesis of unsaturated phosphoroorganosilicon compounds of the siloxane series. *Izv.AN SSSR.Otd.khim.nauk no.9:1664-1666 S '62.* (MIRA 15:10)

1. Institut elementoorganicheskikh soyedineniy AN SSSR.
(Silicon organic compounds) (Phosphorus organic compounds)

5.3833 ,

140912

S/191/62/000/010/005/010
B101/B186

AUTHORS: Golubtsov, S. A., Popeleva, G. S., Andrianov, K. A., Zaslavskaya, H. I.

TITLE: Thermal condensation of trichloro silane and methyl dichloro silane with vinyl chloride

PERIODICAL: Plasticheskiye massy, no. 10, 1962, 21 - 27

TEXT: Thermal condensation of vinyl chloride with methyl dichloro silane into methyl vinyl dichloro silane (I), and vinyl chloride with trichloro silane into vinyl trichloro silane (II) was conducted in a reaction tube of stainless steel under atmospheric pressure and at 30 sec contact time. Optimum reaction conditions were determined, and the condensation mechanism and thermal decomposition were studied. Results: The optimum temperature for synthesizing (I) is 530 - 540°C, the yield is 75 - 80% as referred to the reacting methyl dichloro silane. The side products are methyl trichloro silane, butadiene, high-boiling products, and gases containing 2% H₂, 5% C₂H₄, 4% C₂H₃Cl, 2% CH₄, and up to 1% C₂H₅. The thermal decomposition of
Card 1/3

S/191/62/000/010/005/010
B101/B186

Thermal condensation ...

methyl dichloro silane starts in at 400°C and reaches 20% at 570°C. The gaseous decomposition products contain 75% H₂ and 25% CH₄. The solid residue contains 65% Si but no free carbon. A radical mechanism is assumed, as the solid decomposition products catalyze the decomposition of methyl dichloro silane. The optimum conditions for the synthesis of II are: a molar ratio HSiCl₃ : C₂H₃Cl = 1 : 1.5 and 500 - 520°C. The yield is 70 - 75% as referred to the reacting HSiCl₃, and 60% referred to the reacting vinyl chloride. If the ratio is reduced to 1 : 0.6, the temperature has to be raised to 560 - 580°C. A ratio of 1 : 1 yielded 80% II with respect to HSiCl₃. The side products are equal amounts of SiCl₄ (15 g-moles per 100 g-moles HSiCl₃) and high-boiling polymers. The gas contains 5% H₂, 12% C₂H₄, 3% C₂H₆, the solid residue contains 10 - 50% Si and 30 - 75% elementary carbon. The thermal decomposition of HSiCl₃ in H₂, SiCl₄, and Si sets in at 560 - 570°C. The effect of the reaction vessel wall on the composition of the reaction products confirms the radical mechanism of the reaction. A surface increase of the reaction vessel by Raschig rings, Card 2/3

Thermal condensation ...

S/191/62/000/010/005/010
B101/B186

increases the yield of SiCl_4 and decreases the yield of II. Purification of the reaction vessel with⁴ alkali also reduces the yield of II. A nitrogen oxide addition of 2% reduces the yield of II to 43% and increases that of SiCl_4 to 13%. In the reaction of II with HCl at 560 - 570^oC, the gas consists of 93% H_2 with 7% C_2H_4 . There are 8 figures and 3 tables. The most important English-language references are: English Patent 752700 (1956), C. A., 51, 7402 (1957); US Patent 2770634 (1956), C. A., 51 10560 (1957), Japan Patent no. 16 (1951), C. A., 52, 3673 (1958).

f

Card 3/3

ANDRIANOV, K.A.; SHAPATIN, A.S.

Interaction of aluminum isopropylate with dialkyl esters of methyl-
phosphinic acid. Izv AN SSSR.Otd.khim.nauk no.10:1753-1756 0 '62.
(MIRA 15:10)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V.
Lomonosova.

(Aluminum compounds)

(Phosphinic acid)

ANDRIANOV, K.A.; TURETSKAYA, R.A.; GOLUBETSOV, S.A.; TROFIMOVA, I.V.

Formation reactions of alkyl(aryl)chlorosilanes in the direct
interaction of alkyl(aryl) chlorides with silicon. Report No. 12:
Effect of hydrogen chloride on the formation of ethylchlorosilanes.
Izv. AN SSSR.Otd.khim.nauk no.10:1788-1794 O '62. (MIRA 15:10)
(Silane) (Hydrochloric acid)

ANDRIANOV, K.A.; ASTAKHIN, V.V.; KOCHKIN, D.A.

Reaction of hexaalkydisilazanes with aluminum and boron halides.

Izv. AN SSSR, Otd. khim. nauk no. 10:1852-1853 0 '62

(MIRA 15:10)

1. Institut elementoorganicheskikh soedineniy i Elektrotekhnicheskii
institut im. V.I. Lenina.

(Silazanes)

(Aluminum halides)

(Boron halides)

KUZNETSOVA, A. G.; ANDRIANOV, K. A.; ZHINKIN, D. Ya.

Hydrolytic condensation of dimethylchlorosilane and phenyl-
trichlorosilane. Plast. massy no.11:15-18 '62.
(MIRA 16:1)

(Silane) (Condensation products(Chemistry))

S/062/62/000/011/004/021
B101/B144AUTHORS: Andrianov, K. A., Volkova, L. M., and Chumayevskiy, N. A.

TITLE: Vibration spectra of organic compounds containing elements of group IV (Si, Ge, Sn). Communication 7. Infrared absorption spectra of substituted amino-methyl siloxanes and stretching vibration frequencies of C-H bonds

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 11, 1962, 1958 - 1964

TEXT: The IR absorption spectra of the following compounds were studied:

$C_6H_5NHCH_2(CH_3)Si(OC_2H_5)_2$, b.p. 130 - 132°C/5 mm Hg, n_D^{20} 1.4975;
 $C_6H_5NHCH_2(CH_3)_2SiOC_2H_5$, b.p. 140 - 144°C/20 mm Hg, n_D^{20} 1.5111;
 $C_6H_5NHCH_2(CH_3)_2SiOSi(C_2H_5)_3$, b.p. 109 - 109.5°C/0.5 mm Hg, d_4^{20} 0.9402,
 n_D^{20} 1.4927; $(C_2H_5)_3SiOSi(CH_3)(CH_2NHC_6H_5)OSi(C_2H_5)_3$, b.p. 159 - 161°C/1 mm Hg,
 d_4^{20} 0.9514, n_D^{20} 1.4819; $(C_2H_5)_3SiOSi(CH_3)[CH_2N(C_2H_5)_2]OSi(C_2H_5)_3$,
Card 1/3

Vibration spectra of organic...

S/062/62/000/011/004/021
B101/B144

b.p. 102 - 106°C/0.5 mm Hg, d_4^{20} 0.8882, n_D^{20} 1.4410;

$(C_2H_5)_3SiOSi(CH_3)(CH_2N \begin{matrix} /CH_2CH_2 \\ \backslash CH_2CH_2 \end{matrix})OSi(C_2H_5)_3$; b.p. 129 - 131°C/1 mm Hg,

d_4^{20} 0.9425, n_D^{20} 1.4525; $C_6H_5NHCH_2(CH_3)_2SiOSi(CH_3)_2C_6H_5$; b.p. 123-128°C/1 mm

Hg, d_4^{20} 1.0047, n_D^{20} 1.5310; $C_6H_5(CH_3)_2SiOSi(CH_3)(CH_2NHC_6H_5)OSi(CH_3)_2C_6H_5$,
b.p. 187 - 196°C/1 mm Hg, d_4^{20} 1.0534, n_D^{20} 1.5381;

$C_6H_5NHCH_2(CH_3)_2SiOSi(C_2H_5)_2OSi(CH_3)_2CH_2NHC_6H_5$; b.p. 173 - 175°C/0.5 mm Hg,

d_4^{20} 1.023, n_D^{20} 1.5218; $(C_2H_5)_3SiOSi(CH_3)(CH_2NHC_6H_4Cl)OSi(C_2H_5)_3$; b.p.
153 - 157°C/0.5 mm Hg, d_4^{20} 1.000, n_D^{20} 1.4885, and

$(C_2H_5O)_2Si(CH_3)CH_2NH(CH_2)_6NH_2$; b.p. 144 - 147°C/7 mm Hg, d_4^{20} 0.9238,
 n_D^{20} 1.4450.

The results confirm the conclusions drawn by N. A. Chumayevskiy
(Optika i spektroskopiya, v. X, no. 1, 1961, p. 69) concerning the
Card 2/3

Vibration spectra of organic...

S/062/62/000/011/004/021
B101/B144

frequencies of Si-O-Si, Si-O-C, Si-CH₃, Si-C₂H₅, and Si-C bonds. In the present paper the frequencies of the C-H bonds in the Si-CH₃ and Si-C₂H₅ groups were identified, using data from the earlier paper. The following interpretation of frequencies is suggested: $\nu_s(\text{CH}_2)$ 2870 - 2880 cm⁻¹; $\nu_{as}(\text{CH}_2)$ 2925 - 2940 cm⁻¹; $\nu_s(\text{CH}_3)$ 2900 - 2910 cm⁻¹, and $\nu_{as}(\text{CH}_3)$ 2956 - 2970 cm⁻¹. There are 4 figures and 4 tables.

ASSOCIATION: Institut elementoorganicheskikh sovedineniy Akademii nauk SSSR (Institute of Elemental Organic Compounds of the Academy of Sciences USSR)

SUBMITTED: March 23, 1962

Card 3/3

S/062/62/000/011/010/021
B101/B144

AUTHORS: Semenova, Ye. A., Zhinkin, D. Ya., and Andrianov, K. A.

TITLE: Reaction of dialkyl dichlorosilanes and alkyl dichlorosilanes with methyl amine

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 11, 1962, 2036 - 2039

TEXT: Methyl dichlorosilane (I), dimethyl dichlorosilane (II), ethyl dichlorosilane (III), and diethyl dichlorosilane (IV) were aminated by bubbling gaseous methyl amine through the benzene solution of the silane at 15 - 20°C, filtering off the precipitated methylamine hydrochloride, distilling off the benzene, and rectifying the end product. Alkyl dichlorosilanes give disilazanes and cyclosilazanes: $\text{RHSiCl}_2 + \text{CH}_3\text{NH}_2 \rightarrow [(\text{CH}_3)\text{HN-SiHR}]_2\text{NCH}_3$ + $[\text{RHSiNCH}_3]_3$ or $[\text{RHSiNCH}_3]_4 + \text{CH}_3\text{NH}_2 \cdot \text{HCl}$. Cyclotetrasilazane is only formed in the aminolysis of I. The liquid reaction product from I contained: 2.5 - 5% bis-(methyl amino methyl silyl)-methyl amine, b.p. 48.5°C/5 mm Hg, n_D^{20} 1.4425, d_4^{20} 0.8871; 35 - 40% trimethyl cyclotrimethyl

Card 1/3

S/062/62/000/011/010/021
B101/B144

Reaction of dialkyl dichlorosilanes...

silazane, b.p. 56°C/5 mm Hg, n_D^{20} 1.4580, d_4^{20} 0.9297, and 20 - 25% tetra-
methyl cyclotetramethyl silazane, b.p. 88°C/2 mm Hg, n_D^{20} 1.4810, d_4^{20} 0.9776.

The total silazane yield was 60%, referring to I. The distillation residue
(31%) contained 38.32% Si, 16 - 17% N, and had molecular weight 1856. The
reaction product from III contained 38 - 40% each of bis-(methyl amino
ethyl silyl)-methyl amine, b.p. 68.5°C/1.5 mm Hg, n_D^{20} 1.4520, d_4^{20} 0.8929,
and triethyl cyclotrimethyl silazane, b.p. 74.5/1.5 mm Hg, n_D^{20} 1.4680,
 d_4^{20} 0.9324, total yield 75%. The distillation residue (23%) contained
32.99% Si, 14.25% N, and had molecular weight 518.3. Dialkyl dichloro-
silanes reacted thus with methyl amine: R_2SiCl_2

+ $CH_3NH_2 \rightarrow [(CH_3)HN-SiR_2]_2NCH_3 + R_2Si(NHCH_3)_2 + CH_3NH_2 \cdot HCl$. The reaction
product from II contained 20% bis-(methyl amino dimethyl silyl)-methyl
amine, b.p. 34.5°C/0.5 mm Hg, n_D^{20} 1.4435, d_4^{20} 0.8886; and 70% bis-(methyl
amino)-dimethyl silane, b.p. 108°C/750.5 mm Hg, n_D^{20} 1.4140, d_4^{20} 0.8219,
total yield 55 - 58%. The distillation residue (5%) contained 32.78% Si,

Card 2/3

Reaction of dialkyl dichlorosilanes...

S/062/62/000/011/010/021
B101/B144

6.87% N, and had molecular weight 445.6. From IV only bis-(methyl amino)-diethyl silane was formed, b.p. 156°C/745 mm Hg, n_D^{20} 1.4330, d_4^{20} 0.8421, yield 57%. The distillation residue (19.5%) contained 27.45% Si, 7.5% N, and had molecular weight 491. There is 1 table. The most important English-language reference is: E. Larsson, L. Bjellerup, J. Amer. Chem. Soc., 75, 995 (1953).

SUBMITTED: March 15, 1962

Card 3/3

S/062/62/000/011/011/021
B101/B144

AUTHORS: Andrianov, K. A., Pakhomov, V. I., and Lapteva, N. Ye.

TITLE: Reactions of allyl phenol and trimethyl siloxy allyl benzene with alkyl alkoxy silanes

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 11, 1962, 2039 - 2046

TEXT: In the reaction of o-allyl phenol with alkyl alkoxy silanes in the presence of $H_2PtCl_6 \cdot 6H_2O$, the following addition was found to take place:
 $=SiH + CH_2=CHCH_2C_6H_4OH \rightarrow =Si(CH_2)_3-C_6H_4OH$ (1); as well as substitution of the hydroxyl hydrogen according to: $=SiH + HOC_6H_4CH_2CH=CH_2 \rightarrow =SiOC_6H_4C_3H_5 + H_2$ (2). Reaction (2) was confirmed by ether formation from o-allyl phenol and triethoxy silane in the presence of KOH. The resulting product was identical with that obtained by reaction (2), its IR spectrum, in contrast to the compound obtained by reaction (1), did not show the 3400 - 3600 cm^{-1} band of the HOC_6H_4 group. To avoid hydrogen substitution, the
 Card 1/5

Reactions of allyl phenol and...

S/062/62/000/011/011/021
B101/B144

reaction of the alkyl alkoxy silanes was carried out with o-trimethyl siloxy allyl benzene synthesized from trimethyl chlorosilane and o-allyl phenol in petroleum ether by bubbling with NH_3 (yield 74%), or in petroleum ether and pyridine (yield 50%), - b.p. 93 - 95°C/7 - 8 mm Hg, d_4^{20} 0.9542, n_D^{20} 1.4885. It reacted with silanes at 125°C in N_2 atmosphere and in the presence of $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$, dissolved in i-propanol, only by addition:

$$\text{CH}_3(\text{RO})_2\text{SiH} + \text{CH}_2=\text{CHCH}_2\text{C}_6\text{H}_4\text{OSi}(\text{CH}_3)_3 \rightarrow (\text{CH}_3)_3\text{SiOC}_6\text{H}_4\text{C}_3\text{H}_6\text{SiCH}_3(\text{OR})_2; \text{R} = \text{CH}_3, \text{C}_2\text{H}_5, \text{C}_4\text{H}_9.$$

The following compounds were synthesized by this reaction:

$(\text{CH}_3)_3\text{SiOC}_6\text{H}_4(\text{CH}_2)_3\text{SiCH}_3(\text{OC}_2\text{H}_5)_2$, yield 54%, b.p. 145 - 146°C/2 - 3 mm Hg, d_4^{20} 0.9527, n_D^{20} 1.4660; $(\text{CH}_3)_3\text{SiOC}_6\text{H}_4(\text{CH}_2)_3\text{SiCH}_3(\text{OC}_4\text{H}_9)_2$, yield 55 %, b.p. 180 - 183°C/2 - 3 mm Hg, d_4^{20} 0.9321, n_D^{20} 1.4663; $(\text{CH}_3)_3\text{SiOC}_6\text{H}_4(\text{CH}_2)_3\text{Si}(\text{OC}_2\text{H}_5)_3$, yield 52%, b.p. 157 - 159°C/1 - 2 mm Hg, d_4^{20} 0.9771, n_D^{20} 1.4620; $(\text{CH}_3)_3\text{SiOC}_6\text{H}_4(\text{CH}_2)_3\text{Si}(\text{OC}_4\text{H}_9)_3$, yield 50%, b.p. 202 - 204°C/1 - 2 mm Hg,

Card 2/5

S/062/62/000/011/011/021
B101/B144

Reactions of allyl phenol and...

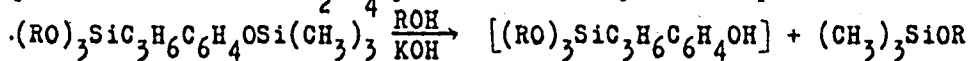
d_4^{20} 0.9442, n_D^{20} 1.4954; $(CH_3)_3SiOC_6H_4(CH_2)_3SiC_2H_5(OC_2H_5)_2$, yield 75%, b.p. 162 - 164°C/ 2 - 3 mm Hg, d_4^{20} 0.9509, n_D^{20} 1.4698;

$(CH_3)_3SiOC_6H_4(CH_2)_3SiC_2H_5(OC_4H_9)_2$, yield 52%, b.p. 184 - 188°C/1 mm Hg,

d_4^{20} 0.9336, n_D^{20} 1.4700; $(CH_3)_3SiOC_6H_4(CH_2)_3SiC_2H_5(OCH_3)_2$, yield 60%, b.p. 143°C/1 - 2 mm Hg, d_4^{20} 0.9729, n_D^{20} 1.4750. The IR spectra of these com-

pounds and of the products of their hydrolysis did not show the 1375 cm^{-1} band characteristic of the C-CH₃ group, but the 1449 cm^{-1} band corresponding to the -CH₂-CH₂- group. Thus the Si adds to the carbon atom situated

at the end of the allyl group. The alcoholysis of these compounds in the presence of KOH or H₂SO₄ yielded heterocyclic compounds:



→ $(RO)_2SiC_3H_6C_6H_4O + ROH$. The following compounds were thus obtained:

Card 3/5 $(RO)_2SiC_3H_6C_6H_4O$, b.p. 180 - 170

Reactions of allyl phenol and...

S/062/62/000/011/011/021
B101/B144

$\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{Si}(\text{OC}_2\text{H}_5)\text{CH}_3}$, yield 79%, b.p. 95 - 97°C/1 - 2 mm Hg, d_4^{20} 1.0359, n_D^{20} 1.5020; $\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{Si}(\text{OC}_4\text{H}_9)\text{CH}_3}$, yield 69 - 70%, b.p. 132 - 134°C/9 mm Hg, d_4^{20} 1.0099, n_D^{20} 1.4952; $\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{Si}(\text{OC}_4\text{H}_9)_2}$, b.p. 143 - 145°C/3 - 4 mm Hg, d_4^{20} 1.0044, n_D^{20} 1.4812; $\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{Si}(\text{OC}_2\text{H}_5)_2}$, yield 59%, b.p. 117-121°C/1.2 mm Hg, d_4^{20} 1.0583, n_D^{20} 1.4918; $\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{SiC}_2\text{H}_5(\text{OC}_2\text{H}_5)}$, yield 50%, b.p. 110 - 112°C/1 - 2 mm Hg, d_4^{20} 1.0023, n_D^{20} 1.5040; $\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{SiC}_2\text{H}_5(\text{OC}_4\text{H}_9)}$, yield 45%, b.p. 148 - 150°C/2 - 3 mm Hg, d_4^{20} 1.0042, n_D^{20} 1.4985; $\overline{\text{OC}_6\text{H}_4\text{C}_3\text{H}_6\text{SiC}_2\text{H}_5(\text{OCH}_3)}$, yield 67%, b.p. 95°C/1 mm Hg, d_4^{20} 1.0504, n_D^{20} 1.509.

The hydrolysis of some of these cyclic compounds yielded polysiloxanes containing oxyphenyl groups. There are 4 figures and 2 tables. The English-language reference is: J. L. Speier, R. Zimmerman, J. Webster, J. Amer. Chem. Soc., 78, 2278 (1956); British patent 769497 (1957).

Card 4/5

Reactions of allyl phenol and...

S/062/62/000/011/011/021
B101/B144

ASSOCIATION: Nauchno-issledovatel'skiy institut plasticheskikh mass
(Scientific Research Institute of Plastics)

SUBMITTED: March 27, 1962

Card 5/5

S/079/62/032/007/006/007
1032/1232>

AUTHORS: Andriyanov, K. A., Vasil'yeva, T. V. and Korotkevich, S. Kh.

TITLE: Reactions of telomerisation of organic cyclosiloxanes with titanium tetrachloride

PERIODICAL: Zhurnal obshchei khimii, v. 32, no. 7, 1962, 2311-2314

TEXT: The reaction of octa-methyl-cyclo-tetra-siloxane with titanium tetrachloride was studied with the aim of elucidating whether the products of the reaction would include, besides α -chloro- ω -trichloro-titanoxy-octa-methyl-tetra-siloxane, also other telomers. It was found that when octa-methyl-cyclo-tetra-siloxane was made to react with $TiCl_4$, at molar ratio 2:1, two compounds were obtained: α -chloro- ω -trichloro-titanoxy-octa-methyl-tetra-siloxane; and a higher telomer, the analysis of which corresponded to the formula $C_{16}H_{48}O_8Si_8TiCl_4$. The yields were 42.4% and 8.51% respectively. The higher telomer is a yellow liquid sensitive to humidity. It could be distilled only under high vacuum at 133-135°C. It is considered to be bis-(chloro-octa-methyl-tetra-siloxane)-dichloro-titanium. The reaction of $TiCl_4$ and tri-methyl-tri-phenyl-cyclo-trisiloxane did not result in the formation of a telomer. Usually poly-titano-methyl-phenyl-siloxane polymer was obtained. At a higher temperature, at 170°C a polymer corresponding to the formula $C_{63}H_{72}O_{10.5}Si_9TiCl_4$ was formed. There is 1 table.

ASSOCIATION: Moskovskii institut tonkoi khimicheskoi tekhnologii (Moscow Institute for Fine Chemical Technology)

SUBMITTED: July 5, 1961

Card 1/1

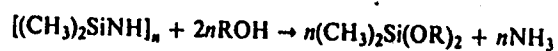
S/079/62/032/007/007/007
I032/I232

AUTHORS: Andriyanov, K. A. and Astakhin, V. V.

TITLE: The reaction of poly-alkyl-amino-silanes with alcohols

PERIODICAL: Zhurnal obshchei khimii, v. 32, no. 7, 1962, 2316-2318

TEXT: The reactions of hexa-methyl-cyclo-triamino-silane and octa-methyl-cyclo-tetra-amino-silane with ethyl-, propyl-, butyl- and iso-butyl-alcohols result in complete destruction of the rings and in formation of ammonia and dimethyl-di-alkoxy-silanes, according to the scheme:



The English-language reference reads: L. Sommer, J. Am. Chem. Soc. 71,, 3254 (1949)

ASSOCIATION: Institut elementoorganicheskikh soyedinenii Akademii nauk SSSR (Institute for Metal-Organic Compounds, AS USSR) ✓

SUBMITTED: July 10, 1961

Card 1/1

S/079/62/032/010/008/008
D214/D307

AUTHORS: Andrianov, K.A., Khayduk, Ionel, Khananashvili, L.M.,
and Nekhayeva, N.I.

TITLE: The synthesis of dimethylcyclosilthioxanes

PERIODICAL: Zhurnal obshchey khimii, v. 32, no. 10, 1962, 3447

TEXT: A description of the synthesis of two examples of a hitherto unknown class of compounds: cyclosilthioxanes. The treatment of 1,3-dichlorotetramethyldisiloxane with H₂S in the presence of pyridine gave a crystalline compound (b. range 116-122°C/2 mm Hg; m.p. 38-42°C). From the quantitative analysis of this compound and from ir, which showed the presence of Si-O-Si, Si-S-Si and Si-CH₃ bonds, the structure was found to be $(\text{CH}_3)_2 \text{Si} \text{O} \text{Si}(\text{CH}_3)_2 \text{S} \text{Si}(\text{CH}_3)_2 \text{O} \text{Si}(\text{CH}_3)_2 \text{S}$. Under similar conditions 1,5-dichloro-hexamethyltrisiloxane gave a colorless, transparent liquid (b. range 170-172.5°C) the structure of which was shown to be $(\text{CH}_3)_2 \text{SiOSi}(\text{CH}_3)_2 \text{SSi}(\text{CH}_3)_2 \text{O}$.

Card 1/2

ANDRIANOV, K. A.; KHANANASHVILI, L. M.; KHAN' EN'-TSZE[Han En-tse];
TIKHONOV, V. S.

Reaction of dimethyldichlorosilane with iron oxide. Zhur. ob.
khim. 32 no.12:3951-3952 D '62. (MIRA 16:1)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni
M. V. Lomonosova.

(Silane) (Iron oxides)

S/080/62/035/007/006/013
D214/D307

AUTHORS: Turetskaya, T.A., Golubtsov, S.A., Tromimova, I.V.
and Andrianov, K.A.

TITLE: The influence of additions of some metals on the
activity of silicon-copper alloy in its reaction
with ethyl chloride

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, no. 7, 1962,
1496-1502

TEXT: The general and selective activities of Si-Cu alloys
in the reaction with EtCl to give a mixture of ethyl chlorosilanes
are affected by the chemical nature of the alloy. The presence of
1-2% Fe, Al, Ca or Ti in the alloy increases its general activity,
while Al, Ca and Ti also increase its selective activity by increas-
ing the yield of EtSiHCl₂. The increase in activity is more evident
at low Cu concentrations. The added metals are localized at the
interphase boundaries in the alloy, these being the active centers
in the reaction. The influence Ca bears on the activity of the

Card 1/2

S/080/62/035/007/006/013
D214/D307

The influence of additions ...

of the alloy is affected by the presence of other metals. Fe in concentrations of up to 10%, does not influence the process. Concentrations of Bi and Sb of the order of 0.001% influence the selective activity and increase the yield of Et_2SiCl_2 . Pb, in these concentrations, acts as a catalytic poison. At higher concentrations, both Bi and Sb also become poisons and at concentrations of 0.01% these metals render the alloy inactive. The mechanism of the action of the added metals cannot as yet be explained. There are 4 figures and 6 tables. ✓

SUBMITTED: December 9, 1960

Card 2/2

10110

S/020/62/145/005/010/020
B106/B144

AUTHORS: Andrianov, K. A., Corresponding Member AS USSR, and Rumba, G. Ya.

TITLE: Rearrangoment of hexamethyl and octamethyl cyclosilazanes

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 145, no. 5, 1962, 1049-1051

TEXT: It has been newly discovered that dimethyl cyclosilazanes may undergo rearrangement of the silicon-nitrogen structure. Ring extension occurs through action of trimethyl chlorosilane and ammonia on hexamethyl cyclo-trisilazane (I) at 20 - 40°C. 36.5% octamethyl cyclotetrasilazane (II) forms 18.9% high-boiling products in addition to the normal reaction products, e. g. octamethyl trisilazane, and 12.3% of a polymer (molecular weight about 2000) are formed. About 3% of II is formed by 48 hrs heating of I together with ammonium chloride to 250°C. The rearrangement is explained as follows: the action of ammonia on trimethyl chlorosilane, or the thermal dissociation of NH_4Cl , produce hydrogen chloride which splits the ring of I in some place. Thereby the chlorine is added to the silicon.

Card 1/5

Rearrangement of hexamethyl ...

B/020/62/145/005/010/020
B106/B144

Haber, J, Am. Chem. Soc., 70, 3888 (1948); E. Larsson, B. Smith, Acta chem. Scand., 3, 467 (1949).

ASSOCIATION: Institut elementoorganicheskikh soedineniy Akademii nauk SSSR (Institute of Elemental Organic Compounds of the Academy of Sciences USSR)

SUBMITTED: May 17, 1962

Card 3/3

Telomerization of organocyclosiloxanes ...

S/020/62/146/003/012/019
B101/B144

17.6%, the residue containing 18.6% telomers with $n > 2$. For the telomer with $n = 1$, the b.p. is $150^{\circ}\text{C}/5$ mm Hg, d_4^{20} 1.0102, n_D^{20} 1.4303; for $n = 2$ the b.p. is $175-178^{\circ}\text{C}/0.01$ mm Hg, d_4^{20} 0.9988, n_D^{20} 1.4364. The reaction of II with III under the same conditions as for I with III yielded 32.9% telomer with $n = 1$, 4.9% $n = 2$, residue 63.2%. Conversion of II was 88.6%, of III 36.5%. Telomer with $n = 1$ had b.p. $198^{\circ}\text{C}/4$ mm Hg, d_4^{20} 1.1276, n_D^{20} 1.5118, and with $n = 2$ b.p. $201-203^{\circ}\text{C}/0.01$ mm Hg, d_4^{20} 1.1310, n_D^{20} 1.5304. Reaction of II with III in a steel autoclave led to cleavage of the siloxane bonds and formation of oligomers $\text{Cl}(\text{CH}_3)_2\text{Si}-[\text{OSi}(\text{CH}_3)(\text{C}_6\text{H}_5)]_n\text{Cl}$ owing to catalytic action of the resulting FeCl_3 . In the reaction of trimethyl trivinyl cyclotrisiloxane with III in glass ampoules at $250-200^{\circ}\text{C}$ no telomers could be identified, owing to intense polymerization. The hexaorganotricyclotrisiloxanes are in the following order of telomerizing capacity:

Card 2/3

Card 3/3

ANDRIANOV, K. A. (Moscow)

"Polymere mit anorganischen Kettenmolekullen fur Elaste und Plaste."

report presented at the Chemistry Technology Assn., Technical Conference on the
Chemistry and Technology of High Polymers, Leipzig, GDR, 17-19 Jan 1963.

KOLESNIKOV, G.S., otv. red.; ANDRIANOV, K.A., red.; DOGADKIN, B.A., red.; DOLGOPLOSK, B.A., red.; YENIKOLOPYAN, N.S., red.; KARGIN, V.A., red.; KOZLOV, P.V., red.; KOROTKOV, A.A., red.; KORSHAK, V.V., red.; LAZURKIN, Yu.S., red.; MEDVEDEV, S.S., red.; MIKHAYLOV, N.V., red.; PASYNSKIY, A.G., red.; SLONIMSKIY, G.L., red.; SMIRNOV, V.S., red.; TSVETKOV, V.N., red.; FREYMAN-KRUPENSKIY, K.A., tekhn. red.

[Carbochain high-molecular weight compounds] Karbotseprnye vysokomolekuliarnye soedinenia; sbornik statei. Moskva, Izd-vo AN SSSR, 1963. 287 p. (MIRA 17:1)

ANDRIANOV, Kuz'ma Andrianovich; EPSHTEYN, Lev Abramovich
[deceased]; RYZHIKHINA, Ye.G., red.; BUL'DYAYEVA, N.A.,
tekhn. red.

[Mica electric insulating materials] Sliudinitovye elek-
troizoliatsionnye materialy. Moskva, Gosenergoizdat, 1963.
230 p. (Polimery v elektroizoliatsionnoi tekhnike, no.8)
(MIRA 17:4)

REF(C) EPP/EPA(D)-2/EWP(J)/EWT(M)/T Pc-4/Pr-4/Ps-4/Pt-10
ACCESSION NR: AR5000799 S/0081/64/0001017/S036/S036

44
40
B

SOURCE: Ref. zh. Khimiya, Abs. 175203

AUTHOR: Andrianov, K. A.; Khananashvili, L. M.; Mil'gotin, I. M.; Shapatin, A. S.; Lomonosov, A. V.

TITLE: The synthesis of polydimethylsiloxanes and silicoorganic polyurethans with a cycloreticular molecular structure

CITED SOURCE: Sb. Vysokomolekul. soyedineniya. Geterotsepn. vysokomolekul. soyedineniya. M., Nauka, 1963, 18-23

TOPIC TAGS: polydimethylsiloxane, silicoorganic polyurethan, heteroorganic polymer, polyurethan synthesis, polysiloxane synthesis, cross-linked polymer, alkylene diisocyanate, glycoxysilane, silicon tetrachloride, transesterification, alkoxysilane

TRANSLATION: Cross-linked oligomers of the dimethylsiloxane series were obtained by opening the rings of octamethylcyclotetrasiloxane under the influence of KOH and then reacting the resultant potassium salts of dimethylsiloxanes with silicon tetrachloride. Glycoxysilanes were synthesized by the transesterification of tetraethoxysilane with glycols at 155C and of phenyltriethoxysilane with glycols