51-.4-2-7/28

Vibrational Spectra of Alkoxysilanes and Siloxanes. I.

SUBMITTED: April 13, 1957.

1. Orthosilicic acid esters-Infrared spectra 2. Infrared spectrum analyzers-Applications

Card 3/3

VORONKOV, M.G.; SKORIK, Yu.I.

Synthesis of trialkylvanadates and trialkylantimonites. Izv. AN SSSR Otd. khim. nauk no.4:506-507 Ap '58. (MIRA 11:5)

1.Institut khimii silikatov Akademii nauk SSSR.
(Antimonates) (Vanadates)

SOV/51-5-4-3/21

AUTHORS: Lazarev, A.N., Voronkov, M.G. and Tenisheva, T.F.

TITLE: On Vibrations of Si--O--Si and Si--CH_2--Si bonds in Hexachlorodisiloxane and in Si-hexachlorodisilmethane(O kolebaniyakh svyazey Si-O-Si i Si-CH_2-Si v geksakhlordisiloksane i Si-geksakhlordisilmetane)

PERIODICAL: Optika i Spektroskopiya, 1958, Vol 5, Nr 4, pp 365-368 (USSR)

ABSTRACT: The authors measured the frequencies and polarization states of the strongest lines in the Raman spectra of hexachlorodisiloxane $Cl_3SiOSiCl_3$ and Si-hexachlorodisilmethane $Cl_3SiCH_2SiCl_3$. The infrared spectra of vapours of these compounds were also obtained and they are given in Fig 1. The Raman spectra were measured using an ISP-51 spectrograph. The infrared absorption spectra were obtained using a single-beam vacuum spectrometer VIKS-M3 with a NaCl prism and an ISP-15-b spectrometer with a KBr prism. The results obtained and their interpretation are given in the table on p 366. The spectra were discussed assuming C_{2v} symmetry for these molecules. The strong absorption band at 1131-1170 cm^{-1} was ascribed to antisymmetrical valence vibrations of the Si--O--Si bond in $Cl_3SiOSiCl_3$. The intense

Card 1/3

SOV351-5-4-3/21

On Vibrations of Si--O--Si and Si--CH_2--Si bonds in Hexachlorodisiloxane and in Si-hexachlorodisilmethane

polarization Raman line at 353 cm^{-1} was due to symmetrical valence vibrations of the Si--O--Si bond in the same molecule. In the $Cl_3SiCH_2SiCl_3$ spectrum the 308 cm^{-1} Raman line corresponds to symmetrical vibrations and the 800 cm^{-1} absorption band corresponds to antisymmetrical vibrations of the Si--C--Si bond. Deformational vibrations of the Si--O--Si and Si--C--Si bonds are probably responsible for the 274 cm^{-1} (or 329 cm^{-1}) and 246 cm^{-1} lines. Of the four deformational vibrations of the CH_2 group in the $Cl_3SiCH_2SiCl_3$ spectrum the internal deformational vibrations are represented by the 1340 cm^{-1} frequency and the external vibrations

Card 2/3

SOV/51-5-4-3/21

On Vibrations of Si--O--Si and Si--CH_2--Si bonds in Hexachlorodisiloxane and in Si-hexachlorodisilmethane

are responsible for the 1080 and probably 690 cm^{-1} bands. There are 1 figure, 1 table and 7 references, 3 of which are American, 3 German and 1 Soviet.

ASSOCIATION: Institut khimiisilikatov, AN SSSR (Institute of Silicate Chemistry, Academy of Sciences of the U.S.S.R.)

SUBMITTED: March 28, 1958

Card 3/3

1. Silicones--Spectra 2. Methanes--Spectra 3. Roman spectra 4. Infrared spectra 5. Molecules--Vibration

SOV/62-58-6-7/37

AUTHORS: ~~Voronkov, M. G.,~~ Davydova, V. P., Dolgov, B. N.

TITLE: Investigations in the Field of Alkoxysilanes (Issledovaniya v oblasti alkoksisilanov) Communication 10. A New Method of Synthetizing Cyclic Ethers of Dialkylsilanediols and Orthosilicic Acid (Soobshcheniye 10. Novyy metod sinteza tsiklicheskikh efirov dialkilsilandiolov i ortokremnevoy kisloty)

PERIODICAL: Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk, 1958, Nr 6, pp. 698-701 (USSR)

ABSTRACT: It was shown already on an earlier occasion that acetoxilanes react without any difficulties with alcohols to the accompaniment of the formation of corresponding alkoxysilanes and acetic acid. In the course of the present paper the authors investigated the possibility of applying this reaction to the synthesis of cyclic ethers of dialkylsilanediols by the interaction of dialkyldiacetoxysilanes with glycols. A new method of synthetization of 2,2-dialkyl-1,3-dioxa-2-silazyklononan by the reaction of dialkyldiacetoxysilanes with β, γ, δ, ε -glycols. In this way 4 of such compounds were obtained, viz. with

Card 1/2

Investigations in the Field of Alkoxysilanes. SOV/62-58-6-7/37
Communication 10. A New Method of Synthetizing
Cyclic Ethers of Dialkylsilanediols and Orthosilicic Acid

6, 7-, 8-, and 9-membered cycles (3 of them for the first time). The reaction of dialkyldiacetoxysilanes with ethylene glycol and propylene glycol leads to the formation of 2,2,7,7-tetraalkyl-1,3,6,8-tetraoxa-2,7-disilacyclodecane or of the corresponding methyl derivatives. In the interaction of tetraacetoxysilane with 1,3 butane diol 4,8-dimethyl-1,5,7,11-tetraoxa-6-silaspiro-5,5-undecane was obtained. There are 1 table and 6 references, 3 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of the Chemistry of Silicates, AS USSR)

SUBMITTED: December 22, 1956

1. Ethers--Synthesis 2. Glycols--Chemical reactions 3. Silicic acid--Chemical reactions

Card 2/2

5(3) SOV/30-58-12-39/46

AUTHORS: Voronkov, M. G., Candidate of Chemical Sciences, Losev, V. B.

TITLE: Chemistry and the Practical Application of Silicon-Organic Compounds (Khimiya i prakticheskoye primeneniye kremniyorganicheskikh soyedineniy) All-Union Conference at Leningrad (Vsesoyuznaya konferentsiya v Leningrade)

PERIODICAL: Vestnik Akademii nauk SSSR, 1958, Nr 12, pp 97 - 100 (USSR)

ABSTRACT: The 2.Conference was held from September 25 to September 27. More than 650 persons from various towns of the Soviet Union as well as from foreign countries took part in the conference, among them also industry functionaries. The conference was organized by:Otdeleniye khimicheskikh nauk (Department of Chemical Sciences), Institut khimii silikatov Akademii nauk SSSR (Institute of Silicon Chemistry, Academy of Sciences, USSR), Vsesoyuznoye khimicheskoye obshchestvo im.D.I.Mendeleyeva (All Union Chemical Society imeni

Card 1/8

D.I.Mendeleyev), Gosudarstvennyy komitet Soveta Ministrov SSSR po khimii (State Committee for Chemistry of the USSR Council of Ministers) and Leningradskiy sovnarkhoz (Leningrad Economic Council). Lectures dealing with the following subjects were held at the plenary session by :K.A.Andrianov: "On the Particular Features and Prospects of the Development of Silicon-Organic Chemistry in the Soviet Union"; V.Bazhant :"On Work Within the Field of Silicon-Organic Compounds at the Chemical Institute of the Czechoslovakian Academy of Sciences". Conference work was carried out in 4 sections: Monomers, polymers and their practical application, as well as analysis and physico-chemical research methods.

N.N.Tishina, K.A.Andrianov, S.A.Golubtsov et al. showed the possibility of developing a new technological method for the purpose of obtaining phenyltrichlorosilanes.

I.V.Trofimova, K.A.Andrianov, S.A.Golubtsov gave

Chemistry and the Practical Application of Silicon-Organic Compounds. All Union Conference at Leningrad SOV/30-58-12-39/46

new data on the synthesis of trichlorosilane by the interaction of hydrogen chloride with silicon. P.Rostsishevskiy gave a report on the works carried out by Polish scientists in the field of methyl-ethyl- and phenylchlorosilane.

M.Ye.Dolgaya, Ye.A.Chernyshov, Li Kuang-liang spoke about methods of synthetizing aromatic silicon-organic monomers by the interaction of hydrosilane with aromatic hydrocarbon in the presence of catalyzers with formation of the compound Si-C and separation of hydrogen.

N.F.Orlov, B.N.Dolgov and M.G.Voronkov reported on the results obtained by means of a new method of synthetizing triorgano-siloxy derivatives.

G.I.Nikishin, A.D.Petrov and S.I.Sadykh-zade spoke about the behavior of various dichloroalkanes and dichloroalkenes with chloric atoms at the conditions of a direct synthetization.

Card 3/8

V.A.Ponomarenko, A.D.Petrov et al. gave a report

Chemistry and the Practical Application of Silicon-Organic Compounds. All Union Conference at Leningrad SOV/30-58-12-39/46

on the catalytic affiliation of hydrosylanes to unsaturated compounds.
V.F.Mironov, V.A.Ponomarenko, A.D.Petrov et al. spoke about the comparison of the properties of organic silicon-, germanium-, and tin-compounds.
S.I.Sadykh-zade, A.D.Petrov, B.N.Dolgov, and N.P. Kharitonov spoke about new methods of synthesis of simple organo-silicon vinyl ether.
A.N.Nesmeyanov, R.Kh.Freydlina and his collaborators dealt with thermal and catalytical telomerization of hydroxylanes with olefins.
Yu.K.Yur'yev recommended a catalytical method of synthesis of silacyclopentane from furan and silane.
N.S.Nametkin, A.V.Topchiyev et al. reported on the research of the reaction of chloromethylation of silicon-organic compounds.
M.G.Voronkov et al. showed that the organosiloxane easily change over into heterolytic fission reactions caused by the influence of nucleophilic and electro-

Card 4/8

SOV/30-58-12-39/46

Chemistry and the Practical Application of Silicon-Organic Compounds. All Union Conference at Leningrad

philic reagents.
A.G.Kuznetsova, K.A.Andrianov and D.Ya.Zhinkin gave new data on the common hydrolysis of aequimolecular mixtures of diethyl-dichlorosilane with phenyltrichlorosilane.
B. Lengyel, T. Szekely, A. Csuppon (Hungary) reported on results obtained by physico-chemical investigations of the process of hydrolysis- and condensation reactions of methylchlorosilane mixtures.
S.N.Dzhenchel'skaya, K.A.Andrianov, Yu.K.Petrashko gave data on working results within the field of catalytical polymerization of the hydrolysis products of di- and tri-functional compounds.
M. Kučera (Czechoslovakia) reported on the investigations carried out of the polymerization of octamethylcyclotetrasyloxanes under the influence exercises by alkalis.
K.A.Rzhendzinskaya, I.K.Stavitskiy gave a report on the synthesis of chlorine-containing polysiloxane-caoutchouc SKT-Kh5.
N.B.Baranovskaya, A.A.Berlin et al. suggested compounds

Card 5/8

on the basis of liquid polydimethylsyloxane which solidify at normal temperature.
A.I.Glukhova, K.A.Andrianov et al. gave a report on obtaining a heat-stable rubber-like polymer.
A.V.Topchiyev, N.S.Nametkin and collaborators spoke about the polymerization of unlimited silicon hydrocarbons by making use of a complex catalyzer triethylaluminum tetrachloro-titanium.
A.A.Zhdanov and K.A.Andrianov spoke about the synthesis of polymers with anorganic chains.
V.V.Korshak, G.M.Frunze, E.V.Kukharskaya, D.N.Andreyev reported on polyamides which are composed from silicon-containing dicarboxylic acids and posses a higher elasticity than similar polymers.
L.M.Volkova, K.A.Andrianov and **assistants** reported on a method of increasing the mechanical strength of silicon-organic polymers.
I.Ya.Guretskiy, A.P.Kreshkov, and P.A.Andreyev under various conditions investigated the interaction of a number of silicon-organic compounds with cellulose nitrates.

Chemistry and the Practical Application of Silicon-Organic Compounds. All Union Conference at Leningrad SOV/30-58-12-39/46

N.P.Kharitonov, and B.N.Dolgov suggested a new material for heat-, moisture-, and electrical insulation on a silicon-organic basis to be used instead of enamel coatings.
B.A.Kiselev, Z.A.Zinov'yev et al. produced a hydrophobous glass Textolite on the basis of polyester compounds which contain silicon-organic compounds.
M.Ya.Borodin, Z.I.Kazakova, et al. obtained heat-resistant and solid foam material on the basis of a combination of silicon-organic polymers with phenol-formaldehyde- and epoxy-resins and nitrile rubber.
A.P.Kreshkov delivered a report on the successful analysis of silicon-organic compounds and gave an outline on further prospects of development.
S.V.Syavtsillo, A.P.Kreshkov, A.P.Terent'yev et al. investigated problems of the elaboration of control methods for the production of silicon-organic monomers and polymers.
I. Čermák, D. Šnobl, M. Dvořák (Czechoslovakia)

Card 7/8

discussed results obtained by the radiographic determination of the activity of silicon-copper alloys which are used for the direct synthesis of methyl chlorosilanes.
In conclusion D.P.Novikov spoke about the present stage of development and the plans for the further development of the production of silicon-organic compounds in the USSR. A number of measures was recommended for the further development of the discussed field of science and technical engineering. A committee was elected for the purpose of working out the nomenclature of silicon-organic compounds. The authors of the present article regret that no reports were delivered at the conference on the theory of technological processes or on the calculation and design of chemical apparatus.

SOV/79-28-7-33/64

AUTHORS: Davydova, V. P., Voronkov, M. G.

TITLE: Investigation in the Field of Alkoxy-Silanes (Issledovaniya v oblasti alkoksisilanov) XI. The Reaction of Dialkyl-Diacetoxy-Silanes With Glycerin and Pentaerythrite (XI. Reaktsiya dialkildiatsetoksisilanov s glitserinom i pentaeritritom)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 7, pp. 1879-1882 (USSR)

ABSTRACT: Earlier (Ref 2) the authors showed that the dialkyl-diacetoxy-silanes react with glycols under the formation of cyclic dialkyl-silanediolethers or their dimers (scheme 1). They were interested in extending this reaction to the polyvalent alcohols, especially to glycerin and pentaerythrite. It appeared that the above mentioned silanes easily enter reaction with pentaerythrite under the formation of the hitherto unknown spiro-cyclic ethers of the dialkyl-silanediols according to scheme 2. This way they obtained, for instance, by the conversion of pentaerythrite with dimethyldiacetoxysilane the crystallized 3,3,9,9-tetramethyl-2,4,8,10-tetraoxa-3,9-disilaspiro (5,5) undecane (Formula I). The reaction of

Card 1/3

SOV/79-28-7-33/64

Investigation in the Field of Alkoxy-Silanes. XI. The Reaction of Dialkyl-Diacetoxy-Silanes With Glycerin and Pentaerythrite

dialkyl-diacetoxy-silanes with glycerin proved to be more complicated. In using diethyl-diacetoxy-silane with glycerin the compound with the formula $C_{18}H_{40}O_6Si_3$ (II) was obtained, for which the structural formulae (IIa) and (IIb) are equally probable. However, in the reaction of dimethyl-diacetoxy-silane with glycerin the authors in all cases obtained an easily polymerizing product of the empiric formula $C_7H_{18}O_5Si$ with two hydroxyl groups and an acetoxy group combined with the carbon. According to the experimental data obtained this compound corresponds with regard to its structure to the formula (III), which fact is explained by the last scheme. There are 5 references, 5 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry, AS USSR)

SUBMITTED: June 6, 1957

Card 2/3

SOV/79-28-7-33/64

Investigation in the Field of Alkoxy-Silanes. XI. The Reaction of Dialkyl-Diacetoxy-Silanes With Glycerin and Pentaerythrite

1. Silicanes--Chemical reactions 2. Dithioglycerol--Chemical reactions
3. Pentaerythritol tetranitrate--Chemical reactions

Card 3/3

SOV/79-28-8-28/66

AUTHORS: Voronkov, M. G., Romanova, N. G.

TITLE: On the Thermal Addition of Trichlorosilane to Diolefine Hydrocarbons (Termicheskoye prisoyedineniye trikhlorsilana k diolefinovym uglevodorodam)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 8, pp. 2122-2128 (USSR)

ABSTRACT: In the previous paper of the authors (Ref 1) the thermal addition reaction of trichlorosilane to the mono-olefinic hydrocarbons was treated. It was interesting for them to extend this reaction to various diolefine hydrocarbons. There are several indications in publications, mostly of patent character, that a thermal and catalytic addition of the trichlorosilane to diolefine hydrocarbons is possible (Refs 2-8). The authors were, however, not able to add the trichlorosilane to a series of diolefines which contain conjugated double bonds in the presence of organic peroxides or under ultraviolet irradiation, since a polymerization of the initial hydrocarbons took place more rapidly than the addition of the trichlorosilane to the double bond. Nevertheless, the reaction could be carried out in the most cases successfully without catalysts at high tem-

Card 1/3

SOV/79-28-8-28/66

On the Thermal Addition of Trichlorosilane to Diolefine Hydrocarbons

peratures and under pressure. Thus the thermal addition of the trichlorosilane to diolefine hydrocarbons with isolated and conjugated double bonds was investigated. In the first case the addition reaction takes a normal course, according to the rule of free radical addition. The addition of the trichlorosilane to 1,3 cyclopentadiene and dicyclopentadiene leads mainly to the formation of bis (trichlorosilyl) cyclopentane. The reaction of the trichlorosilane with 1,3-cyclohexadi[illegible], and above all with dipentene, takes an abnormal course, under formation of a product of the addition to the corresponding cyclomono-olefine product of the "direct catalytic transformation" of the initial hydrocarbon. 6 additive compounds of the trichlorosilane to diolefines are described the most of which were newly synthesized. There are 2 tables and 17 references, 12 of which are Soviet.

ASSOCIATION: Leningradskiy gosudarstvennyy universitet i Institut khimii silikatov Akademii nauk SSSR (Leningrad State University and Institute of Silicate Chemistry, AS USSR)

Card 2/3

SOV/79-28-8-28/66

On the Thermal Addition of Trichlorosilane to Diolefine Hydrocarbons

SUBMITTED: July 4, 1957

Card 3/3

VORONKOV, M.; ROMANOVA, N.; SMIRNOVA, L.

"Thermal addition of trichlorosilane to mono-olefins."

p.640 (Vol. 52, no. 4, Apr. 1958, Praha, Czechoslavakia)

Monthly Index of East European Accession (EEAI) LC, Vol. 7, No. 8, August 1958

SOV/79-28-8-27/66

AUTHORS: Voronkov, M. G., Davydova, V. P., Lazarev, A. N.

TITLE: Investigations in the Field of Alkoxy Silanes (Issledovaniya
v oblasti alkoksisilanov) XII. Hexa-Tert.-Butoxycyclo Tri-
siloxane (XII. Geksa-tret.-butoksitsiklotrisiloksan)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol. 28, Nr 8, pp. 2128-2131
(USSR)

ABSTRACT: Polydialkoxy-cyclosiloxanes $[(RO)_2SiO]_n$ have hitherto been synthesized according to the hydrolysis reaction (Refs 1-4),

$$n(RO)_2SiX_2+nH_2O \longrightarrow [(RO)_2SiO]_n + 2nHX,$$ where $R=C_2H_5, C_4H_9;$

$X = Cl, OC_2H_5, NH_2;$ $n=3-8$.

The authors observed a very interesting case of formation of a previously unknown hexa-tert.-butoxy-cyclotrisiloxane by the way of an intermolecular condensation of the tri-tert.-butoxy-acetoxysilane in a tert.-butyl alcohol medium in the presence of tert.-sodium butylate according to scheme 2. Such a reaction under formation of a siloxane compound at

Card 1/3

SOV/79-28-6-19/66

Investigations in the Field of Alkoxy Silanes.
XII. Hexa-Tert.-Butoxycyclo Trisiloxane

the expense of the alkoxy- and acyloxy silane condensation (Scheme 3) has hitherto been known to an only small extent in the organosilicon chemistry. The main product of the above mentioned reaction is the completely resistant cyclic trisiloxane and not, as expected, the tetrasiloxane which circumstance is assumed to be caused by the influence of the tert.-butoxy groups. Hexaethoxy-cyclotrisiloxane is, in contrast to octaethoxy-cyclotetrasiloxane, obtained only with difficulty and is very unstable. The infrared absorption spectrum of the hexa-tert.-butoxy-cyclotrisiloxane was investigated. Its interpretation permits to draw several conclusions on the structure of the cycle. This is shown by the figure. There are 1 figure and 22 references, 9 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR
(Institute of the Chemistry of Silicates, AS USSR)

SUBMITTED: July 11, 1957

Card 2/3

SOV/79-28-8-29/66

Investigations in the Field of Alkoxy Silanes.
XII. Hexa-Tert.-Butoxycyclo Trisiloxane

Card 3/3

VORONKOV, M.G., kand.khim.nauk; LOSEV, V.B.

Chemistry and practical applications of silicon organic compounds; All-Union conference in Leningrad. Vest.AN SSSR 28 no.12: 97-100 D '58. (MIRA 11:12)
(Silicon organic compounds)

SOV/79-28-12-30/41

AUTHORS: Borisov, S. N., Voronkov, M. G., Dolgov, B. N.

TITLE: On Reactions of Alkyl Dichloro Silanes With Chlorohydrocarbons (O reaktsiyakh alkildikhlorsilanov s khloruglevodorodami)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 12, pp 3292-3297 (USSR)

ABSTRACT: According to reference 1 methyl dichloro silane reacts with chlorobenzene at 150-300° in the presence of $AlCl_3$ according to the scheme

$$CH_3SiHCl_2 + C_6H_5Cl \longrightarrow CH_3(ClC_6H_4)SiCl_2 + H_2.$$

At high temperature without catalyst two parallel reactions take place (I and II). According to references 4 and 5 the reaction of the alkyl dichloro silanes with allyl and methallyl chloride (with 1 % platinum) takes place mainly according to scheme 4, with by-products also occurring. As is known, the formation of alkyl trichloro silanes and dialkyl dichloro silanes in the reaction of $RSiHCl_2$ with halogen olefins (Ref 5) is explained according to schemes (V) and (VI). Based on what was said the authors were interested in investigating more in detail the reaction (V) hitherto experimentally not dealt with. The reaction of ethyl dichloro silane with

Card 1/3

n.-propyl, isopropyl and allyl chloride was carried out. This reaction $C_2H_5SiHCl_2 + RCl \xrightarrow{AlCl_3} C_2H_5SiCl_3 + RH$ (VII), where $R=C_3H_5$, iso- and n.-C_3H_7, takes place very slowly at room temperature, however, very rapidly at 50-60°. From the table it may be seen that in the case of isopropyl chloride, where the chlorine atom is more movable than in n.-C_3H_7Cl, the transformation of ethyl dichloro silane to ethyl trichloro silane is correspondingly higher. In the reaction (VII) in the aliphatic series the reactivity of the S-H bonds, and not that of the C-Cl bonds, plays a decisive role. It may be assumed that in the reaction (VII) first the mobile complex of ethyl dichloro silane with $AlCl_3$ is formed, which causes the transformation of ethyl dichloro silane into ethyl chloro silane. Thus, it was demonstrated that ethyl dichloro silane with aliphatic chlorohydrocarbons in the presence of $AlCl_3$ enters a combined hydrogenation - halogenation reaction, reduces them to the corresponding hydrocarbons and is transformed into ethyl trichloro silane itself. There are 1 table and 13 references, 12 of which

On Reactions of Alkyl Dichloro Silanes With Chlorohydrocarbons SOV/79-28-12-30/41

are Soviet.

ASSOCIATION: Leningradskiy gosudarstvennyy universitet i Institut khimii silikatov Akademii nauk SSSR (Leningrad State University and Institute of the Chemistry of Silicates of the Academy of Sciences, USSR)

SUBMITTED: November 20, 1957

Card 3/3

KALUGIN, N.V.; VORONKOV, M.G.

Waterproof soaking of cotton fabrics with silicon organic compounds.
Zhur. prikl. khim. 31 no.9:1390-1397 S '58. (MIRA 11:10)
(Silicon organic compounds) (Cotton fabrics)
(Waterproofing of fabrics)

AUTHORS: Orlov, N. F., Dolgov, B. N., Voronkov, M. G. SOV/20-122-2-22/42

TITLE: Tris(Triorganosilyl)-Vanadates (Tris(Triorganosilil)vanadaty)

PERIODICAL: Doklady Akademii nauk SSSR, 1958, Vol 122, Nr 2, pp 246 - 249 (USSR)

ABSTRACT: The organic derivatives of vanadium are little investigated, especially, because vanadium is not liable to form stable organic compounds with a vanadium-carbon bond(Ref 1). But also compounds in which a vanadium atom is combined with a carbon by means of oxygen are deficiently described (Ref 2). The present paper is an investigation of the synthesis of hitherto unknown organosilicon vanadium derivatives which contain a binding V-O-Si (see the title). The authors have produced these derivatives by interaction of triorganosilanes with V_2O_5, $VOCl_3$ or with trialkyl vanadates $OV(OR)_3$ as well as the reaction of the sodium triethyl silanolate. The first method is discussed. Another simple method of synthesis of triorganosilyl ethers of the ortho-vanadic acid is based

Card 1/3

Tris(Triorganosilyl)-Vanadates

SOV/20-122-2-22/42

on the reaction of vanadium oxychloride with triorgano-silanolene in the presence of hydrogen chloride-acceptors, (ammonia, tertiary amines, and others). The yield of ethers is 60-80%. A suitable synthesis of the silicoorganic ethers of the ortho-vanadic acid is the trans-etherification of the trialkyl vanadates by means of triorganosilanolene in the presence of catalytic amounts of sodium derivatives of the latters. They are formed by addition of metallic sodium. Finally, the compounds in question can be produced with a good yield by the reaction of sodium triorganosilanolates with vanadium oxychloride. The properties of these compounds are discussed and the constants tabulated in table 1. There are 1 table and 7 references, 4 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry, AS USSR)

PRESENTED: April 30, 1958, By A.N.Nesmeyanov, Member, Academy of Sciences, USSR

Card 2/3

VORONKOV, M.G.

PAGE

Khimiya i Primeneniye Fosfororganicheskikh Soyedineniy (Chemistry and Application of Organophosphorus Compounds) A. Ye. Arbuzov, Ed. publ. by Kazan' Affil, Acad. Sci. USSR, Moscow, 1962 632pp.

Collection of complete papers presented at the 1959 Kazan Conference on Chemistry of Organophosphorus Compounds.

VORONKOV, M. G.

N. V. Kalugina andM. G. Voronkov, "The Application of Silicon-Organic Substances for Water-repulsing Impregnation."

Report presented at the Second All-Union Conference on the Chemistry and Practical Application of Silicon-Organic Compounds held in Leningrad from 25-27 September 1958.
Zhurnal prikladnoy khimii, 1959, Nr 1, pp 238-240 (USSR)

VORONKOV, M. G.

N. F. Orlov, B. N. Dolgov and M. G. Voronkov, "The New Synthesis Methods of Trialkyl-(aryl)-Siloxiderivative Elements of the III, IV and V Groups of the Periodic System."

Report presented at the Second All-Union Conference on the Chemistry and Practical Application of Silicon-Organic Compounds held in Leningrad from 25-27 September 1958.
Zhurnal prikladnoy khimii, 1959, Nr 1, pp 238-240 (USSR)

VORONKOV, M. G.

N. A. Afonchikov, G. V. Kolobava, P. N. Mikhaylov, and M. G. Voronkov, "Their Application for Glueing Paper."

Report presented at the Second All-Union Conference on the Chemistry and Practical Application of Silicon-Organic Compounds held in Leningrad from 25-27 September 1958.
Zhurnal prikladnoy khimii, 1959, Nr 1, pp 238-240 (USSR)

VORONKOV, M.G., kand.khim.nauk; SHOROKHOV, N.V., inzh.

Using silicon organic compounds for increasing the water resistance and prolonging the life of building materials. Stroi. mat. 5 no.7:12-17 Jl '59. (MIRA 12:10)
(Waterproofing) (Silicon organic compounds)

SOV/62-59-5-30/40

5(3)

AUTHORS: Orlov, N. F., Voronkov, M. G.

TITLE: Trialkyl-ortho-vanadates (Trialkilortovanadaty)

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 5, pp 933-934 (USSR)

ABSTRACT:

In accordance with papers by Prandtl and Hess (Ref 2) the synthesis of trialkylvanadate from V_2O_5 with alcohols is carried out according to the reaction equation $V_2O_5 + 6ROH \rightleftarrows 2OV(OR)_3 + 3H_2O$. As this reaction is reversible by the formation of water, it was necessary to take measures in order that water be removed. This was done by a continuous azeotropic distillation with benzene. In this way a synthesis of 6 trialkyl-ortho-vanadates was successfully carried out, the isomeric butyl- and amyl esters of orthovanadic acid, two of which had hitherto been unknown. This method does not produce a very large yield (20-25%), but is very simple and was therefore preferred by the authors also to the synthesis from $VOCl_3$, which is known from publications. Mention is then made of the fact that in the reaction of V_2O_5 with triorgano-

Card 1/2

SOT/62-59-5-30/40

Trialkyl-ortho-vanadates

silanoles taking place at similar conditions the corresponding silicon-organic esters, the tris-(triorganosilyl)-orthovanadates are formed with a good yield. The table gives the physical data of the synthetized compounds. There are 1 table and 6 references, 2 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR)

SUBMITTED: October 21, 1958

Card 2/2

SOV/62-59-6-10/36

5(4)

AUTHORS: Ryskin, Ya. I., Voronkov, M. G., Shabarova, Z. I.

TITLE: The Infrared Absorption Spectrum of Triethyl Silanole-d, $(C_2H_5)_3SiOD$ (Infrakrasnyy spektr pogloshcheniya trietilsilanola-d, $(C_2H_5)_3SiOD$)

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 6, pp 1019 - 1024 (USSR)

ABSTRACT: Two syntheses for the preparation of trialkylsilanole-d_1 and dialkylsilandiole-d_2 were worked out. They consist in an addition of etheric- or dioxane ether solutions of D_2O to a mixture of trialkyl chlorosilane, tertiary amine, and ethers, which was cooled down to 0°. In the experimental part the synthesis is described still more in detail. The infrared spectrum was taken from the synthesized triethyl silanole (TES). Figure 1 represents the spectrum of the pure (TES) a) and of (TES) dissolved in CCl_4 b). For the purpose of comparison, also the spectra of $(C_2H_5)_3SiOH$ and $(C_2H_5)_3SiOX$ with X=F, Cl, NH_2, CH_3, C_2H_5 were recorded.(Fig 2). The fundamental oscillation

Card 1/2

The Infrared Absorption Spectrum of Triethyl Silanole-d, SOV/62-59-6-10/36
$(C_2H_5)_3SiOD$

frequencies in the oscillation range of from 4000-700 cm^{-1} were assigned to the different bonds in the compounds. The ratio of the isolated oscillation frequencies of the groups OH and OD was 1.34. The authors thank Ye. F. Grcss for discussing the results obtained. There are 2 figures and 18 references, 6 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Chemistry of Silicates of the Academy of Sciences, USSR)

SUBMITTED: July 27, 1957

Card 2/2

SOV/62-59-8-11/42

5(3)

AUTHORS: Dolgov, B. N., Orlov, N. F., Voronkov, M. G.

TITLE: New Titaniumsilicon-organic Compounds - Trialkylsiloxy-titanium-halides

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 8, pp 1408-1411 (USSR)

ABSTRACT:

In the present paper a report is made on the syntheses of silicon-organic titaniferous compounds. These compounds are hardly known in publications. Mention is made of Andrianov, Ganina and Khrustaleva (Ref 2) who described the synthesis of polyorgano-titanium siloxanes. The silicon-organic titaniferous compounds can be obtained according to the following pattern by the effect of hexaalkyl disiloxanes on titanium tetrachloride:

$$nR_3SiOSiR_3 + TiCl_4 \longrightarrow (R_3SiO)_nTiCl_{4-n} + nR_3SiCl \quad n=1-2$$

The reaction takes place as soon as the materials have been heated for some time. A reflux cooler is used. 7 different compounds of the general type $(R_3SiO)_n TiX_{4-n}$ X=halogen were synthesized. The individual synthesis processes are described in the experimental part. The analysis of the com-

Card 1/2

SOV/62-59-8-11/42

New Titaniumsilicon-organic Compounds - Trialkylsiloxy-titanium-halides

pounds obtained was carried out by Yu. N. Platonov. If heating is continued over some time, the compounds tend to decompose. There are 1 table and 15 references, 4 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR)

SUBMITTED: December 16, 1957

Card 2/2

SOV/62-59-8-36/42

AUTHORS: Orlov, N. F., Voronkov, M. G.

TITLE: Tris(trialkylsilyl)-antimonites

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 8, p 1506 (USSR)

ABSTRACT: The silicon-organic esters of antimonic acid which have been unknown up to now were synthesized by the authors according to 2 different methods: (1) by means of an azeotropic separation of H_2O from a mixture of trialkylsilane and Sb_2O_3 : $6R_3SiOH + Sb_2O_3 \leftrightarrows 2(R_3SiO)_3Sb + 3H_2O$. The $(R_3SiO)_3Sb$ yields according to this method were up to 70%. (2) By means of an esterification of the trialkylantimonites with triorganosilanes $(R'O)_3Sb + 3R_3SiOH \leftrightarrows (R_3SiO)_3Sb + 3R'OH$. In this case the yields were up to 90%. Thus the compounds $[CH_3(C_2H_5)_2SiO]_3Sb$, boiling point 160° (5mm Hg); $[(C_2H_5)_3SiO]_3Sb$, boiling point 170° (3mm Hg) were obtained. The refraction of the Sb-O binding (5.25 ml) was also determined for the first time. More exact data will be published.

Card 1/2

SOV/62-59-8-36/42

Tris(trialkylsilyl)-antimonites

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry, Academy of Sciences, USSR)

SUBMITTED: May 11, 1959

Card 2/2

RYSKIN,Ya.I.; VORONKOV,M.G

Vibration spectraof molecules of $(C_2H_5)_3SiX$ type. Coll Cz chem 25 no.12:3816-3828 '59. (MIRA 9:6)

1. Institut khimii silikatov Akademii nauk, SSSR, Leningrad.
(Ethyl group) (Spectrum analysis) (Raman effect)
(Molecules) (Silicon) (Silcates)
(Absorption spectra)

5 (3)

SOV/79-29-3-33/61

AUTHOR: Voronkov, M. G.

TITLE: Investigations in the Field of Alkoxy Silanes (Issledovaniya v oblasti alkoksisilanov). XIII. Reaction of the Siloxanes With Alkoxy Silanes. A New Method of Synthesizing Alkoxy Silanes and Siloxanes (XIII. Vzaimodeystviye siloksanov s alkoksisilanami. Novyy metod sinteza alkoksisilanov i siloksanov)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 3, pp 907-915 (USSR)

ABSTRACT: The author previously (Ref 2) synthesized organoalkoxy silanes by cleavage of the organosiloxanes with alcohols in the presence of alkaline catalysts according to the general scheme 1, under conditions which make it possible to remove the water formed by dehydrating agents. For this purpose the author used above all tetraalkoxy silanes which can be easily hydrolyzed in alcoholic-alkaline medium by water (Ref 3):

$$Si(OR)_4+4H_2O \longrightarrow 4ROH+Si(OH)_4 \longrightarrow SiO_2+2H_2O .$$

The formation of dimethyl diethoxy silane from polydimethylsiloxane and ethyl alcohol in the presence of silicon ethyl ether can thus be represented by scheme 3. By summing up these equations the following general scheme

Card 1/3

SOV/79-29-3-33/61

Investigations in the Field of Alkoxy Silanes. XIII. Reaction of the Siloxanes With Alkoxy Silanes. A New Method of Synthesizing Alkoxy Silanes and Siloxanes

results:

$$2\left[(CH_3)_2SiO\right]_n \; nSi(OC_2H_5)_4 \xrightarrow{KOH} 2n(CH_3)_2Si(OC_2H_5)_2 + (SiO_2)_n \,.$$

It follows from it that the alcohol need practically not take part in the reaction and that dimethyl diethoxy silane is formed by the reaction of polydimethyl siloxane with tetraethoxy silane. The experiments completely confirm this assumption. Thus the simple distillation of the mixture of normal, branched, and cyclic organosiloxane with tetra- or organoalkoxy silane in the presence of an alkaline catalyst (e.g. KOH) yields the corresponding organoalkoxy silane. This new and convenient method is of preparative importance. The reaction in the case of $Si(OR)_4$ can be illustrated by scheme 5, where n=2-3 and m can vary in wide limits. The dialkyl-dialkoxy- and trialkylalkoxy silanes synthesized in the above mentioned way are listed in table 1.

Card 2/3

SOV/79-29-3-33/61

Investigations in the Field of Alkoxy Silanes.
XIII. Reaction of the Siloxanes With Alkoxy Silanes. A New Method of Synthesizing Alkoxy Silanes and Siloxanes

The possibility of an inter- and intramolecular rearrangement of the bonds Si-O-Si and Si-O-C was thus shown and their mechanism suggested. 20 compounds of this type were obtained. The disproportionation of normal and cyclic polyalkoxy siloxanes was investigated. There are 2 tables and 17 references, 7 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR)

SUBMITTED: February 4, 1958

Card 3/3

SOV/79-29-5-27/75

5(3)

AUTHORS: Voronkov, M. G., Shabarova, Z. I.

TITLE: Investigations in the Field of Alkoxy-silanes (Issledovaniya v oblasti alkoksisilanov).
14. Cleavage of Organosiloxanes With Alcohols as Method of Synthesizing Organoalkoxy-silanes (14. Rasshchepleniye organosiloksanov spirtami kak metod sinteza organoalkoksisilanov)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 5, pp 1528-1534 (USSR)

ABSTRACT: It is known (Refs 3 - 9) that organosiloxanes are cleaved by hydroxides of alkali metals with the corresponding silanolates and silanols being formed. There are further indications (Refs 10 - 12) that silanols react with alcohols and yield alkoxy-silanes. In order to devise a method for synthesizing organoalkoxy-silanes by means of cleavage of organosiloxanes with alcohols in the presence of alkali, the two reaction mentioned were combined in the work under review. The scheme of this new method can be demonstrated in a general form by the equation (4)

$$\geqslant\text{SiOSi}\leqslant + 2\text{ROH} \xrightleftharpoons{\text{MOH}} 2 \geqslant\text{SiOR} + H_2O \qquad (4)$$

M - atom of the alkali metal. By the application of this new

Card 1/3

SOV/79-29-5-27/75

Investigations in the Field of Alkoxy-silanes.
14. Cleavage of Organosiloxanes With Alcohols as Method of Synthesizing Organoalkoxy-silanes

method it was possible to obtain 35 organoalkoxy silanes $R'_nSi(OR)_{4-n}$ (Table). n - 1,2 and 3; R' - CH_3 or C_6H_5; R - primary (normal or iso) or secondary alkyl radical with 4-12 carbon atoms. 8 of these compounds were synthesized for the first time. The reaction mechanism of the formation of organoalkoxy-silanes from organosiloxanes and alcohols can be illustrated by the following scheme: $\underset{\overset{\uparrow}{OR^-}}{\geqslant Si} \curvearrowleft O - Si\leqslant \rightleftarrows \geqslant SiOR + {}^-O - Si\leqslant$ (5)

$$\geqslant SiO + ROH \rightleftarrows \geqslant SiOH + RO^- \qquad (6)$$

$$\geqslant Si - OH + ROH \rightleftarrows \geqslant SiOR + HOH \qquad (7)$$

In addition to a further reaction of silanol with alcohol (7) also the condensation not desired (8) is possible.

$$2 \geqslant SiOH \longrightarrow \geqslant SiOSi\leqslant + H_2O \qquad (8)$$

There are 1 table and 18 references, 7 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR)

Card 2/3

SOV/79-29-5-28/75

5(3)

AUTHORS: Voronkov, M. G., Chudesova, L. M.

TITLE: Cleavage of Siloxanes With Halogen-silanes, Reported on May 14, 1958 at the Symposium for Organosilicon Chemistry in Dresden (Rasshchepleniye siloksanov galogensilanami) New Synthesis Method of Organosiloxanes (Novyy metod sinteza organosiloksanov)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 5, pp 1534-1541 (USSR)

ABSTRACT:

The present paper is a further development of the initiated
investigations (Refs 1,2,7,8,17,20-26) of the cleavage of
organosiloxanes by means of electro- and nucleophilic reagents. The
possibility of a cleavage of the siloxane bond by means of halogen-
or organohalogen silanes according to the general scheme
$(4 - n)R_3SiOSiR_3 + R'_nSiX_{4-n} \rightleftarrows R'_nSi(OSiR_3)_{4-n} + (4 - n)R_3SiX$ is
demonstrated. n = 0 - 3, X = halogen, R and R' = equal or different
organic (aliphatic and aromatic) or organosilicon radicals, H,
halogen, etc. This reaction which represents a rearrangement of the
Si-O-Si and Si-X bonds is a new, convenient and simple method of
synthesizing different organosiloxanes. By this method 14 compounds
were synthesized. 6 of them were so far unknown (Table 1). This

Card 1/3

SOV/79-29-5-20/75

Cleavage of Siloxanes With Halogen-silanes, Reported on May 14, 1958 at the Symposium for Organosilicon Chemistry in Dresden. New Synthesis Method of Organosiloxanes

reaction is in equilibrium. The reaction mechanism of the cleavage of siloxanes with halogen silanes in the presence of ferric chloride can be illustrated by the following scheme:

$$R'_3SiX + FeX_3 \rightleftarrows R'_3Si^+ + FeX_4^-$$

$$R_3SiOSiR_3 + R'_3Si^+ \rightleftarrows (R_3Si)_2\overset{+}{O}-SiR'_3 \rightleftarrows R_3SiOSiR'_3 + R_3Si^+$$

By the interaction of the R_3Si^+ ions formed in this way with FeX_4^- anion the catalyst is regenerated and a new halogen silane formed. In the course of the reaction the catalyst gradually loses its activity and must always be regenerated. The total consumption of the catalyst amounts up to 5-8 mole%. The cleavage of organosiloxanes with halogen silanes is catalyzed by electrophilic halides of the elements (L'yuis (Lewis) acids) and first of all by ferric chloride. This new reaction offers the possibility of an easy and simple synthesis of a number of various organosiloxanes (as well as of organohalogen siloxanes), among them also difficultly accessible ones. Table 2 - physical properties of the

Card 2/3

Cleavage of Siloxanes With Halogen-silanes, Reported on May 14, 1958 at the Symposium for Organosilicon Chemistry in Dresden. New Synthesis Method of Organosiloxanes

SOV/79-29-5-28/75

initial compounds. All organosiloxanes synthesized were analyzed by Yu. N. Platonov. There are 2 tables and 31 references, 18 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR)

SUBMITTED: April 16, 1958

Card 3/3

SOV/80-32-2-42/56

AUTHORS: Afonchikov, N.A., Kolobova, G.V., Mikhaylov, P.N., Voronkov, M.G.

TITLE: The Application of Silicon-Organic Compounds for the Gluing of Paper (Primeneniye kremneorganicheskikh soyedineniy dlya prokleyki bumagi)

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol XXXII, Nr 2, pp 445-446 (USSR)

ABSTRACT: Silicon-organic compounds were used a) for treating the finished paper with vapors of methyltrichlorosilane; b) for impregnating the paper by these compounds; c) for gluing the paper mass by such substances. The last procedure shows the best results. The compound MN-1 $(CH_3SiHO)_n$ is most efficient. Thermal processing of the finished paper is necessary, however, in order to obtain a great depth of gluing. If certain catalysts are used, e.g. lead or zinc acetate, triethanolamine, etc, thermal processing is not necessary. The catalyst is also added to the paper mass where it has the best effect. Professor B.N. Dolgov is mentioned in the article.

Card 1/2 There is 1 table.

SOV/80-32-2-42/56

The Application of Silicon-Organic Compounds for the Gluing of Paper

ASSOCIATION: Fabrika "Goznak" i institut khimii silikatov AN SSSR (Factory "Goznak" and the Institute of the Chemistry of Silicates of the USSR Academy of Sciences)

SUBMITTED: April 22, 1958

Card 2/2

5(3) SOV/80-32-5-31/52

AUTHORS: Voronkov, M.G., Davydova, V.P., Grishanina, N.P.

TITLE: The Hydrophobization of Paper by Alkylacetoxysilanes and -Siloxanes. Communication V.

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol 32, Nr 5, pp 1106-1112 (USSR)

ABSTRACT: Acetoxysilanes cited in Tables 1 and 2 were used for the hydrophobization of paper. The water-resistance of the paper increases, if it is kept longer in 2.5 - 10%-solutions of methyltriacetoxysilane. The optimum are 2.5 - 5% solutions and 10 min. For ethylacetoxysilane the concentration must be above 5% and the time more than 10 min. Phenyl- and n-butyl-triacetoxysilane are more effective than methyltriacetoxysilane. The effect of tetraacetoxysilane disappears with the time due to the esterification of the hydroxyl groups, which form orthosilicic cellulose esters. A mixture of 20% of methylalkoxydiacetoxysilane and 80% of methyltriacetoxysilane increases the hydrophobic properties and improves the quality of the treated material. The hydrophobization by the preparations A-12 and A-16 increases the water-resistance of filter paper from 2 to 100-110 cm, of wrapping paper from 15 to 108-119 cm and of kraft paper from 132 to 190-220 cm.

Card 1/2

SOV/80-32-5-31/52

The Hydrophobization of Paper by Alkylacetoxysilanes and -Siloxanes. Communication V.

In all experiments the treated paper was heated finally for 3 hours to 105-110°C. To reduce this long time the ethyl ether of the orthotitanic acid was used as a catalyst. It reduced the time to 10-30 min at 105-110°C and to 2 min at 150°C. For hydrophobization 2-5%-solutions of the preparations A-12 and A-16 are recommended. There are: 6 tables, 2 graphs and 2 Soviet references.

ASSOCIATION: Institut khimii silikatov AN SSSR (Institute of the Chemistry of Silicates of the AS USSR)

SUBMITTED: November 27, 1957

Card 2/2

5(3) SOV/20-125-3-24/63

AUTHORS: Voronkov, M. G., Davydova, V. P.

TITLE: Photochemical Chlorination of Organochlorosilanes by Sulphuryl Chloride (Fotokhimicheskoye khlorirovaniye organokhlorsilanov khloristym sul'furilom)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 125, Nr 3, pp 553-556 (USSR)

ABSTRACT: The chlorination mentioned in the title which is not carried out photochemically is used to a great extent in the organic synthesis (Ref 1). It is catalyzed by organic peroxides or halides of several elements. Against this the photochemical reaction of organic compounds with SO_2Cl_2 is used only as its sulfochlorination reaction (Ref 2). The use of SO_2Cl_2 for the introduction of chlorine atoms into the alkyl- or aralkyl radicals connected with a Si-atom which is described in many papers is limited in the case of the chlorination of silicon organic compounds. Under normal conditions the methyl- (Refs 4, 7,9) or phenyl groups (Ref 4) connected with the Si-atom are not chlorinated by SO_2Cl_2 in the presence of benzoyl peroxide.

Card 1/4

SOV/20-125-3-24/63

Photochemical Chlorination of Organochlorosilanes by Sulphuryl Chloride

Though the chlorination of methyl derivatives in the medium of a high-boiling solvent (Refs 7,11,12) is successful, only a small yield is obtained by it. The authors observed that an ultraviolet irradiation catalyzes the last mentioned reaction to a much greater extent than organic peroxides. Thus the use of SO_2Cl_2 for the chlorination under comparatively mild conditions is made possible even in the case of compounds which can be chlorinated only with difficulty, like methyl trichlorosilane and methyl dichlorosilane. Ethyl trichlorosilane and the higher alkylchlorosilanes react as easily with SO_2Cl_2 at ultraviolet irradiation. An additional communication on this topic follows. An intensive irradiation of a boiling mixture of methyl trichlorosilane and SO_2Cl_2 leads to the formation of mono-, di-, and trichloro-derivatives (Table 1). This procedure is recommended especially for the production of $CHCl_2SiCl_3$. In the presence of $FeCl_3$ neither a photochemical nor a dark chlorination takes place. Quinoline, on the other hand, is a catalyst

Card 2/4

SOV/20-125-3-24/63

Photochemical Chlorination of Organochlorosilanes by Sulphuryl Chloride

of this reaction. $FeCl_3$ is a good catalyst (it is an inhibitor in the case of CH_3SiCl_3) for the photochemical chlorination of phenyl trichlorosilane, i.e. in the dark (Table 3), though with lower velocity than that of the photochemical reaction. Dimethyl dichlorosilane reacts with SO_2Cl_2 considerably more quickly than methyl trichlorosilane (Table 2). Mono- as well as dichloro-derivatives may be obtained with a yield of 45-50% according to the ratio of the initial reagents. Phenyl trichlorosilane is slowly chlorinated photochemically by SO_2Cl_2. The yield of chlorine phenyl trichlorosilane is not high (approximately 15%). In all cases hexachlorobenzene is the main product. Therefore the $Si-C_6H_5$ bond is ruptured and the chlorobenzene (or perhaps benzene) which is split off is chlorinated. Quinoline is an inhibitor (2 mole-%) of the photochemical chlorination of phenyl chlorosilane by SO_2Cl_2. The boiling of benzene with SO_2Cl_2 in the dark does not lead to an interaction in the presence or absence of benzoyl peroxide. There are 4 tables and 22 ref-

Card 3/4

erences, 7 of which are Soviet.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR)

PRESENTED: November 5, 1958, by A. V. Topchiyev, Academician

SUBMITTED: October 29, 1958

DOLGOV, B.N. [deceased]; SERGEYEVA, Z.I.; ZUBKOVA, N.A.; MATVEYEVA, N.M.;
VORONKOV, M.G.

Organosilicon esters of oximes. Izv.AN SSSR Otd.khim.nauk
no.5:951 My '60. (MIRA 13:6)

1. Institut khimii silikatov Akademii nauk SSSR i Leningradskiy gosudarstvennyy universitet imeni A.A. Zhdanova.
(Oximes) (Silicon organic compounds)

87122

S/062/60/000/009/009/021
B023/B064

15.2220

AUTHORS: Orlov, N. F., Dolgov, B. N., and Voronkov, M. G.

TITLE: Tris(triorganosilyl)borate

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1960, No. 9, pp. 1607-1609

TEXT: In their development of new methods of synthesizing silicon elemental-organic compounds, the authors aimed at completing the previous methods of producing triorganosilyl esters of boric acid - tris(triorganosilyl)borate by new methods. Three new methods were developed. They distinguish themselves from the older ones by high yields (80-95%). The first two are based on the reactions of triorganosilanoles with boric anhydride or boric acid:

$$3R_3SiOH + B(OH)_3 \longrightarrow (R_3SiO)_3B + 3H_2O \qquad (1)$$

$$6R_3SiOH + B_2O_3 \longrightarrow 2(R_3SiO)_3B + 3H_2O \qquad (2)$$

The third method of synthesis is based on the interaction between triorganosilanes and boric acid in the presence of colloidal nickel:

Card 1/2

87112

Tris(triorganosilyl)borate

S/062/60/000/009/009/021
B023/B064

$$3R_3SiH + B(OH)_3 \longrightarrow (R_3SiO)_3B + 3H_2 \qquad (3).$$

The reactions (1) and (2) are based on an uninterrupted azeotropic distilling off of water from the mixture of triorganosilanoles with boric acid or with boric anhydride, containing an inert solvent (benzene). Tris(triorganosilyl)borate was produced according to scheme (3) by heating mixtures of triorganosilane and boric acid in the presence of colloidal nickel, forming in the interaction of nickel chloride with triorganosilane. 6 tris(triorganosilyl)borates were obtained by the three methods of synthesis. 4 of the compounds have hitherto been unknown. There are 1 table and 21 references: 8 Soviet, 6 US, 3 British, 1 German, and 3 Japanese.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences USSR)

SUBMITTED: May 4, 1959

Card 2/2

VORONKOV, M.G.; RABKINA, S.M.

1-Alkoxycyclohexenes. Izv. AN SSSR.Otd. khim. nauk no.12:2240-2242 D '60. (MIRA 13:12)

1. Institut khimii silikatov AN SSSR.
(Cyclohexene)

S/051/60/008/005/004/027
E201/E491

AUTHORS: Lazarev, A.N. and Voronkov, M.G.

TITLE: Vibrational Spectra of Alkoxysilanes and Siloxanes. III. Vibrations of Silicon-Oxygen Chains in the Spectra of Polyalkoxysiloxanes

PERIODICAL: Optika i spektroskopiya, 1960, Vol.8, No.5, pp.614-622

TEXT: The structure of polyalkoxysiloxane molecules resembles in many respects the structure of silicates. To establish a correlation between the structure and the vibrational spectra of polyalkoxysiloxanes and silicates two problems had to be solved. Firstly, it was necessary to identify the frequencies of normal vibrations of the SiO_4 group and this was done by investigation of the spectra of orthosilicates (Ref.1, 2) and the spectra of SiO_4 ions in solution (Ref.3); vibrations of the SiO_4 group in the spectra of tetraalkoxysilanes, $Si(OR)_4$, were discussed in an earlier communication (Ref.4). Secondly, it was necessary to study changes in the vibrations of silicon-oxygen tetrahedra on polymerization; this is dealt with in the present paper. The authors investigated the vibrational spectra of polymethoxysiloxanes and polyethoxysiloxanes with various numbers of SiO_4 tetrahedra

VC

Card 1/3

S/051/60/008/005/004/027
E201/E491

Vibrational Spectra of Alkoxysilanes and Siloxanes. III. Vibrations of Silicon-Oxygen Chains in the Spectra of Polyalkoxysiloxanes

in the molecular chain. Polymethoxysiloxanes and polyethoxysiloxanes were prepared by hydrolysis of large amounts (1 to 1.5 kg) of $Si(OCH_3)_4$ and $Si(OC_2H_5)_4$ in a medium of the appropriate alcohol and with HCl as a catalyser. Polyalkoxysiloxanes were separated by repeated fractional sublimation in vacuum in the presence of traces of phosphoric acid. Physical constants of the first members of polymethoxysiloxane and polyethoxysiloxane families are given in Table 1, together with results of chemical analysis of these compounds. The infrared absorption spectra of polymethoxysiloxanes are shown in Fig.1 and those of polyethoxysiloxanes in Fig.2. The vibrational frequencies of polymethoxysiloxanes are listed in Table 2 and those of polyethoxysiloxanes are given in Table 3. Table 4 gives the selection rules for vibrations of the SiO_4 and Si_2O_7 groups and Table 5 lists the valence vibrations of the Si_2O_7 groups. Analysis of the results obtained showed that changes in the spectra of

Card 2/3

S/051/60/008/005/004/027
E201/E491

Vibrational Spectra of Alkoxysilanes and Siloxanes.
III. Vibrations of Silicon-Oxygen Chains in the Spectra of Polyalkoxysiloxanes

polyethoxysiloxanes with increase of the degree of polymerization can be interpreted as due to changes in vibrations of the SiO_4 groups when the latter are joined into a chain. This makes it possible to use polyalkoxysiloxanes as organic "models" of silicates. There are 2 figures, 5 tables and 7 references: 3 Soviet, 2 English and 2 German.

SUBMITTED: August 11, 1959

Card 3/3

VORONKOV, M.G.; SHABAROVA, Z.I.

Alkoxysilanes. Part 15: Cleavage of hexaalkyldisiloxanes by phenols. New method of synthesizing trialkylsiloxy derivatives of aromatic hydrocarbons. Zhur.ob.khim. 30 no.6:1955-1958 Je '60. (MIRA 13:6)

1. Institut khimii silikatov Akademii nauk SSSR.
(Siloxanes) (Phenols) (Hydrocarbons)

82296

S/079/60/030/007/010/020
B001/B063

5.3700B

AUTHORS: Orlov, N. F., Voronkov, M. G.

TITLE: Tris(triorganosilyl)phosphates

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 7, pp. 2223-2229

TEXT: The present paper describes new syntheses of tris(triorganosilyl) phosphates on the basis of triorganosilanols, hexaorganodisiloxanes, or triorganosilanes. It was found that these phosphates are formed with a yield of 50% by a reaction of phosphoric anhydride with triorganosilanols according to the following scheme:

$12R_3SiOH + P_4O_{10} \longrightarrow 4(R_3SiO)_3PO + 6H_2O$ (1).

A side reaction, $6R_3SiOSiR_3 + P_4O_{10} \longrightarrow 4(R_3SiO)_3PO$ (2), is to be assumed because of the comparatively high tendency of triorganosilanols to intermolecular condensation with water separation, which is accompanied by the formation of hexaalkyldisiloxanes, with P_4O_{10} acting as a catalyst (Ref. 18). The mechanism of this reaction is illustrated by Scheme (3).

Card 1/3

82296
S/079/60/030/007/010/020
B001/B063

Tris(triorganosilyl)phosphates

Another synthesis of tris(triorganosilyl)phosphates (yield of 90%), worked out by the authors, is based on a reaction of triorganosilanols with orthophosphoric acid: $3R_3SiOH + H_3PO_4 \rightleftarrows (R_3SiO)_3PO + 3H_2O$ (4) in which the water must be distilled off (Ref. 22). A mechanism of this reaction is suggested in Scheme (5). An attempt to synthesize tris(triethylsilyl) phosphate according to Scheme (7) was unsuccessful, whereas orthophosphoric acid could be reacted with hexaalkyl disiloxanes according to Scheme (8). The mechanism of this reaction is shown in Scheme (9). A new interesting synthesis of tris(trialkylsilyl)phosphates with colloidal nickel acting as a catalyst is shown in reaction (10) in which a side reaction occurs, so that a yield of only 60% is obtained. All tris(triorganosilyl)phosphates obtained are only stable in dry air, whereas moist air splits the SiOP bond. The synthesized products are tabulated. A mechanism of the catalytic action of colloidal nickel is also suggested. There are 1 table and 26 references: 13 Soviet, 7 US, 1 Italian, 3 German, and 2 British.

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Card 2/3

82296

Tris(triorganosilyl)phosphates

S/079/60/030/007/010/020
B001/B063

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences USSR)

SUBMITTED: July 11, 1959

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Card 3/3

84878

5.3700 only 2209.1273

S/079/60/030/010/019/030
B001/B066

11.1250

AUTHORS: Dolgov, B. N. (Deceased), Sergeyeva, Z. I., Zubkova, N. A. and Voronkov, M. G.

TITLE: Organosilicon Ethers of Aliphatic Aldoximes

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 10, pp. 3347 - 3352

TEXT: On the basis of Ref.1, the authors tried to synthesize compounds in which the nitrogen is separated from the silicon by some other elements, to investigate their stability to hydrolysis. The present paper deals with the synthesis of organosilicon ethers of oximes. Taking into account the papers of Refs. 3-7, the authors found the trialkyl-chloro silanes to react with aliphatic aldoximes in the presence of pyridine according to the following Scheme:
$R_3SiCl + HON = CHR' + C_5H_5N \longrightarrow R_3SiON = CHR' + C_5H_5N.HCl$. This reaction already proceeds at room temperature and lasts for 4-5 hours with continuous stirring. The yields of trialkyl-silyl ethers of aldoximes

Card 1/3

21876

Organosilicon Ethers of Aliphatic Aldoximes S/079/60/030/010/019/030
B001/B066

were 52.5-80%. In addition to the main product, the hydrochloride of pyridine was separated out in yields of 60-100%. O-trialkyl-silyl-aldoximes are stable liquids (Table), soluble in ordinary solvents, and distillable at standard pressure. To determine the structure of the resultant products, they were reduced with platinum black, and then hydrolyzed. The catalytic hydrogenation of $(CH_3)_3SiON = CH\text{-n-}C_3H_7$ and $(C_2H_5)_3SiON = CH\text{-iso-}C_3H_7$ did not yield O-trialkyl-silyl-N-alkyl-hydroxylamines, but amines, ammonia, and the corresponding trialkyl-silanols, which indicates a cleavage of the O-N bond. Hydrogenation thus proceeds in the same way as the reduction of the O-alkyl ethers of oximes (Ref.6). According to K. W. Rosenmund (Ref.10) and Vasil'yev (Ref.11), the primary amine may be catalytically converted into a mixture of ammonia and primary, secondary, and tertiary amines. Contrary to this reduction, that of N-alkyl oximes readily yields N,N-dialkyl-hydroxylamines, both on $LiAlH_4$ and on a platinum catalyst (Ref.6). O-trialkyl-silyl-aldoximes can be hydrolyzed only with 5% potash lye (90°C), but 60% of the starting material remains unchanged. Hydrolysis in an acid medium gives aldehydes, oximes, and a resin containing nitrogen.

Card 2/3

84878

Organosilicon Ethers of Aliphatic Aldoximes S/079/60/030/010/019/030
B001/B066

The structure of the eight compounds (Table) has thus been proven by reduction and hydrolysis. Their infrared spectra confirm the above-mentioned results. There are 1 table and 20 references: 11 Soviet, 1 US, 5 German, 2 British, and 3 Czechoslovakian.

ASSOCIATION: Leningradskiy gosudarstvennyy universitet (Leningrad State University)

SUBMITTED: November 5, 1959

Card 3/3

80064
S/020/60/132/01/38/064
B011/B126

5.3630

AUTHORS: Sanin, P. I., Voronkov, M. G., Shepeleva, Ye. S., Ionin, B. I.

TITLE: The Interaction Between Dialkyl-phosphorous Acids and Quinones

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 132, No. 1, pp. 145-148

TEXT: The organophosphorus compounds are highly active as additions to lubricating oils (Refs. 1-3). Some derivatives of dithiophosphorous, phosphoric, and phosphorous acids belong to them. The authors have taken the trouble to obtain organophosphorus compounds which are, amongst other things, also anti-oxidants, which hinder the oxidation of hydrocarbons by atmospheric oxygen. Thus, the authors tried to add acid esters of the phosphorous acid to the quinones. The reaction of dialkyl-phosphorous acids or phosphites with p-benzoquinone can take place in two ways and lead to: a) esters of dihydroxyphenylphosphoric acids (I) and (II), or b) compounds in which phosphorus is bonded with oxygen (III) and (IV) (Ref. 11). The authors have established that dialkyl-p-oxyphenylphosphates are formed on the reaction of dialkylphosphorous acids with p-benzoquinone. As a result, the phosphorous group adds to the oxygen atom of the benzoquinone (see scheme). This addition is accompanied by a conversion of the

4

Card 1/3

80064

The Interaction Between Dialkyl-phosphorous Acids and Quinones

S/020/60/132/01/30/064
B011/B126

quinoid structure into a benzoid structure. The reaction between dialkylphosphorous acids and α-naphthoquinone is similar. Table 1 shows the melting temperatures and the results of analyses of the compounds produced. They are crystalline substances, soluble in aqueous alkali solutions. They give the characteristic color reaction for phenylhydroxyl with ferric chloride, but no reaction for the carbonyl group. The hydrolysis of the substances obtained with HCl (1:1), and the saponification with alcoholic alkalis at 40-50° gives a yield of 80%. All compounds produced contain only one hydroxyl group. On the basis of the ultraviolet absorption spectra the authors have stated that esters of p-oxyphenyl-phosphorous acid are concerned. As can be seen from table 2, the absorption maximum of the products is shifted towards short waves, and agrees with the maximum of dimethyl-p-methoxyphenylphosphate. Thus, the results given above show that the said substances are really dialkyl-p-oxyphenylphosphates (see scheme). The following were also quoted: V. S. Abramov, A. N. Pudovik, Yu. P. Kitayev, and G. Zametayeva. There are 2 tables and 18 references, 10 of which are Soviet.

4

ASSOCIATION: Institut neftekhimicheskogo sinteza Akademii nauk SSSR (Institute of Petroleum-chemical Synthesis of the Academy of Sciences, USSR)

Card 2/3

84674

S/020/60/134/006/019/031
B016/B067

5.3700 2209, 1318, 1312 only

AUTHORS: Sergeyeva, Z. I., Tsitovich, D. D., and Voronkov, M. G.

TITLE: A New Reaction of Trialkyl Silanes With Acid Chlorides of Aliphatic Monocarboxylic Acids in the Presence of Aluminum Chloride

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 134, No. 6, pp. 1371-1373

TEXT: In the presence of $AlCl_3$ alkyl halides are easily reduced from trialkylsilanes to the corresponding hydrocarbons (Refs. 1,3) whereas acid chlorides of aromatic acids are reduced to aldehydes (Ref. 4). This reaction proceeds according to the general scheme:

$$R_3SiH + R'X \xrightarrow{AlCl_3} R_3SiX + R'H$$

where R' is a carbon- or acyl radical and X a halogen. The authors studied this reaction by applying it to the acid chlorides of the aliphatic monocarboxylic acids. They studied the reduction of the acid chlorides of

Card 1/3

84674

A New Reaction of Trialkyl Silanes With Acid Chlorides of Aliphatic Monocarboxylic Acids in the Presence of Aluminum Chloride

S/020/60/134/006/019/031
B016/B067

acetic, n-butyric, trimethyl acetic, and β-trimethyl silyl propionic acid by means of triethyl silane. In this connection it was found that in the absence of $AlCl_3$ practically no interaction of the reagents occurred. If, however, catalytic amounts of $AlCl_3$ (2-3 mol%) were introduced into the reaction mixture strong heating was observed. In contrast to what had been expected and to the data of Ref. 4 the corresponding aldehydes were not formed although the initial triethyl silane was converted into triethyl chlorosilane with a yield of 66-92%. Corresponding esters which were isolated in good yields proved to be the reaction products of the acid chlorides. These results make it possible to express the new reaction discovered by the authors by the following equation:

$$2R_3SiH + 2R'COCl \xrightarrow{AlCl_3} 2R_3SiCl + R'COOCH_2R',$$

where $R = C_2H_5$, $R' = CH_3$, $n-C_3H_7$, $(CH_3)_3C$, $(CH_3)_3SiCH_2CH_2$. The mechanism of this reaction could not be definitely determined. Apparently an intermediate reduction of the acid chloride to a corresponding aldehyde takes

Card 2/3

81674

A New Reaction of Trialkyl Silanes With Acid Chlorides of Aliphatic Monocarboxylic Acids in the Presence of Aluminum Chloride

S/020/60/134/006/019/031
B016/B067

place which reacts with the acid chloride excess according to the following scheme:

$$R_3SiH + R'COCl \longrightarrow R'CHO + R_3SiCl,$$

$$R'CHO + R'COCl \longrightarrow R'COOCHClR' \quad \text{(described in Ref. 9)}$$

$$R'COOCHClR' + R_3SiH \longrightarrow R'COOCH_2R' + R_3SiCl.$$

The possibility of a direct ester condensation of the aldehydes formed cannot be excluded. Table 1 gives the reaction products obtained. There are 1 table and 10 references: 2 Soviet, 1 US, 1 Danish, 2 Belgian, 3 French, and 1 German.

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ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR (Institute of Silicate Chemistry of the Academy of Sciences, USSR). Leningradskiy gosudarstvennyy universitet im. A. A. Zhdanova (Leningrad State University imeni A. A. Zhdanov)

PRESENTED: June 3, 1960, by A. V. Topchiyev, Academician

SUBMITTED: June 13, 1960

Card 3/3

VORONKOV, M.G., kand. khim. nauk, otv. red.; BORISOV, S.N., kand. khim. nauk, red.; TRAVIN, N.V., red. izd-va; KRUGLIKOVA, N.A., tekhn. red.

[Chemistry and practical utilization of silicon organic compounds; papers] Khimiia i prakticheskoe primenenie kremneorganicheskikh soedinenii; trudy. Leningrad, Izd-vo Akad. nauk SSSR. No.6.[Papers, discussion, resolutions] Doklady, diskussii, reshenie. 1961. 351 p. (MIRA 14:11)

1. Vsesoyuznaya konferentsiya po khimii i prakticheskomu primeneniyu kremneorganicheskikh soyedineniy, 2d, Leningrad, 1958. (Silicon organic compounds)

VORONKOV, M. G., DOC CHEM SCI, "HETEROLYTIC REACTIONS OF THE BREAKDOWN OF A SILOXEN BOND." ~~A PAPER~~ Report ON SCIENTIFIC WORKS SUBMITTED IN COMPETITION FOR THE ~~UNIVERSITY~~ academic DEGREE OF DOC~~TOR~~S OF CHEM~~ICAL~~ SCI~~ENCES~~. MOSCOW, 1961 (ACAD SCI USSR, INSTITUTE OF PETROCHEMICAL SYNTHESIS). KL-DV, 11-61, 210).

S/661/61/000/006/026/081
D205/D302

AUTHORS: Orlov, N. F., Dolgov, B. N. and Voronkov, M. G.

TITLE: Synthesis of triorganosyloxy-derivatives of boron, titanium, phosphorous and vanadium, from triorgano-silanols and hexaalkyl disiloxanes. Reaction of hexaalkyl disiloxanes with aluminum and titanium halides

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniy; trudy konferentsii, no. 6, Doklady, diskussii resheniye. II Vses. Konfer. po khimii i prakt. prim. kremneorg. soyed., Len., 1958. Leningrad, Izd-vo AN SSSR, 1961, 123-126

TEXT: The field of elemental-silico-organic compounds is of practical interest because of the possibility of synthesis of compounds which are similar to inorganic compounds in their thermal stability while being soluble in organic solvents. The syntheses proposed give better yields than those previously described in literature. As to the application of the synthesized compounds, the silico-

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Card 1/2

organic borates may serve as catalysts in the synthesis of aryl halogenosilanes and the synthesized titanates may serve as starting compounds for preparing thermally stable titanium silico-organic compounds. The synthesized compounds are interesting also in connection with the study of the Si-O-M bond. The use of triorganosiloxy-derivatives of Ti and Al chlorides as catalysts in preparing trialkyl chlorosilanes is thought to be possible. A. L. Klebanskiy (VNIISK, Leningrad), A. A. Zhdanov (INEOS AN SSSR, Moscow), N. S. Leznov (Moscow) and Ya. I. Mindlin (Moscow) took part in the discussion which followed.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR, Leningrad (Institute of Silicate Chemistry of the AS USSR, Leningrad)

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S/661/61/000/006/029/031
D205/D302

AUTHORS: Davydova, V. P., Voronkov, M. G. and Dolgov, B. N.

TITLE: Cyclic esters of dialkylsilane diols and orthosilicic acid

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniy; trudy konferentsii. no. 6: Doklady, diskussii, resheniye. II Vses. konfer. po khimii i prakt. prim. kremneorg. soyed., Len., 1958. Leningrad, Izd-vo AN SSSR, 1961, 134-135

TEXT: It is assumed that, depending on the temperature, the reactions between acyloxysilanes and alcohols proceed by different mechanisms. At lower temperatures:

$$\equiv SiOAc + ROH \rightarrow \equiv Si - OR + AcOH \text{ (silylation)}$$

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This is changed at higher temperatures to:

Card 1/2

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D205/D302

Cyclic esters of ...

$$\equiv SiOAc + ROH \rightarrow \equiv Si - OH + AcOR \text{ (acylation)}$$

The interaction product of dimethyl diacetoxysilane with glycerin, $(CH_3)_2$, $Si(OH)OCH_2 \cdot CHOHCH_2OCOCH_3$, polymerizes on standing and splits off acetic acid. The gelatinous polymer is thermally depolymerized on distillation, changing into a liquid. The latter is changed back to a gel on cooling. This polymer is soluble in water. All the cyclic ethers of dimethyl silane diol with glycols or glycerine are reversibly polymerizable. They are obtained in good yields and do not decompose on distillation. These substances possess a strong odor resembling that of menthol. R. Kh. Freydlina (INEOS AN SSSR, Moscow) took part in the discussion which followed.

ASSOCIATION: Institut khimii silikatov, AN SSSR, Leningrad (Institute of Silicate Chemistry of the AS USSR, Leningrad)

Card 2/2

S/661/61/000/006/030/081
D205/D302

AUTHOR: Voronkov, M. G.

TITLE: On heterolytic reactions of the splitting of siloxanes

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniy; trudy konferentsii. no. 6: Doklady, diskussii, resheniye. II Vses. konfer. po khimii i prakt. prim. kremneorg. soyed., Len., 1958. Leningrad, Izd-vo, AN SSSR, 1961, 136-153

TEXT: A general review of the subject, paying special attention to the theoretical side of the problem. Mechanisms of the splitting processes of organo-siloxanes by electrophilic and nucleophilic reagents are proposed. On the basis of these mechanisms new methods of synthesis of a series of silico-organic and elemental-silico-organic compounds were proposed and performed. There are 172 references: 27 Soviet-bloc and 145 non-Soviet-bloc. The 4 most recent references to the English-language publications read as follows: W. Gerrard and M. F. Lappert, Chem. Rev., 58, 1081, (1958),

Card 1/2

On heterolytic reactions ...

S/661/61/000/006/030/081
D205/D302

A. H. Cowley, F. Fairbrother and N. Scott, J. Chem. Soc., 717, (1959); Brit. Pat. 795,185 (1959); J. R. Thomas and L. de Vues, J. Phys. Chem., 63, 254, (1959).

ASSOCIATION: Institut khimii silikatov, AN SSSR, Leningrad (Institute of Silicate Chemistry of the AS USSR, Leningrad)

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Card 2/2

S/661/61/000/006/064/081
D243/D302

AUTHORS: Lazarev, A. N. and Voronkov, ~~M. G.~~

TITLE: Oscillations of the bonds of silico-oxygen tetrahedrons in the spectra of polyalkoxysiloxanes

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniy; trudy konferentsii, no. 6: Doklady, diskussii, resheniye. II Vses. konfer. po khimii i prakt. prim. kremneorg. soyed., Len. 1958. Leningrad, Izd-vo AN SSSR, 1961, 283-285

TEXT: This is a discussion of the authors' paper (this publication, no. 3, p. 52) in which Yu. P. Yegorov (IOKh AN SSSR, Moscow) and P. G. Maslov (Leningrad) took part. The authors stated that their results could not be compared with those of Stepanov and Prim, due to simplifications introduced by the latter authors. The theoretical interpretation of metasilicate spectra is thought to be far from complete. The symmetrical oscillation of the SiO_4 group was studied because its higher sensitivity permitted a more detailed investiga-

Card 1/2

Oscillations of the ...

S/661/61/000/006/064/081
D243/D302

tion. A full discussion of the spectra investigated is to be published. Iguchi's results were used for calculating the $(MeO_4)Si$ spectrum. The particular attention paid to the Si-O bond in the materials investigated is explained by the wish to utilize the observed phenomena in analyzing the spectra of certain silicates.

ASSOCIATION: Institut khimii silikatov Akademii nauk SSSR, Leningrad (Institute of Silicate Chemistry, Academy of Sciences, USSR, Leningrad)

Card 2/2

S/661/61/000/006/075/081
D287/D302

AUTHORS: Voronkov, M. G. and Kalugin, N. V.

TITLE: Imparting hydrophobic properties to cellulose materials by using organosilicon compounds

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniy; trudy konferentsii, no..6: Doklady, diskussii, resheniye. II Vses. konfer. po khimii i prakt. prim. kremneorg. soyed., Len. 1958. Leningrad, Izd-vo AN SSSR, 1961, 328-335

TEXT: This is a continuation of earlier investigations on treating textile fibers with organosilicon compounds (polyalkyl hydrosiloxanes, alkyl acyloxysilanes and polyalkyl siloxanols of some metals). The authors assume that the high stability of water-repellant organosilicon impregnating agents is due to the chemical interaction of the reactive functional groups in the utilized monomers and polymers of organosilicon compounds and the hydroxyl groups in cellu-

Card 1/3

S/661/61/000/006/075/081
D287/D302

Imparting hydrophobic properties ...

lose so that a C-O-Si bond is formed. This reaction can occur during thermal treatment of the fibers but also at room temperature (especially in the presence of a catalyst); in the latter case the reaction is much slower. This hypothesis was confirmed by experiments on model systems. A water-repellent, chemisorbed layer is formed on the cellulose fibers. The effect of the orientation of the hydrocarbon radicals of the organosilicon compounds, and of the hydrophilic hydroxyl groups of cellulose on the hydrophobic properties of this layer are discussed. Hydrophobing of materials, containing OH groups (including cellulose) by using polyalkyl hydrosiloxanes is stated to proceed effectively in the absence of oxygen or oxidising agents. Salts of Zn, Cd, Hg, Co and Ni (acetates or chlorides) are satisfactory catalysts. Organosilicon polymers which do not contain reactive functional groups and therefore cannot combine chemically with cellulose do not give stable water-repellant coatings on textile fibers. The reaction of organosilicon compounds with a reactive functional group X(X=H, OCOR, OR) with H_2O is discussed. Materials treated with trifunctional organosili-

Card 2/3

con monomers of the type $RSiX_3$ (but not polymers) acquire a high degree of stiffness, due to the formation of hard, non-elastic steric polymers. Bifunctional monomers do not induce this stiffness but also do not impart water-repellant properties. At present, mixtures of tri- and bifunctional monomers are used as hydrophobing agents. The authors have also found that organosilicon hydrophobing agents combine well with Cu-and Cr-salts, forming stable compounds which are retained for long periods on the textile materials. In the discussion the authors stated that ЕН-8 (YeN-8) was the hydrolysis product of ethyl dichlorosilane and dimethyl dichlorosilane. P. A. Simigin (TsNIKhBI, Moscow), G. D. Nessonova (Moskovskiy tekstil'nyy institut (Moscow Textile Institute)) and I. A. Zubkov (Moscow) discussed their observations on various hydrophobing agents. There are 2 figures, 1 table and 19 references: 13 Soviet-bloc and 6 non-Soviet-bloc. The references to the English-language publications read as follows: F. Fortess, Ind. Eng. Chem., v. 46, (1954), 2325; H. A. Schuyter, J. W. Weaver and J. D. Reid, J. Am. Chem. Soc., v. 70, (1948), 1919; R. W. Kerr and K. C. Hobbs, Ind. Eng. Chem., v. 45, (1953), 2542.

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S/661/61/000/006/076/081
D287/D302

AUTHORS: Afonchikov, N. A., Kolobova, G. V., Mikhaylov, P. N. and Voronkov, M. G.

TITLE: The use of organosilicon compounds in paper-sizing

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniy; trudy konferentsii, no. 6: Doklady, diskussii, resheniye. II Vses. konfer. po khimii i prakt. prim. kremneorg. soyed., Len. 1958, Leningrad, Izd-vo AN SSSR, 1961, 336

TEXT: The Leningradskaya bumazhnaya fabrika 'Gosnak' (Leningrad Paper Factory 'Goznak') carried out, in conjunction with the Institut khimii silikatov AN SSSR (Institute for Silicate Chemistry, AS USSR), experiments on the use of organosilicon compounds in the paper industry. The paper should possess hydrophobic properties which prevent the soaking in of ink. Organosilicon compounds do not show the same disadvantages as colophony (which is generally used for this purpose). The authors used the substance МН-1 (MN-1) (which

Card 1/2

S/661/61/000/006/076/081
D287/D302

The use of organosilicon ...

was most satisfactory) and tested the material ГКЖ-94 (GKZh-94). The latter was, however, unstable. Thermal treatment gave paper of suitable sizing properties, but it is difficult to carry out this type of processing in the machine itself. Paper treated with organosilicon compounds has improved print-receptivity. The compounds do not affect the other properties of the paper.

Card 2/2

S/062/61/000/001/016/016
B101/B220

AUTHORS: Voronkov, M. G. and Shemyatenkova, V. T.

TITLE: Gasometric determination of alkyl radicals in polyalkyl siloxanes and silicon-functional organosilicon compounds

PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, no. 1, 1961, 178-180

TEXT: The authors state that so far no method has been available for the quantitative determination of alkyl radicals bound to the silicon atom. Proceeding from the fact that the Si-C bond can be easily disrupted under the influence of strong nucleophilic reagents, such as alkali hydroxides, the optimum conditions for this reaction and for the formation of the stoichiometric volume of the hydrocarbon were investigated. Fig. 1 represents the apparatus used for the purpose. 1 is a gas burette in a thermostat, 2 is a leveling tube filled with acidified, saturated NaCl solution or Hg. By means of three-way cock 3 and absorber 4 which contains concentrated H_2SO_4, burette 1 is connected with steel vessel 7. The

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latter contains the substance to be tested (0.1-0.15 g), which is covered by 2-3 g of KOH powder and heated to 250-270°C in an electric furnace till no more gas is set free. After the cooling of 7, the gas is measured in burette 1. A table indicates the experimental data. The possibility of analyzing also alkyl halosilanes in this way is mentioned: methyl-phenyl dichlorosilane gave CH_3 = 7.67% and 8.54%; calculated value: 7.87%. If, however, the Si atom bound to Cl is bound at the same time to the oxygen atom or to groups being more electrophilic than the central silicon atom, this method cannot be used. The experiments were made with the assistance of A. A. Khvoshchevskaya, L. M. Kharchevnikova, and Z. I. Shabarova. There are 1 figure, 1 table, and 6 references: 2 Soviet-bloc and 4 non-Soviet-bloc.

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