SOV/79-29-5-12/75

Tetraacyl-oxy-silanes in Organic Synthesis.
21. Preparation of Acyl-oxy-trichloro Silanes From Acids of Aromatic Nature

solubility of the acids in silicon tetrachloride plays an important part in the synthesis. Under the experimental conditions silicon tetrachloride did not react with ether or acetone. The use of the latter to attain a better solubility of the acids accelerates, however, the formation of acyl-oxy-trichloro silanes. The formation mechanism of acyl-oxy-trichloro silanes was investigated on the example of the interaction of silicon tetrachloride with o-chloro benzoic- and benzoic acid. By the influence of excess silicon tetrachloride upon the organic acid acyl-oxy-trichloro silane is formed without any intermediate products. When studying the properties of the acyl-oxy-trichloro silanes the cleavage reaction under various conditions was investigated with special thoroughness. It was found on benzoyl-trichloro silane that its cleavage is especially considerable during distillation under atmospheric pressure, in which connection benzoyl chloride, silicon dioxide and silicon tetrachloride are formed (Ref 1). On the basis of experimental data the following scheme of the thermal decomposition of benzoyl trichloro silane can be established: 1) Benzoyl-oxy-trichloro silane is disproportionated in vacuum and on standing to give silicide and silicon tetrachloride. 2) The

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Tetraacyl-oxy-silanes in Organic Synthesis. SOV/79-29-5-12/75
21. Preparation of Acyl-Oxy-trichloro Silanes From Acids of Aromatic Nature

benzoic acid silicide is further cleaved to form benzoic anhydride and silicon dioxide. 3) The formed benzoic anhydride yields under the influence of silicon tetrachloride on one hand and of benzoyl-trichloro silane on the other hand benzoyl chloride and silicon dioxide. Table 1 - the rate of the disproportionation of acyl-trichloro silanes; table 2 - acyl-oxy-trichloro silanes; table 3 - disproportionation products of the acyl-oxy-trichloro silanes; table 4 - tetra-acyl-oxy-silanes. There are 4 tables and 8 references, 3 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: May 19, 1958

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SOV/79-29-6-42/72

5(3)

AUTHORS: Korobitsyna, I. K., Severina, T. A., Yur'yev, Yu. K.

TITLE: Synthesis of the 4-Oxymethylene-2,2,5,5-tetraalkyl Furanidones-3 (Sintez 4-oksimetilen-2,2,5,5-tetraalkilfuranidonov-3)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 6, pp 1960 - 1964 (USSR)

ABSTRACT: In continuation of a previous paper (Ref 1) the authors describe the synthesis of the β-ketoaldehyde of the 4-formyl-2,2,5,5-tetraalkyl furanidones-3. They found that the 2,2,5,5-tetraalkyl furanidones-3 enter the condensation with ethyl formate according to the Claisen reaction. In carrying out the reaction in absolute ether with finely ground sodium 4-oxymethylene-2,2,5,5-tetraalkyl furanidones-3 are formed (yield 56-66%).

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Synthesis of the 4-Oxymethylene-2,2,5,5-tetraalkyl Furanidones-3

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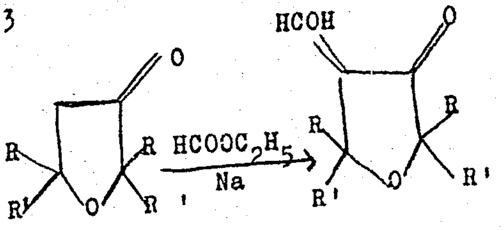
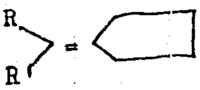


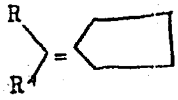
(I) $R=R'=CH_3$

(II) $R=CH_3, R'=C_2H_5$

(III)



(IV)



These furanidones are crystalline products very unstable in air. They must be stored in dark glasses if possible in unpurified state. They take a cherry-red color with $FeCl_3$ and form green copper salts with copper acetate. The absorption spectra of these copper salts in methanol show in the ultraviolet range the maxima characteristic of the copper salts of the β-dicarbonyl compounds. The percent content of the enol form was determined according to K. Meyer (Ref 2) (Table). The data in the table show that the β-ketoaldehydes of the 2,2,5,5-tetraalkyl furanidine series as well as of the alicyclic series (Refs 3,4) are a mixture of the formyl and oxymethylene form which is in equi-

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Synthesis of the 4-Oxymethylene-2,2,5,5-tetraalkyl Furanidones-3 SOV/79-29-6-42/72

librium. In this case the latter considerably predominates. The data on the table also show that with the increase of the radicals in the positions 2 and 5 of the furanidine cycle the enolization of the formyl group increases in position 4. In the action of the benzoyl chloride on the pyridine solutions of the compounds (I), (III), (IV) the corresponding O-benzoates (VII),(V) and (IX) were obtained. In the case of the action of the sodium compounds of the same oxymethylene ketones the compounds (VI), (VIII) and (X) were obtained (Scheme 2). There are 1 table and 4 references, 1 of which is Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: May 9, 1958

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SOV/79-29-6-43/72

5(3)

AUTHORS: Yur'yev, Yu. K., Zaytseva, Ye. L.

TITLE: The Chemistry of Selenophene (Khimiya selenofena). XVII. Condensation of the 5-Nitroselenophene-2-aldehyde With Compounds Containing Active Methylene Groups (XVII. Kondensatsiya 5-nitroselenofen-2-al'degida s soyedineniyami, soderzhashchimi aktivnyye metilenovyye gruppy)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 6, pp 1965 - 1969 (USSR)

ABSTRACT: In continuation of the previous paper (Ref 1) 5-nitroselenophene-2-aldehyde was condensed with compounds containing active methylene groups which led to a series of 5-nitroselenenal-2-derivatives. Thus, in heating 5-nitroselenophene-2-aldehyde with acetic anhydride and anhydrous sodium acetate β-(5-nitroselenienyl-2)-acrylic acid (42%) was obtained. The same acid was synthesized by condensation of the same aldehyde with malonic acid in the presence of pyridine with subsequent decarboxylation of the formed α-carboxy-β-(5-nitroselenienyl-2)-acrylic acid (Scheme 1). Thus, the condensation of 5-nitroselenophene-2-aldehyde with compounds containing active methylene groups

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The Chemistry of Selenophene XVII. Condensation of the 5-Nitroselenophene-2-aldehyde With Compounds Containing Active Methylene Groups SOV/79-29-6-43/72

leads to the following 5-nitroselenenal-2-derivatives: β-(5-nitroselenienyl-2)-acrylic acid (in the condensation with acetic anhydride or malonic acid-scheme 1); α-alkyl-β-(5-nitroselenienyl-2)-acroleins (in the condensation with acetic, propionic and butyric acid aldehyde-scheme 2); ω-(5-nitroselenienyl-2)-nitroethylene (in the condensation with nitromethane-scheme 3); 5-(5-nitroselenenal-2)-thiazolidone-4-thione-2 (in the condensation with rhodanine); 2 phenyl-4-(5-nitroselenenal-2)-oxazolone-5 (in the condensation with hippuric acid-scheme 4). There are 6 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: May 9, 1958

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SOV/79-29-6-44/72

5(3)

AUTHORS: Yur'yev, Yu. K., Sadovaya, N. K., Gal'bershtam, M. A.

TITLE: Chemistry of Selenophene (Khimiya selenofena). XVIII. Synthesis of the Isomeric Trimethyl Selenophene and of Tetramethyl Selenophene (XVIII. Sintez izomernykh trimetilselenofenov i tetrametilselenofena)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 6, pp 1970 - 1973 (USSR)

ABSTRACT: The authors continued their investigation on the methyl homologues of selenophene (Ref 1). They made use of the possibility of a subsequent formylation of the selenophene and the reduction of the aldehyde group into the methyl group for the synthesis of the trimethyl selenophene and of tetramethyl selenophene, hitherto unknown. The synthesis of 2,3,5-trimethyl selenophene was based on 2,3-dimethyl selenophene which, by the action of dimethyl formamide, led to the 2,3-dimethyl selenophene-5-aldehyde. This aldehyde yielded the 2,3-dimethyl selenophene-5-carboxylic acid in the case of oxidation with silver oxide. In the reduction according to the general method by Kizhner (Ref 3) the 2,3,5-trimethyl selenophene was obtained.

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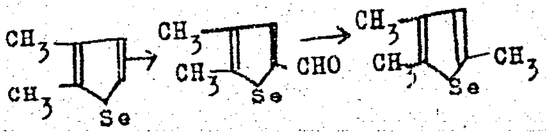
Chemistry of Selenophene. XVIII. Synthesis of the Isomeric Trimethyl Selenophene and of Tetramethyl Selenophene



3,4-dimethyl selenophene served as initial product for the synthesis of 2,3,4-trimethyl selenophene. Its formylation yielded 3,4-dimethyl selenophene-2-aldehyde the oxidation of which led to the 3,4-dimethyl-selenophene-2-carboxylic acid. In the reduction of 3,4-dimethyl selenophene-2-aldehyde 2,3,4-trimethyl selenophene was obtained, which served as initial product for the synthesis of tetramethyl selenophene. In the formylation of 2,3,4-trimethyl selenophene 2,3,4-trimethyl selenophene-5-aldehyde was obtained, which in the case of oxidation formed 2,3,4-trimethyl selenophene-5-carboxylic acid, in the case of reduction 2,3,4,5-tetramethyl selenophene (Scheme 2). From the two isomeric trimethyl selenophene only 2,3,4-dimethyl selenophene yielded the 5-chloro mercuri-2,3,4-trimethyl selenophene by means of mercurization; the 2,3,5-trimethyl selenophene did not enter this reaction. There are 7 Soviet references.

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SOV/79-29-7-20/83

5 (3)

AUTHORS: Korobitsyna, I. K., Zhukova, I. G., Yur'yev, Yu. K.

TITLE: Reactions of the 4-Bromo- and 4-Oxy-2,2,5,5-tetraalkyl Furanidones-3 (Reaktsii 4-brom- i 4-oksi-2,2,5,5-tetraalkilfuranidonov-3)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 7, pp 2190-2196 (USSR)

ABSTRACT: The bromine in the 4-bromo-2,2,5,5-tetramethyl furanidone-3 is not substituted by the oxy group (in the hydrolysis with soda solution), by iodine (in the action of KJ), by the thiocyanogen group (in heating with potassium thiocyanate), by the amino group (with ammonia); only in the reaction with sodium cyanide the corresponding nitrile is formed in good yield (Refs 1, 2). The authors used such halides for the synthesis of the condensated systems which contain furanidine- and thiazole rings. In the reaction with crystalline sodium sulphide at 135-140° only the 4-bromo-2,2,5,5-tetraalkyl furanidones-3 easily separated HBr, with the condensation taking place under the formation of the γ-diketone with two furanidine rings (Scheme 1). In the hydrogenation of (I) in the presence of nickel the γ-ketone (IV) resulted [the di-(2,2,5,5-tetramethyl

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Reactions of the 4-Bromo- and 4-Oxy-2,2,5,5-tetraalkyl Furanidones-3 SOV/79-29-7-20/83

furanidone-3-yl-4)], which with aniline led to dianil (V) (Scheme 2). Already earlier (Ref 3) the authors used the furanidones (VI) for the synthesis of nitrogenous heterocyclic systems with the furanidine ring. By this method the compounds (VII) and (VIII) were obtained from 4-oxy-2,2,5,5-tetramethyl- and 4-oxy-2,2,5,5-bispentamethylene furanidone-3 (Scheme 3) which are weak acids. The furanidones (VI) react with ammonium thiocyanate in melting (150°) to form furanidine thiazoles (IX), (X), (XI) (Scheme 4). A scheme of the formation of these compounds is suggested. There are 8 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 2, 1958

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5 (3) SOV/79-29-7-21/83

AUTHORS: Korobitsyna, I. K., Zhukova, I. G., Yur'yev, Yu. K.

TITLE: 4-Acetyl-2,2,5,5-tetraalkyl Furanidones-3 (4-Atsetil-2,2,5,5-tetra-alkilfuranidony-3)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 7, pp 2196-2201 (USSR)

ABSTRACT: Earlier (Ref 1) the authors showed that 4-acetyl-2,2,5,5-tetramethyl furanidone-3 forms in the thermal isomerization of the enol acetate of 2,2,5,5-tetramethyl furanidone-3. In the present paper they tried to apply this method also to the synthesis of the higher homologs of 4-acetyl-2,2,5,5-tetraalkyl furanidones-3. It was found that in passing the enol acetate of 2,5-dimethyl-2,5-diethyl furanidone-3 through a quartz tube filled with glass wool and which had been heated to 500° this enol acetate isomerizes into 4-acetyl-2,5-dimethyl-2,5-diethyl furanidone-3 (15,6% yield)(Scheme 1). Further investigations showed that with increasing radicals in the positions 2 and 5 the yields of the products of thermal isomerization in the enol acetates of 2,2,5,5-tetraalkyl furanidones-3 (of the corresponding 4-acetyl-2,2,5,5-tetraalkyl furanidones-3)

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4-Acetyl-2,2,5,5-tetraalkyl Furanidones-3 SOV/79-29-7-21/83

are abruptly reduced. This method, however, is still the only one possible for the synthesis of 4-acetyl-2,2,5,5-tetramethyl furanidone-3 and of 4-acetyl-2,5-dimethyl-2,5-diethyl furanidone-3 since other experiments failed. The two oxy-ketones (III) and (IV) may occur as tautomeric forms of 4-acetyl-2,2,5,5-tetramethyl furanidone-3(II). 4-acetyl-2,2,5,5-tetramethyl furanidone-3 is enolized in a high degree in the direction of the exocyclic carbonyl group; it forms C- and O- derivatives according to the conditions of acylation. The β-diketones of the 2,2,5,5 tetraalkyl furanidine series require much more rigid conditions in the reactions leading to the condensated heterocyclic systems than the aliphatic or aromatic β-diketones. There are 8 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 2, 1958

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5(3)

SOV/79-29-7-42/83

AUTHORS: Yur'yev, Yu. K., Novitskiy, K. Yu., Demina, M. N.

TITLE: On the Structure of the Products of the Reaction of 2-Amino-4-methylthiazole With α-Oxides (O stroyenii produkta vzaimodeystviya 2-amino-4-metiltiazola s α-okisyami)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 7, pp 2299-2302 (USSR)

ABSTRACT: In connection with a previous paper by Yur'yev and coworkers (Ref 1) the authors synthesized the hitherto unknown 2-(β-oxyalkyl)-aminothiazoles by two different methods, in order to ascertain simultaneously the structure of the oxyalkyl derivatives of 2-amino-4-methylthiazole obtained by reaction of the latter with α-oxides. Apparently thiazole derivatives with alkylated "ring-nitrogen"-atoms are not formed in the reaction of 2-chloro-4-methylthiazole with alkanolamines in the manner described here (Ref 3). By reaction of 2-chloro-4-methylthiazole with ethanolamine and 2-propanolamine 2-(β-oxyethyl) amino-4-methylthiazole and 2-(β-oxypropyl) amino-4-methylthiazole

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On the Structure of the Products of the Reaction of 2-Amino-4-methylthiazole with α-Oxides SOV/79-29-7-42/83

respectively were obtained. A comparison of the constants of these compounds and their derivatives with the constants of the compounds synthesized by the authors by reaction of 2-amino-4-methylthiazole with the corresponding α-oxides (Ref 1) proved to be identical. This was also confirmed by the identity of the infrared absorption spectra of the 2-(β-oxyethyl) aminothiazoles obtained by different methods and the ultraviolet absorption spectra of their hydrochlorides (reaction scheme) Thus α-oxides react differently with 2-amino-4-methylthiazole than with 2-aminopyridine and yield 2-(β-oxyalkyl) aminothiazoles, thereby retaining the thiazole structure. There are 1 figure, 1 table, and 9 references, 4 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: June 18, 1958

Card 2/2

SOV/79-29-8-30/81

5(3)

AUTHORS: Yur'yev, Yu. K., Belyakova, Z. V., Kostetskiy, P. V., Prokof'yev A. I.

TITLE: Tetraacyloxy-silanes in Organic Synthesis. XXIII. Acylation of Amines, Arylhydrazines and Acid Hydrazides With Tetraacyloxy-silanes

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 8, pp 2594 - 2597 (USSR)

ABSTRACT: Previously (Ref 1) the authors described the acylation of diethyl-amine with tetraacyloxy-silanes as a convenient synthesis of the N-N-diethylamides of the saturated monobasic organic acids (yields 60-90%)

$$(RCOO)_4Si + 4\ NHR'R" \longrightarrow 4\ RCONR'R" + Si(OH)_4.$$

It suggested itself to synthesize also other N,N-dialkyl- and N-alkylamides of the acids in the same way, and to use this method for the synthesis of the N,N-diethylamides of the aromatic acids, especially benzoic acid, o- and n-toluic acids (Refs 2,3). The acylation of dibutylamine was carried out with the silicic anhydrides of acetic, propionic, butyric and caproic acid,

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Tetraacyloxy-silanes in Organic Synthesis. XXIII. Acylation of Amines, Arylhydrazines and Acid Hydrazides With Tetraacyloxy-silanes

in which connection the N,N-dibutyl-acylamides of these acids were obtained in yields of 65-81%. Acylation of diethyl amine with the silicic anhydrides of benzoic acid, o- and n-toluic acids yielded the N,N-diethyl-benzamide (63%), N,N-diethyl-o-toluamide (24%), and N,N-diethyl-p-toluamide (37%). The decrease in the acylation capability of the tetraacyloxy-silane with increasing acidity was already previously observed by the authors (Ref 4). According to C. Friedel and A. Ladenburg (Ref 5), acetamide and N-ethyl acetamide were obtained in yields of only 12% and, accordingly, 5-6% on letting through ammonia and ethyl amine into the benzene solution of the silicon-acetic anhydride. The acylation of ethyl amine on heating with tetraacyloxy-silanes in the autoclave at 100° gave the N-ethyl amides of the acetic, propionic, butyric, valerianic, isovalerianic and caproic acid in yields of 33-82%. The acylation of phenyl hydrazine with the silicic anhydrides of acetic and propionic acid yielded the phenyl hydrazides of these acids (67 and 61%):

$4\ C_6H_5NHNH_2 + (RCOO)_4Si \longrightarrow 4\ C_6H_5NHNHCOR + Si(OH)_4$. The re-

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Tetraacyloxy-silanes in Organic Synthesis. XXIII. SOV/79-29-8-30/81
Acylation of Amines, Arylhydrazines and Acid Hydrazides With Tetraacyloxy-silanes

sultant substituted amides and hydrazides of the acids obtained by the above-mentioned acylation, their yields and constants, are presented in the table. There are 1 table and 29 references, 7 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 2, 1958

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SOV/79-29-8-31/81

5(3)

AUTHORS: Yur'yev, Yu. K., Mezentsova, N. N., Kashutina, E. A.

TITLE: Chemistry of Selenophene. XIX. 2-Aceto-selenophene in the Synthesis of α- and β-Keto-aldehydes in the Selenophene Series

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 8, pp 2597 - 2601 (USSR)

ABSTRACT: In addition to their previous papers (Refs 1-4), the authors used, in the present paper, the α-acyl-selenophenes for the synthesis of the α- and β-dicarbonyl compounds in the selenophene series. In this way, they obtained by oxidation of 2-aceto-selenophene with selenium dioxide the selenienyl-2-glyoxal, a keto-aldehyde, the bright-yellow color of which is due to a conjugation of the double bonds of two carbonyl groups and of the selenophene nucleus. The ultraviolet absorption spectrum of the selenienyl-2-glyoxal (Fig 1) has two maxima at λ 275 and 310 mμ. The compound is easily condensed with o-phenylene diamine, and forms quantitatively the 2-(selenienyl-2')-quinoxaline (Scheme 1). By the action

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Chemistry of Selenophene. XIX. 2-Aceto-selenophene in the Synthesis of α- and β-Keto-aldehydes in the Selenophene Series SOV/79-29-8-31/81

of alkali lyes on its monosemicarbazone and monothiosemicarbazone, water is split off and, accordingly, the 3-oxy- and the 2-mercapto-5-(selenienyl-2')-triazine-1,2,4 (Scheme 2). On condensation of 2-aceto-selenophene with the ethyl ester of formic acid under the influence of sodium, the sodium alcoholate of oxymethylene-(selenienyl-2)-ketone is obtained which is of dark-violet color in the thiophene series. Its absorption spectrum is characterized in figure 2 by curve I, the one of its intramolecular complex compound with Cu^{++} by curve II. There are 2 figures and 4 Soviet references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 2, 1958

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5(3)

SOV/79-29-9-30/76

AUTHORS: Yur'yev, Yu. K., Novitskiy, K. Yu., Bolesov, I. G.

TITLE: Investigation in the Series of Furan. I. Synthesis of the N-(β-Oxyalkyl)-furfuryl Amine

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 2951-2954 (USSR)

ABSTRACT: The present paper deals with the synthesis of the mono-N-(β-oxyalkyl)-furfuryl amines from 2-furfuryl amine and the simplest α-oxides (oxides of ethylene and propylene) according to a method already earlier developed by the authors (Refs 6,7) and taking account of the papers quoted in references 1-5, among them the paper of A. A. Ponomarev et al. (Ref 5). According to the previous method, ethylene oxide was allowed to flow into the amine. The corresponding N-(β-oxyalkyl)-furfuryl amines resulted in an 86%-yield. The yields were 83,5-92,5% at higher molar percentages of the α-oxides in the reaction with furfuryl amine. The properties of di-N-(β-oxyethyl)-furfuryl amine as synthesized by the authors were completely different from those of the preparation described by German authors (Ref 5): it is a colorless oil; its

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Investigation in the Series of Furan.
I. Synthesis of the N-(β-Oxyalkyl)-furfuryl Amine

picrate melts at 127-128°; its refractive index is higher. Its infrared absorption spectrum confirms its structure: instead of the lacking frequencies which are characteristic of the N-H bond there is a broad band characterizing the group O-H. No crystalline hydrochloride was obtained in the passage of hydrogen chloride through the chloroform solution of the preparation. In the reaction with thionyl chloride the hydrochloride of di-N-(β-ethyl chloride)-furfuryl amine resulted which yielded N-(2-furfuryl)-thiomorpholine under the action of sodium sulphide (2 Schemes). The given data which confirm the structure of di-N-(β-oxyethyl)-furfuryl amine show that G. Drefahl and K. König (Ref 5) mistook this compound for another one. With α-oxides di-(2-furfuryl amine forms N-(β-oxyalkyl)-difurfuryl amines in high yields (Scheme 3). There are 9 references, 4 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 14, 1958

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5(3) SOV/79-29-9-31/76

AUTHORS: Yur'yev, Yu. K., Zefirov, N. S.

TITLE: Investigation in the Series of Furan. II. Reaction of Compounds of the Furan Series With the Diethyl Ester of Azodicarboxylic Acid

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 2954-2960 (USSR)

ABSTRACT: On the basis of a number of papers quoted in references 1-30 the authors investigated the reactions of the diethyl ester of azodicarboxylic acid with furan, 2-methyl furan (silvane), and furyl alcohol. After the experiments were finished, P. Baranger and J. Levisalles (Ref 8) published a paper describing the failed experiment in which the attempt had been made to cause furan, 2-methyl furan, and 2,5-dimethyl furan to react with the above ester. The mentioned research workers did not succeed in isolating the formed adducts and in carrying out an alkali and acid hydrolysis of the reaction products. In the experiment described the reaction of furan and 2-methyl furan with the above ester was found to proceed like a diene synthesis in which compound (I) and, accordingly, (II) result qualitatively. A large quantity of ether

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SOV/79-29-9-31/76

Investigation in the Series of Furan.
II. Reaction of Compounds of the Furan Series With the Diethyl Ester of Azodicarboxylic Acid

has to be used for these reactions as without a solvent they would proceed vigorously and cause resinification. Adducts (I) and (II) are non-crystalline, solid, glassy products, softening in powder form at 50-60°, in contrast to the adduct of the same ester with the diacetate of furfurole (Ref 9). The adducts (I) and (II) add easily to bromine, yielding glassy dibromides (III). The reaction with phenyl azide (Refs 31-32) leads to a crystalline triazoline derivative (IV), but only in the adduct (I). Hydrogen is added to the adducts (I) and (II) which yield dark, glassy products (V). In vacuum distillation a pyrolysis takes place (compound (VI)). When heated with phosphorus pentoxide (I) and (II) resinify, developing CO$_2$, ethylene and forming a small amount of diethyl ester of hydrazo dicarboxylic acid, which in turn decomposes under the formation of ethylene, CO$_2$, and nitrogen. The chemical properties of the adducts (I) and (II) and especially the formation of the crystalline addition product of phenyl azide to adduct (I) indicate that the reaction of

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Investigation in the Series of Furan.
II. Reaction of Compounds of the Furan Series With the Diethyl Ester of Azodicarboxylic Acid

furan and silvane with azodicarboxylic ester proceeds according to the normal diene synthesis. There are 1 figure and 35 references, 3 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 17, 1958

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5(3)

SOV/79-29-9-32/76

AUTHORS: Yur'yev, Yu. K., Belyakova, Z. V.

TITLE: Tetraacyloxysilanes in Organic Synthesis. XXIV. Reaction of Tetraacyloxysilanes With Organomagnesium- and Organocadmium Compounds

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 2960-2964 (USSR)

ABSTRACT: Two kinds of reaction are characteristic of acyloxysilanes: (1) Reactions taking place under the decomposition of the C-O-bond; (2) reactions taking place under the decomposition of the Si-O-bond. All acylation reactions with tetraacyloxysilanes belong to the first kind: acylation of the benzene cycle, thiophene, selenophene, pyrrole (Refs 1-4), of malonic ester, acetoacetic ester (Ref 5), and amines (Refs 6,7) (Scheme 1). In continuation of the collected investigation data of the papers quoted in references 1-11 the authors of the present paper investigated the reaction of tetraacyloxysilanes with organomagnesium- and organocadmium compounds; theoretically, two directions of reaction were possible due to the decomposition of the C-O- or Si-O-bonds, i.e. according to schemes (IV) and (V). Taking account of the papers

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SOV/79-29-9-32/76

Tetraacyloxysilanes in Organic Synthesis. XXIV. Reaction of Tetraacyloxysilanes With Organomagnesium- and Organocadmium Compounds

quoted in references 12-17, the reaction of silico-acetic anhydride with alkyl magnesium halides was investigated; in all cases tertiary alcohols were obtained in yields of 58-68,5%, tetraalkyl silanes (51-72%), and fractions with a high boiling point (apparently siloxanes). All these data as well as the absence of silicic acid show that the reaction of tetraacyloxysilanes with organomagnesium compounds takes place under the decomposition of the Si-O-bond according to scheme (V). The stability of this bond decreases with increasing acidity of the organic acid in the silico-acetic anhydride which results in a higher reactivity of tetraacyloxysilane towards the organomagnesium compound. Ketones (20-46%) and silicic acid resulted in the reaction of silico-acetic anhydride apparently under the decomposition of the C-O-bond according to scheme (IV). There are 28 references, 13 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 4, 1958

Card 2/2

SOV/80-32-2-25/56

AUTHORS: Shcherbakov, A.A., Yur'yev, Yu.K.

TITLE: The Effect of Preliminary Processing of Plant Raw Material by Organic Solvents on the Formation Dynamics and Yield of Furfurole (Vliyaniye predvaritel'noy obrabotki rastitel'nogo syr'ya organicheskimi rastvoritelyami na dinamiku obrazovaniya i vykhod furfurola)

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol XXXII, Nr 2, pp 374-382 (USSR)

ABSTRACT: The preliminary processing of pentosan-containing raw materials by various solvents increases the output of furfurole by preventing resinification during hydrolysis. In the experiments hydrolysis was carried out by a 12-% solution of hydrochloric acid. Table 1 shows that the pentosans in corn cobs, barley husks, oats chaff, etc are most easily hydrolyzed. The preliminary treatment of the raw materials by alcohol, ether, chloroform, benzene, gasoline, CCl_4, etc has a considerable effect on the furfurole output as well as on the dynamics of its formation. If the raw material is preliminarily treated by vaseline oil, diesel fuel, etc and then hydrolyzed by a

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SOV/80-32-2-25/56

The Effect of Preliminary Processing of Plant Raw Material by Organic Solvents on the Formation Dynamics and Yield of Furfurole

10-% solution of sulfuric acid, the output of furfurole increases by 43.4% in comparison with the yield without the solvents.
There are 2 tables and 11 references, 8 of which are Soviet, 1 American, 1 English, and 1 German.

SUBMITTED: July 1, 1957

Card 2/2

SOV/20-126-4-32/62

5(3)

AUTHORS: Yur'yev, Yu. K., Novitskiy, K. Yu., Zhingareva, V. N.

TITLE: Investigations Into the Furan Series (Issledovaniye ryadu furana). The Synthesis of Symmetric 2,5-bis-(dialkylamino-methyl)-furan (Sintez simmetrichnykh 2,5-bis-(dialkilamino-metil)-furanov)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 126, Nr 4, pp 806-808 (USSR)

ABSTRACT: The furans mentioned in the subtitle are hardly described in publications. 2,5-bis-(dimethyl-aminomethyl) furan was obtained with a very small yield (Ref 1). Diamines of such structure, as well as their dihaloid alkylates are of interest as potential, ganglion-blocking, and curare-like compounds, with regard to their physiological activity. The authors used 2,5-bis-chloromethyl furan for synthesizing symmetric diamines of the mentioned structure. Its reciprocal action with secondary amines showed satisfactory results in the production of the corresponding diamines (see scheme). The reaction develops easily with the reciprocal action of etheric solutions of bis-chloromethyl furan with secondary amine in the presence of caustic alkali. From among the secondary amines dimethyl-

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SOV/20-126-4-32/62

Investigations Into the Furan Series. The Synthesis of Symmetric 2,5-bis-(dialkylaminomethyl)-furan

amine and diethylamine, piperidine, and morpholine were added to the reaction. Thus 2,5-bis-(dimethyl-aminomethyl)-2,5-bis-(diethylaminomethyl), 2,5-bis-(piperidine-methyl), and 2,5-bis-(N-morpholine-methyl)-furan were produced. The constants corresponded to those produced by means of another method (Ref 1). The reciprocal action here described of 2,5-bis-chloromethylfuran with secondary amines, up to now has been the only comfortable way producing the corresponding symmetric amines of the furan series. There are 3 references, 1 of which is Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

PRESENTED: March 5, 1959, by A. N. Nesmeyanov, Academician

SUBMITTED: February 28, 1959

Card 2/2

SOV/20-128-1-32/58

5(2)

AUTHORS: Yur'yev, Yu. K., Makarov, N. V.

TITLE: Transformation of Furanidine and Tetrahydropyrane Into the Respective Silicon-containing Heterocycles. Transformation of the Latter Into Sulfur-containing Heterocycles

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 128, Nr 1, pp 121-123 (USSR)

ABSTRACT: In the present paper the catalytic method of interchangeable transformations of heterocycles discovered by one of the authors (Ref 2) was applied. The furanidine was caused to pass over aluminum oxide in the monosilane flow at 375°, thus obtaining simultaneously tetramethylene silane (4%) and ditetramethylene silane (1.5%). The latter is the transformation product of furanidine under the action of the tetramethylene silane formed. Subsequently, a mixture of furanidine and tetramethylene silane was passed over aluminum oxide, and ditetramethylene silane (4.5%) was formed. A similar transformation of furanidine under the action of ethyl silane proved to be impossible since the latter completely decomposes at

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SOV/20-128-1-32/58

Transformation of Furanidine and Tetrahydropyrane Into the Respective Silicon-containing Heterocycles. Transformation of the Latter Into Sulfur-containing Heterocycles

the contact with aluminum oxide at 350°. Tetramethylene silane was also obtained by a common catalytic dehydrogenation of butanediol-1.4 and monosilane under the same conditions. In the case of action of hydrogen sulfide on tetramethylene silane or tetramethylene dichloro silane the cyclically bound silicon atom is replaced by sulfur, thus forming thiophane (14% and 4% respectively). Tetrahydropyrane transforms to pentamethylene silane (9.5%) under the action of monosilane at the contact with aluminum oxide at 375°. Under the same conditions pentamethylene silane is transformed to tetrahydrothiopyrane under the action of hydrogen sulfide. There are 7 references, 5 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

Card 2/3

67264

5.4600
5.3100
~~5(3)~~

SOV/20-129-4-28/68

AUTHORS: Stradin', Ya., Giller, S., Academician AS LatvSSR, Yur'yev, Yu.

TITLE: Polarographic Reduction of 2-Nitrofuran Derivatives and 2-Nitroselenophene Derivatives

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 129, Nr 4, pp 816 - 819 (USSR)

ABSTRACT: The authors ascertained the influence exercised by the substituents in the 5th position of the furan- and selenophene cycle on the polarographic reduction process of the nitro groups in the second position. Thus, they completed the data of publications by new examples. The derivatives mentioned in the title may now be compared to the nitro derivatives of the aromatic series. Table 1 gives the derivatives I-XXV investigated in the present paper under vigorous conditions. It was found that the mechanism of polarographic reduction of the mentioned derivatives is the same as that of nitrobenzene- (Ref 7) and of 2-nitrothiophene (Ref 11) derivatives. Also the semiwave potentials $E_{1/2}$ of the nitro derivatives of the mentioned series are closely related. The comparison of these series leads to the conclusion that the nitro group of 2-nitrofuran derivatives is the most easily to be

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Polarographic Reduction of 2-Nitrofuran Derivatives and 2-Nitroselenophene Derivatives SOV/20-129-4-28/68

reduced. This is more difficult in the case of 2-nitrothiophene- and 2-nitroselenophene (which requires by 20-30 mv more) and still more difficult for nitrobenzene derivatives (by 40 mv more). From the investigation of this series of derivatives the influence exercised by the substituents on the polarographic reduction of the nitro group may be quantitatively estimated on the basis of the $E_{1/2}$ displacement of the substituted compound compared to the non-substituted one. In the series of nitrobenzene and nitrothiophene this displacement may be expressed by the Hammet equation. It may be concluded from the data given by the authors that this holds also for the derivatives mentioned in the title if the same values of σ are assumed for the substituents in the heterocycles as apply for the aromatic series, and if the numerical values of $\Delta E_{1/2}$ and ρ are compared for an equal pH value in a weakly acid medium. The behavior of the 2,5-substituted derivatives of the 5-membered heterocycles corresponds to the behavior of the p-substitutes of the aromatic series. The behavior of the former however strongly differs from that of the m-substitutes. This agrees on the whole with the rules of

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67264

Polarographic Reduction of 2-Nitrofuran Derivatives and SOV/20-129-4-28/68
2-Nitroselenophene Derivatives

orientation found in the study of the reactivity of the substituted furans. However, further polarographic measurements are necessary in this case. The influence exercised by the substituents over an additional group -CH=CH- in the side chain is in general not high. The reduction of 5-nitro furfurol proceeds in a characteristic manner (Scheme). There are 1 table and 16 references, 9 of which are Soviet.

4

ASSOCIATION: Institut organicheskogo sinteza Akademii nauk LatvSSR (Institute of Organic Synthesis of the Academy of Sciences of the Latviyskaya SSR). Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University imeni M. V. Lomonosov)

SUBMITTED: July 21, 1959

Card 3/3

YUR'YEV, Yu.K.; ROZANTSEV, E.G.

Synthesis of 2, 5-dimethyl-3-alkyl- and 2, 5-dimethyl-3-arylthiophanes.
Khim.sera-i azotorg.soed.sod.v neft.i nefteprod. 3:19-24 '60.
(MIRA 14:6)

1. Moskovskiy gosudarstvennyy universitet imeni M.V.Lomonosova.
(Thiophene)

YUR'YEV, Yu.K.; BELYAKOVA, Z.V.; VOLKOV, V.P.; OSADCHAYA, R.A.;
SHAYDEROVA, L.P.

Tetraacyloxysilanes in organic synthesis. Part 28: Acylation of benzene by silicon-β-chloropropionic and γ-chlorobutyric anhydrides. Part 29: Preparation of organic acid anhydrides from their silicon anhydrides. Vest.Mosk.un.Ser. 2: Khim. 15 no.1:61-67 '60. (MIRA 13:7)

1. Kafedra organicheskoy khimii Moskovskogo universiteta.
(Silicon organic compounds)
(Anhydrides)
(Acylation)

YUR'YEV, Yu.K.; REVENKO, O.M.

Synthesis of 2-alkyltetrahydropyrans. Vest. Mosk un. Ser. 2: Khim. 15 no.4:74-76 Jl-Ag '60. (MIRA 13:9)

1. Kafedra organicheskoy khimii Moskovskogo universiteta.
(Pyran)

YUR'YEV, YU. K.

pa

81974
S/074/60/029/07/01/004
B020/B068

5.3700

AUTHORS: Yur'yev, Yu. K., Belyakova, Z. V.

TITLE: Acyloxysilanes

PERIODICAL: Uspekhi khimii, 1960, Vol. 29, No. 7, pp. 809-832

TEXT: Acyloxysilanes covered in this review are monomeric organosilicon compounds with at least one acyloxy group bound to the silicon atom. Well-known types of acyloxysilanes are tetraacyloxysilanes with the general formula $Si(OCOR)_4$, alkylacyloxysilanes $R_xSi(OCOR')_{4-x}$, alkoxyacyloxysilanes $(RO)_xSi(OCOR')_{4-x}$, alkylalkoxyacyloxysilanes $R_x(R'O)_ySi(OCOR'')_{4-x-y}$, and acyloxytrichlorosilanes $Cl_3SiOCOR$. Compounds like aminoacyloxysilanes $(NH_2)_xSi(OCOR)_{4-x}$ and (alkylthio)-acyloxysilanes $(RS)_xSi(OCOR)_{4-x}$ are unknown. From the day when the first acyloxysilane - tetraacetoxysilane - was synthetized (1868) until 1947, only 15 papers

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Acyloxysilanes

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B020/B068

dealing with these compounds had been published, while during the last 10 to 12 years more than 100 theoretical and practical papers have appeared on this subject. This is due to their growing practical importance. A review of methods used for the preparation of tetra-acyloxysilanes and acyloxytrichlorosilanes, alkyl- and arylacyloxysilanes, alkylalkoxyacyloxy- and alkoxyacyloxysilanes, as well as of the reactions of these compounds is given. Yu. N. Vol'nov, G. B. Yelyakov, V. P. Volkov, B. N. Dolgov, N. P. Kharitonov, M. G. Voronkov, I. G. Khaskin, V. P. Davydova, K. D. Petrov, M. I. Itkina, Ye. S. Lagucheva, A. N. Nesmeyanov, E. G. Perevalova, A. N. Vysokosov, K. A. Andrianov, V. G. Dubrovina, and T. N. Ganina are mentioned. There are 151 references: 74 Soviet, 28 US, 18 British, 18 German, 7 French, 2 Italian, 1 Austrian, 1 Swedish, 1 Indian, and 1 Japanese.

ASSOCIATION: Khimicheskiy fakul'tet MGU im. M. V. Lomonosova (Chemical Department of Moscow State University imeni M. V. Lomonosov)

Card 2/2

77358
SOV/79-30-1-19/78

5.3610

AUTHORS: Yur'yev, Yu. K., Zaytseva, Ye. L.

TITLE: Chemistry of Selenophene. XXIV. Condensation of 5-Nitroselenophene-2-aldehyde and β-(5-Nitroselenienyl-2)-acrolein With Hydrazine Derivatives

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 1, pp 98-101 (USSR)

ABSTRACT: Condensation of 5-nitroselenophene-2-aldehyde and β-(5-nitroselenienyl-2)-acrolein with hydrazides of cyanacetic, furancarboxylic, and isonicotinic acids yields the following compounds, not described in literature: 1-(5-nitroselenenal-2)-2- cyanoacetylhydrazone, yield 93%, mp 241-242°; 1-(5-nitroselenenal-2)-2-(2-furoyl)hydrazone, yield ≈ 100%, mp 266-267° (decomp, from alcohol); 1-[β -(5-nitroselenienyl-2)-allylidene] -2-cyanoacetylhydrazone, yield ≈ 100%, mp 219-221° (decomp, from alcohol); 1-[β -(5-nitroselenienyl-2)-allylidene] -2-(2-furoyl)hydrazone, yield 96%, mp 225-227° (decomp, from alcohol); 1-(5-nitroselenenal-2)-2-

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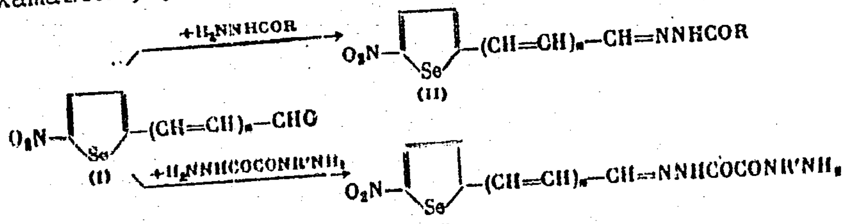
isonicotinoylhydrazone, yield 91%, mp 243-244° (decomp, from alcohol); 1-[β-(5-nitroselenienyl-2)allylidene]-2-isonicotinoylhydrazone, yield 94%, mp 244-245° (decomp, from alcohol). Reaction of 5-nitroselenophene-2-aldehyde with semioxamazide and 5-(β-hydroxyethyl)-semioxamazide yields the following semioxamazones, not described in literature: 1-(5-nitroselenenal-2)semioxamazone, yield ≈ 100%, mp 252-253° (decomp, from alcohol), and 1-(5-nitroselenenal-2)-5-(β-hydroxyethyl)-semioxamazone, yield 90%, mp 251-252° (decomp, from alcohol).



(I $n = 0$ & 1); (II $n = 0$ & 1; R = CH_2CN, C_4H_3O, C_5H_4N); (III $n = 0$; R' = H, CH_2CH_2OH).

Chemistry of Selenophene. XXIV

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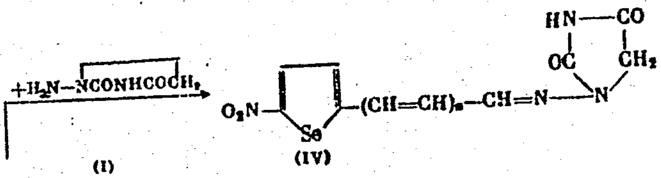
SOV/79-30-1-19/78

Condensation of 5-nitroselenophene-2-aldehyde and β-(5-nitroselenienyl-2)-acrolein with 1-aminohydantoin and 1-amino-2-thiohydantoin yields the following compounds, not described in literature: 1-(5-nitroselenenal-2)-aminohydantoin, yield 81.5%, mp 263-264° (decomp, from alcohol); 1-[β-(5-nitroselenienyl-2) allylidene] aminohydantoin, yield 84%, mp 262-264° (decomp., from alcohol); 1-(5-nitroselenenal-2)amino-2-thiohydantoin, yield 95%, mp 248-250° (decomp., from alcohol); and 1-[β-(5-nitroselenienyl-2)allylidene] amino-2-thiohydantoin, yield 93%, mp 265-267°(decomp., from acetone), respectively.



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Chemistry of Selenophene. XXIV

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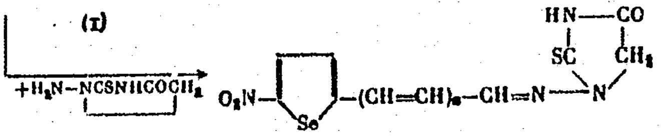
SOV/79-30-1-19/78



(V)

I, IV & V n = 0 & 1.

There are 4 references, 2 Soviet, 2 Japanese.

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

SUBMITTED: January 5, 1959

Card 4/4

5.3600.

77860
SOV/79-30-2-11/78

AUTHORS: Yur'yev, Yu. K., Rozantsev, E. G.

TITLE: Catalytic Conversions of Heterocyclic Compounds. LV. Synthesis of 2,5-Dimethyl-3-Alkyl- and 2,5-Dimethyl-3-Arylthiophanes

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 2, pp 406-410 (USSR)

ABSTRACT: The authors converted 2,5-dimethyl-3-alkylfuranidines into corresponding 2,5-dimethyl-3-alkylthiophanes (aluminum oxide or thorium dioxide were used as catalysts for the reaction which was conducted at 330°; the space velocity of hydrogen sulfide was 0.3 hr^{-1}). Constants and yields of the obtained (new) products are listed in Table A. (1) Name of the compound; (2) bp (pressure in mm); (3) found; (4) calculated; (5) Yield (in %); (6) Results of analysis for sulfur (in %); (7) 2,5-Dimethyl-3-butylthiophane; (8) 2,5-Dimethyl-3-isobutylthiophane; (9) 2,5-Dimethyl-3-amylthiophane; (10) 2,5-Dimethyl-3-

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Moscow State Univ

Catalytic Conversions of Heterocyclic Compounds. 77860
LV. Synthesis of 2,5-Dimethyl-3-Alkyl-and SOV/79-30-2-11/78
2,5-Dimethyl-3-Arylthiophanes

-isoamylthiophane; (11) 2,5-Dimethyl-3-cyclohexylthiophane; (12) 2,5-Dimethyl-3-phenylthiophane; (13) 2,5-Dimethyl-benzylthiophane.

Table A. Constants and yields of 2,5-dimethyl-3-alkyl-(or aryl)-thiophanes.

1	2	n_D^{20}	d_4^{20}	MR_D		5	6	
				3	4		3	4
7	210—211° (748)	1.4757	0.8930	54.40	54.18	26	18.34, 18.37	18.61
8	208—209 (752)	1.4740	0.8922	54.27	54.18	36	18.33, 18.31	18.61
9	237—238 (765)	1.4734	0.8868	58.99	58.80	22	16.96, 16.97	17.20
10	233—234 (752)	1.4736	0.8862	59.07	58.80	19	16.91, 16.93	17.20
11	117—118 (10)	1.5510	1.0102	62.05	61.71	35	15.80, 15.79	16.16
12	95—96 (3)	1.5650	1.0260	61.05	60.20	11.20*	16.29, 16.34	16.67
13	111—112 (3)	1.5300	0.9783	65.15	64.86	21.41*	15.22, 15.29	15.54

* The catalyst is thorium oxide on alumina (25% of ThO_2).

Card 2/3

Catalytic Conversions of Heterocyclic
Compounds. LV. Synthesis of 2,5-Di-
methyl-3-Alkyl- and 2,5-Dimethyl-3-
-Arylthiophanes

77860
SOV/79-30-2-11/78

It was noted that the speed for the catalytic reaction of 2,5-dimethyl-3-butylfuranidine is only half that for the 2,5-dimethyl-3-propylfuranidine. This is explained by steric hindrance caused by the longer butyl radical in the β -position to the furanidine cycle and in trans-position to the α -radical (see Fig. 5). Due to the side reactions (dehydration and elimination of H_2S from the intermediate 1,4-mercaptooxy-compound), the yields of 2,5-dimethyl-3-arylthiophanes are very low. For these compounds (see note to Table A) thorium oxide was found to give better yields than alumina. There are 7 figures; 1 table; and 14 references, 12 Soviet, 1 Japanese, 1 U.S. The U.S. reference is: D. Rank, N. Sheppard, G. Szasz, J. Chem. Phys., 17, 831 (1950).

Card 3/3

5.3400

77861
SOV/79-30-2-12/78

AUTHORS: Yur'yev, Yu. K., Zefirov, N.S., Shteynman, A. A., Gurevich, V. M.

TITLE: Study of the Furan Series. III. Reaction of 2-Methyl- and 2-Ethylfuran with Mesityl Oxide

PERIODICAL: Zhurnal obshchei khimii, 1960, Vol 30, Nr 2, pp 411-415 (USSR)

ABSTRACT: The authors synthesized 1,1-dimethyl-1-(5-methylfuryl-2) butanone-3 (I) and 1,1-dimethyl-1-(5-ethylfuryl-2) butanone-3 (II) by reacting mesityl oxide with 2-methyl- and 2-ethylfuran, respectively, demonstrating that the furan ring can react with β, β-dimethylvinyl group of the α - β-unsaturated ketones (see scheme A).

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Study of the Furan Series. III. Reaction of 2-Methyl- and 2-Ethylfuran with Mesityl Oxide

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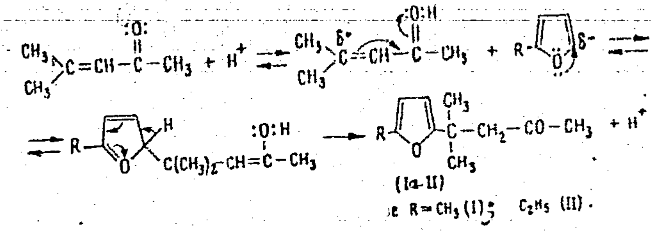

Scheme A

This reaction was conducted in a round-bottom flask provided with a mixer and a reflex condenser. The reaction mixture (the reagents were dissolved in hydroquinone) was heated for 8 hr on the water bath. The reaction mass was then diluted with ether, washed with sodium carbonate and water, and dried over $CaCl_2$. The best catalysts were found to be concentrated sulfuric acid and boron trifluoride etherate. Repeated distill-

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Study of the Furan Series. III. Reaction of 2-Methyl- and 2-Ethylfuran with Mesityl Oxide

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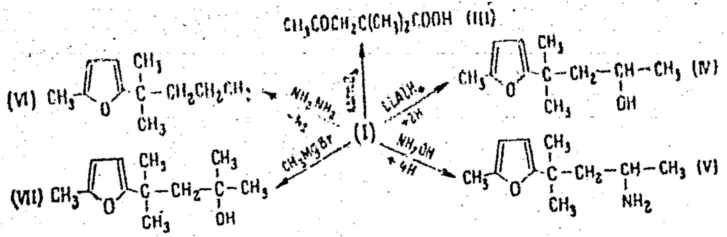
ation yielded the addition products. Characteristics of 1,1-dimethyl-1-(5-methylfuryl-2)butanone-3 (I): bp 106-107° (15 mm); n_D^{20} 1.4700; d_4^{20} 0.9723; its semicarbazone, white leaflets, mp 136-137°, 2,4-dinitrophenylhydrazone, yellow needles; mp 109.5-110°, was characterized by infrared spectrum. The 1,1-dimethyl-1-(5-ethylfuryl-2)butanone-3 (II): bp 114° (13 mm); n_D^{20} 1.4682; d_4^{20} 0.9577; 2,4-dinitrophenylhydrazone, orange needles; mp 90.5-91°, characterized by infrared spectrum. Reactions of prepared ketones were studied on example of 1,1-dimethyl-1-(5-methylfuryl-2)butanone-3. Scheme B shows the reactants and the products of the five reactions studied.

Card 3/5

Study of the Furan Series. III. Reaction of 2-Methyl- and 2-Ethylfuran with Mesityl Oxide

77861
SOV-79-30-2-12/78



Scheme B

The constants of the derived compounds: (1) α, α dimethyllevulinic acid (III): mp 76-76.5°; (2) 1,1-dimethyl-1-(5-methylfuryl-2)butanol-3 (IV), bp 106° (10 mm), n_D^{20} 1.4770, d_4^{20} 0.9690, characterized by infrared spectrum; (3) 1,1-dimethyl-1-(5-methylfuryl-2)-3-aminobutane (V), bp 104-105° (10 mm), n_D^{20} 1.4580, d_4^{20} 0.8814; (4) 1,1-

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Study of the Furan Series. III. Reaction of 2-Methyl- and 2-Ethylfuran with Mesityl Oxide

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-dimethyl-1-(5-methylfuryl-2)butane (VI): bp 75-76° (20 mm), n_D^{20} 1.4529, d_4^{20} 0.8738; (5) 1,1,3-trimethyl-1-(5-methylfuryl-2)butanol-3 (VII): bp 89-90° (6mm), n_D^{20} 1.4800, d_4^{20} 0.9703. The authors thank L. A. Kazitsyna for measurement of spectra. There are 8 references, 3 Soviet, 3 German, 1 French, 1 U.S. The U.S. reference is Ch. A., 47, 1744 (1953).

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

SUBMITTED: February 4, 1959

Card 5/5

5.3400,5.3600,5.3700

77862
SOV/79-30-2-13/78

AUTHORS: Yur'Yev, Yu. K., Belyakova, Z. V., Kostetskiy, P. V., ~~Prokof'yev, A. I.~~

TITLE: Triacyloxyboranes and Tetraacetooxygermanium in Acylation of Benzene and Thiophene

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 2, pp 415-420 (USSR)

ABSTRACT: The authors studied relative acylating ability of mixed anhydrides of various organic acids (acetic, propionic, and butyric) and inorganic acids, $B(OH)_3$, $Al(OH)_3$, $Ge(OH)_4$, and $Si(OH)_4$. The acylation of benzene was performed with the anhydrides formed in situ from the organic acid and the halides of the nonmetals (boron bromide, germanium tetrachloride, aluminum chloride, and silicon tetrachloride) in benzene solution in presence of anhydrous aluminum chloride. If the latter is absent, the mixed anhydrides can be isolated. The yields of the synthesized ketones are shown in Table 1.

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Triacyloxyboranes and Tetraacetooxygermanium in Acylation of Benzene and Thiophene

Table 1.

(A)	(B)			(C)
	$R=CH_3$	$R=C_2H_5$	$R=n\text{-}C_3H_7$	
(D)	66	56	63.3	$7.3 \cdot 10^{-10}$
(E)	47	49.5	55	$2.2 \cdot 10^{-10}$
(F)	31	—	—	$6 \cdot 10^{-12}$
(G)	28	—	—	

Key to Table 1: (A) Acylating agent; (B) Yield of the ketone C_6H_5COR (in %); (C) The first dissociation constant of inorganic acid of the mixed anhydride; (D) Boroanhydride of organic acid (boran triacetate); (E) Silicon anhydride of organic acid (silicon tetraacetate); (F) Aluminum triacetate; (G) Germanium triacetate

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Triacyloxyboranes and Tetraaceto-
oxygermanium in Acylation of Benzene
and Thiophene

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It can be seen that the acylation capacity of the mixed anhydrides decreases with decreasing acidity of the inorganic acid:

$$B(OH)_3 > Si(OH)_4 > Al(OH)_3 > Ge(OH)_4$$

Thiophene was acylated by boroanhydrides of acetic and butyric acids and by germanium tetraacetate (yields of acetothienone and propyl-2-thienyl ketone were 68.5%) using stannic chloride as catalyst. There are 2 tables and 20 references, 5 Soviet, 6 German, 1 French, 1 Finnish, 1 U.K., and 6 U.S. The 5 most recent U.K. and U.S. re references are: D. Tarbell, J. Price, J. Org. Chem., 22, 245 (1957); H. Anderson, J. Am. Chem. Soc., 74 2371 (1952); Chem. Abst., 41, 5481 (1947); H. Cook, J. Ilett, B. Saunders, G. Stacey, J. Chem. Soc., 1950, 3125; J. Johnson, J. Am. Chem. Soc., 73, 5888 (1951).

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

SUBMITTED: February 9, 1959

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5.3600

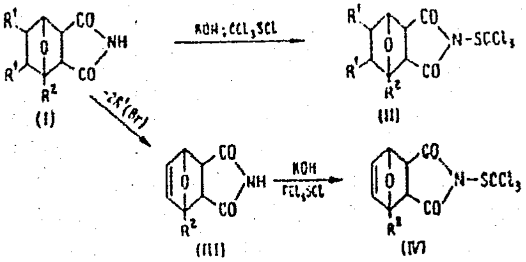
78270
SOV/79-30-3-24/69

AUTHORS: Yur'yev, Yu. K., Zefirov, N. S.

TITLE: Investigation of Furan Series. V. Synthesis of Derivatives of N-Trichloromethylmarcaptoimide of 3,6-Endoxohexahydrophthalic Acid

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 3, pp 855-859 (USSR)

ABSTRACT: The following two methods were used in the synthesis of the above derivatives.



(I; II): $R^1 = R^2 = H$; $R^1 = Cl$, $R^2 = H$; $R^1 = Cl$, $R^2 = CH_3$; $R^1 = Br$, $R^2 = H$; $R^1 = H$, $R^2 = CH_3$; $R^1 = H$, $R^2 = CH_2OCOCH_3$; (III; IV): $R^2 = H$;

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Investigation of Furan Series. V.

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SOV/79-30-3-24/69

The reaction of perchloromethylmercaptan with an alkaline solution of the imide of 3,6-endoxyhexahydrophthalic acid or its derivatives is preferred because of a high yield (80-90%) of corresponding N-trichloromethylmercapto derivatives (without forming the amide salt). The following compounds were synthesized: N-trichloromethylmercaptoimide of exo-3,6-endoxohyxahydrophthalic acid, mp 157.5-158°; N-trichloromethylmercaptoimide of exo-3,6-endoxo-Δ^4-tetrahydrophthalic acid (yield 83%), mp 132-132.5°; N-trichloromethylmercaptoimide of exo-4,5-dibromo-3,6-endoxohexahydrophthalic acid (yield 87%), mp 184-185°; N-trichloromethylmercaptoimide of exo-4,5,-dichloro-3,6-endoxohexahydrophthalic acid (yield 81%), mp 169.5-170°; N-trichloromethylmercaptoimide of 3-methyl-3,6-endoxohexahydrophthalic acid (yield 85%), mp 148.5-149°; N-trichloromethylmercaptoimide of 4,5-dichlor-3-methyl-3,6-endoxohexahydrophthalic acid (yield 83%), mp 214-215°; N-trichloromethylmercaptoimide of 3-acetoxymethyl-3,6-endoxohexa-

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Investigation of Furan Series. V.

78270
SOV/79-30-3-24/69

hydrophthalic acid (yield 78%), mp 158°. There are 17 references, 13 U.S., 2 Soviet, 2 German. The 5 most recent U.S. references are: Groxall W., Shropshire, E., Lo C., J. Am. Chem. Soc., 75, 5420 (1953); Stoltz E., Rogers C., Ch. A., 48, 9085 (1954); Kittleson A., J. Agr. Food Chem., 1, 677 (1953); Berson, J., Swidler R., J. Am. Chem. Soc., 76, 4060 (1954); Kwart H., Burchuk J., J. Am. Chem. Soc., 74, 3094 (1952).

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

SUBMITTED: March 12, 1959

Card 3/3

5.3600

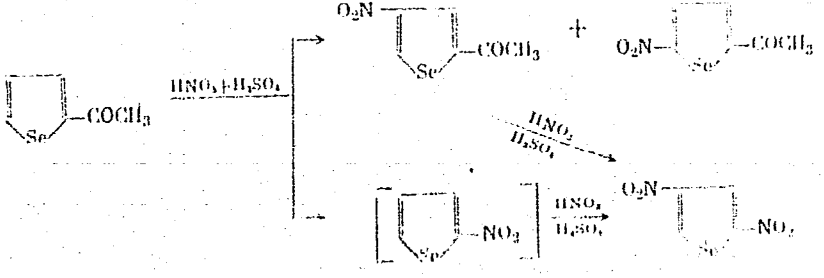
78271
SOV/79-30-3-25/69

AUTHORS: Yur'yev, Yu. K., Zaytseva, Ye. L.

TITLE: Chemistry of Selenophene. XXV. 4-Nitro-2-Acetoselenophene and 4-Nitroselenophene-2-Carboxylic Acid

PERIODICAL: Zhurnal obshchey khimii, 1950, Vol 30, Nr 3, pp 859-864 (USSR)

ABSTRACT: Nitration of 2-acetoselenophene yields a mixture of two products: 4-nitro-2-acetoselenophene (yield 50%), mp 123-123.5° and 5-nitro-2-acetoselenophene (yield 8.5%), and 2,4-dinitroselenophene (yield 41.5%), mp 78.5-79°.



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Chemistry of Selenophene. XXV. 4-Nitro-2-Acetoselenophene and 4-Nitroselenophene-2-Carboxylic Acid

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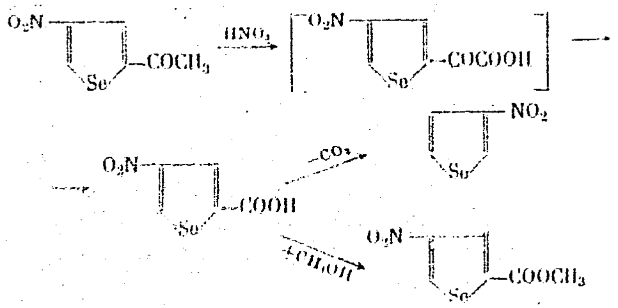
Oxidation of 4-nitro-2-acetoselenophene with dilute nitric acid yields a mixture of 4-nitroselenophene-2-carboxylic acid and 4-nitroselenophene-2-glyoxylic acid, which on further oxidation with hydrogen peroxide, yields 4-nitroselenophene-2-carboxylic acid (yield 36%), mp 170-171°. Esterification of this acid yields the methyl ester of 4-nitroselenophene-2-carboxylic acid (yield 78%), mp 103.5-104°, and decarboxylation, 3-nitroselenophene (yield 50%), mp 77.5-78°.



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Chemistry of Selenophene. XXV. 4-Nitro-2-Acetoselenophene and 4-Nitroselenophene-2-Carboxylic Acid

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SOV/79-30-3-25/69

The ultraviolet absorption maxima, of 4-nitro- and 5-nitro-2-acetoselenophene are 260 mμ and 315 mμ, respectively. There are 2 figures; and 11 references, 4 Soviet, 2 U.S., 2 Dutch, 1 U.K., 1 German, 1 Japanese. The U.S. references are: Blatt, A., Bach, S., Kresch, L., J. Org. Chem., 22, 1693 (1957); Fove, W. O., Heffern, J. J., Feldman, E. I., J. Am. Chem. Soc., 76, 1378 (1954).

ASSOCIATION: Moscow State University (Moskovskiy gosudarstvennyy universitet)

SUBMITTED: March 20, 1959

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S/079/60/030/05/47/074
B005/B016

AUTHORS: Yur'yev, Yu. K., Mezentsova, N. N., Vas'kovskiy, V. Ye.

TITLE: Selenophene Chemistry. XXVI. 2-Cyclopropyl Selenophene and 2-Propenyl Selenophene

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 5, pp. 1628-1631

TEXT: In the present paper the syntheses of 2-cyclopropyl selenophene (I) and 2-propenyl selenophene (II) are described. The authors synthesized (I) on the basis of β-(selenienyl-2-)-acrolein (III). Contrary to a method used previously (Ref. 1), this compound was obtained by condensation of selenophene-2-aldehyde with acetaldehyde in the presence of lye. By treating the unsaturated ketone (III) with hydrazine hydrate, 5-(selenienyl-2')-pyrazoline (IV) was obtained. This product was not isolated but decomposed at once according to the well-known method by N. M. Kishner (with platinized carbon and potassium hydroxide). By this degradation, compound (I) results with impurities of a selenienyl-2-alkene. Compound (I) was also synthesized from the hydrochloride of 2-(β-dimethyl-amino-propio)-selenophene (Ref. 2) by the action of hydrazine hydrate and Kishner degradation of the resultant (selenienyl-2)-pyrazoline. This method is simpler and more convenient than the one described above. The purification of product (I) from the selenienyl-2-alkene impurity was carried out by treatment with potassium permanganate solution and subsequent working up with 2,4-dinitro-benzene-sulfenyl chloride. Contrary to the unstable monosubstituted pyrazoline (IV), the disubstituted pyrazoline derivative 3-methyl-5-(selenienyl-2')-pyrazoline (V) obtained by condensation of selenal-2-acetone with hydrazine hydrate is a stable compound which is distillable in vacuo without decomposition. In the same way, 1-phenyl-5-(selenienyl-2')-pyrazoline was prepared by condensation of selenal-2-acetone with phenyl hydrazine. This product melts without decomposition. On degradation of compound (V) according to Kishner, 2-(2'-methyl-cyclopropyl)-selenophene (VI) results. This product is contaminated by small quantities of selenienyl-2-butene which may be separated in the above-mentioned way. The ultraviolet absorption spectra of methanolic solutions of compounds (I) and (VI) show no differences in the electron transitions. The spectra were taken on an SF-4 spectrophotometer. Compound (II) was obtained from ethyl-(selenienyl-2)-carbinol by dehydration with potassium bisulfate. The initial product was produced

Selenophene Chemistry. XXVI. 2-Cyclopropyl Selenophene and 2-Propenyl Selenophene

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B005/B016

by an organomagnesium synthesis from selenophene-2-aldehyde and ethyl bromide. In an experimental part, all operations performed are described in detail. For each of the resultant products, yield, boiling (or melting) point, refractive index, density, molar refractivity, and data of the ultimate analysis are given. The schemes of the reactions performed are presented as well. R. Ya. Levina and co-workers (Ref. 5) are mentioned in this paper. There are 6 Soviet references.

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ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: June 12, 1959

Card 3/3

NOVITSKIY, K.Yu.; YUR'YEV, Yu.K.; AFANAS'YEVA, Yu.A.; BOLESOV, I.G.;
OLEYNIK, A.F.

Furan series. Part 6: β-Chloroethylamines of the furan series. Zhur.ob.khim. 30 no.7:2199-2202 Jl '60.
(MIRA 13:7)

1. Moskovskiy gosudarstvennyy universitet.
(Furfurylamine) (Thionyl chloride)
(Furan)

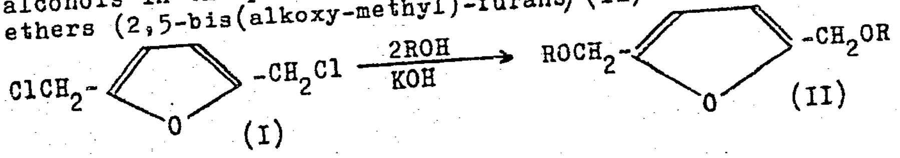
S/079/60/030/007/027/039/XX
B001/B066

AUTHORS: Novitskiy, K. Yu., Volkov, V. P., Kostetskiy, P. V., and Yur'yev, Yu. K.

TITLE: Investigation in the Furan Series. VII. 2,5-Bis(chloro-methyl)-furan in the Synthesis of 2,5-Bis(alkoxy-methyl)-furan and 2,5-Bis(alkyl-mercapto-methyl)-furan

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 7, pp. 2203-2207

TEXT: Following Ref. 1 the authors synthesized 2,5-bis(alkoxy-methyl)- and 2,5-bis(alkyl-mercapto-methyl)-furan by treating 2,5-bis(chloro-methyl)-furan with alcohols and mercaptans. The substitution of alkoxy groups for two chlorine atoms in compound (I) takes readily place under the action of alcohols in the presence of alkali lye; the yield of the corresponding ethers (2,5-bis(alkoxy-methyl)-furans) (II) was between 60 and 70%.



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Investigation in the Furan Series. VII. 2,5-Bis(chloro-methyl)-furan in the Synthesis of 2,5-Bis(alkoxy-methyl)- and 2,5-Bis(alkyl-mercapto-methyl)-furan

S/079/60/030/007/027/039/XX
B001/B066

($R = CH_3$, C_2H_5, $n - C_4H_9$, $n - C_5H_{11}$). When using the corresponding alcoholates instead of alcohols, the ether yield increases to 74-82%. 2,5-bis(methoxy-methyl)-furan was also obtained by methylation of 2,5-bis(hydroxy-methyl)-furan. The latter was synthesized from the acetoxy derivative. The reaction of thiourea with 2,5-bis(chloro-methyl)-furan, followed by cleavage of the resultant bis-isothiuronium salt gave 2,5-bis(mercapto-methyl)-furan (III). The high mobility of chlorine in 2,5-bis(chloro-methyl)-furan is seen in its reaction with sodium mercaptides. The corresponding furan derivatives (IV) were obtained in yields of between 72 and 89%; the yield of 2,5-bis(phenyl-mercapto-methyl)-furan was 60%. The two first-mentioned representatives of the resultant bis-sulfides ($R = C_3H_7$, iso-C_4H_9) form crystalline complexes with $HgCl_2$, while the latter form oils. There are 1 table and 5 references: 2 Soviet, 1 Dutch, 1 British, and 1 German.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 15, 1959

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S/079/60/030/007/028/039/XX
B001/B066

AUTHORS: Yur'yev, Yu. K., Zaytseva, Ye. L., and Rozantsev, G. G.

TITLE: Chemistry of Selenophene. XXVII. Composition of the Nitration Product of Selenophene

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 7, pp. 2207-2209

TEXT: It may be seen from the papers of Refs. 1-5 on the nitration of selenophene that the largest component of the reaction product obtained by nitration of selenophene is 2-nitro-selenophene in its α-form, and that the α-form, being a lower-melting form which is more easily soluble, "decrystallizes" only after further treatment, i.e., by separating the crystals from the oil fraction and by repeated crystallization. This assumption is supported by the fact that the α-form is lost to a larger extent than the β-form, i.e., 3-nitro-selenophene. The loss in the α-form and the concentration of the β-form in crystals last until both begin to crystallize in the ratio mentioned above. To confirm the correctness of this conclusion, the ultraviolet absorption spectra of pure 2-nitro- and 3-nitro-selenophene were studied (Ref. 1) and compared with the spectrum of

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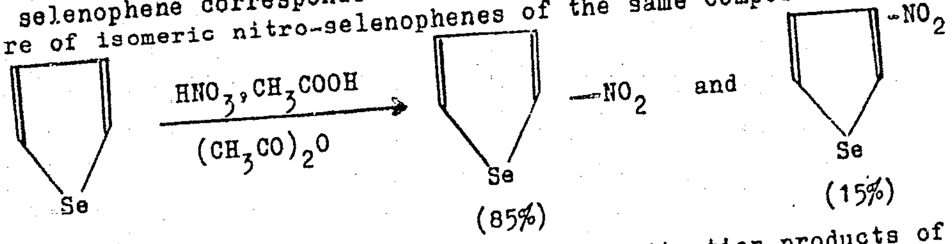
the nitration product of selenophene, which was taken by the method of S. Umezawa (Ref. 2) in pure condition. A comparison of the curves of the ultraviolet spectra of the nitro-selenophene samples and of the nitration product of selenophene (Diagram) confirmed the authors' assumption and indicated that the latter compound is a mixture of mononitro-selenophenes in which the α-isomer is actually predominant: The content of 2-nitro-selenophene in the mixture is 85%, whereas 3-nitro-selenophene has only a 15% yield. The adsorption curve of this mixture of nitration products of selenophene corresponds to the adsorption curve of an artificial mixture of isomeric nitro-selenophenes of the same composition:



By improving the method of separating the nitration products of selenophene from the reaction mixture it was possible to increase the nitro-

Chemistry of Selenophene. XXVII. Composition of the Nitration Product of Selenophene S/079/60/030/007/028/039/XX B001/B066

selenophene yield from 15 to 25%. A higher yield is, apparently, prevented by the considerable resinification of the product in the course of reaction. There are 1 figure, 1 table, and 5 references: 1 Soviet, 1 French, 2 German, and 1 Japanese.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 1, 1959

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Card 3/3

S/079/60/030/007/029/039/XX
B001/B066

AUTHORS: Yur'yev, Yu. K., Zaytseva, Ye. L., and Nikiforova, A. N.

TITLE: Chemistry of Selenophene. XXVIII. Reactions of 4-Nitro- and 5-Nitro-2-acetoselenophene

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 7, pp. 2209-2214

TEXT: The authors of the present paper synthesized derivatives of 5-nitro- and 4-nitro-2-acetoselenophenes which they had obtained in Refs. 1, 2. The former was condensed with various hydrazine derivatives by a method described in Ref. 3. The following compounds resulted: 4-phenyl semicarbazone (96%), isonicotinoyl hydrazone (60%), furoyl hydrazone (33.5%), and cyano-acetyl hydrazone (83.5%) of 5-nitro-2-acetoselenophene. Bromination of 5-nitro- and 4-nitro-2-acetoselenophene was made with bromine in glacial acetic acid and with dioxane dibromide. When treating 5-nitro-2-acetoselenophene with bromine in glacial acetic acid at 80°C, the authors obtained 5-nitro-2-bromo-acetoselenophene (73.5%), but also resinous by-products and, apparently, some dibromide. Bromination of this nitro ketone with dioxane dibromide at room temperature gave a fairly pure 5-nitro-2-bromo-acetoselenophene (80%). 4-nitro-2-acetoselenophene did not react

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Chemistry of Selenophene. XXVIII. Reactions of 4-Nitro- and 5-Nitro-2-acetoselenophene

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in this way. Its bromination was only possible with bromine in glacial acetic acid (85.5%). Both nitro-2-bromo-acetoselenophenes were allowed to react with urotropin to convert them to the corresponding α-amino ketones of the selenophene series. In the first stage of this synthesis, the complex of 4-nitro-2-bromo-acetoselenophene with urotropin is formed easily (73%) when mixing the components in an equimolecular ratio in chloroform, and when the mixture is allowed to stand for two days at room temperature. This was not possible in the case of 5-nitro-2-acetoselenophene since the complex yield was only 38%. When the reaction was carried out in dry chloro benzene at 50° by the method of Ref. 7, the urotropin complex of 5-nitro-2-bromo-acetoselenophene was obtained in an 83% yield. Hydrolysis of the complex of 4-nitro-2-bromo-acetoselenophene with urotropin took place easily with a mixture of alcohol and concentrated hydrochloric acid in the cold within 48 hours (Ref. 7). Hydrolysis of the complex of 5-nitro-2-bromo-acetoselenophene with urotropin was only possible with a much smaller quantity of hydrochloric acid in alcohol and by distilling off the resultant diethyl formal. The hydrolysis of these two complexes, with subsequent acetylation, thus gives 4-nitro- and 5-nitro-2-acetyl-amino-acetoselenophenes. The authors mention a paper by N. O. Saldakol. There are 8 references:

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Chemistry of Selenophene. XXVIII. Reactions of 4-Nitro- and 5-Nitro-2-acetoselenophene

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B001/B066

4 Soviet, 1 US, 1 German, and 2 Italian.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: July 10, 1959

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Card 3/3

KOROBITSYNA, I.K.; IN' CHEN'-LE; YUR'YEV, Yu.K.

4-Methylene-2,2,5,5,-tetramethyl-3-furanidone. Zhur.ob. khim. 30 no.7:2214-2218 Jl '60. (MIRA 13:7)

1. Moskovskiy gosudarstvennyy universitet.
(Furanidone)

YUR'YEV, Yu.K.; MEZENTSOVA, N.N.; MONAKHOVA, A.T.

Chemistry of selenophene. Part 29: 5-Bromo- and 5-chloro-2-selenophenal. Zhur.ob.khim. 30 no.8:2726-2731 Ag '60. (MIRA 13:8)

1. Moskovskiy gosudarstvennyy universitet.
(Selenophene)

YUR'YEV, Yu.K.; SADOVAYA, N.K.; LYUBIMOVA, Ye.N.

Chemistry of selenophene. Part 30: chloromethylation of ketones of the selenophene series. Zhur.ob.khim. 30 no.8:2732-2737 Ag '60. (MIRA 13:8)

1. Moskvoskiy gosudarstvennyy universitet.
(Selenophene) (Chloromethylation)

KOROBITSYNA, I.K.; OLEYNIK, A.F.; YUR'YEV, Yu.K.

Acylhydrazones of 2,2,5,5-tetraalkyl-3,4-furanidinediones. Zhur. ob. khim. 30 no.9:2820-2825 S '60. (MIRA 13:9)

1. Moskovskiy gosudarstvennyy universitet.
(Furandione)

84872

S/079/60/030/010/008/030
B001/B075

11.1210

AUTHORS: Yur'yev, Yu. K., Zefirov, N. S., and Minacheva, M. Kh.

TITLE: Investigation of the Furan Series. VIII. Tetramethylfuran in Diene Synthesis

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 10, pp. 3214-3217

TEXT: Following the papers of Refs. 1-9 on the behavior of furan and its derivatives in diene synthesis, the authors studied the behavior of tetramethylfuran in diene synthesis. On the basis of Refs. 10-11, they assumed that diene synthesis in the furan series proceeds according to the ionic mechanism. They observed a vigorous reaction of tetramethylfuran with fumaric nitrile. The principal purpose of the present work was to determine the qualitative difference between the reactivities of tetramethylfuran and furan, and to select those dienophiles which react only with the former. Thus, it was found that tetramethylfuran reacts with methyl maleic anhydride under the formation of a crystalline adduct, whereas furan, 2-methylfuran, and 2,5-dimethylfuran do not react with this dienophile. Chloro- and bromo maleic anhydrides, together with tetramethyl-

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Investigation of the Furan Series. VIII. Tetramethylfuran in Diene Synthesis

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B001/B075

furan, lead to the corresponding addition products, contrary to the non-reactive phenyl maleic anhydride. Phenyl-β-benzoyl-vinyl sulfone and phenyl-β-acetyl-vinyl sulfone also react with tetramethylfuran only. However, contrary to the former, the latter reacts with the furan itself. Thus, it is shown that there is a great difference between the reactivity of tetramethylfuran and that of furan. Attempts to react the former with dimethyl maleic anhydride, benzal malonic acid ester, acrolein, methyl isopropenyl ketone, acrylonitrile, and cinnamic acid aldehyde failed. Apparently, tetramethylfuran and furan are not so reactive as to react with dienophiles whose double bond is activated only from one side (Refs. 14-16). H. Wienhnhaus and H. Dässlep (Ref. 17) used menthofuran for the reaction with acrolein and crotonic acid aldehyde, taking this reaction for a diene synthesis. The failure of the authors' attempt to react tetramethylfuran with acrolein indicates that the data mentioned by the authors (Refs. 15, 16) are possibly incorrect. Further investigations are therefore necessary. There are 17 references: 5 Soviet, 9 US, and 3 German.

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ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: November 20, 1959

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84873

S/079/60/030/010/009/030
B001/B075

11.1260

AUTHORS: Novitskiy, K. Yu., Yur'yev, Yu. K., and Zhingareva, V. N.

TITLE: Investigation of the Furan Series. IX. Synthesis of 2,5-Bis-(amino-methyl) Furans

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 10, pp. 3218-3220

TEXT: F. Gill and H. Ing (Ref. 1) have recently described the synthesis of the symmetric diamines of the furan series. They aminomethylated dimethylfurfuryl-amine with hydrochloric dimethyl amine and, thus, obtained 2,5-bis-(dimethyl-amino-methyl) furan (70%). Proceeding from the methyl ester of pyromucic acid, A. L. Mndzhoyan and his collaborators (Ref. 2) synthesized 2,5-bis-(dipropyl-amino-amethyl) furan. The present paper shows that the reaction of 2,5-bis-(chloro-methyl) furan with secondary aliphatic, aliphatic-aromatic, and heterocyclic amines leads to the corresponding N-substituted 2,5-bis-(amino-methyl) furans in sufficiently good yields. Thus, the following compounds resulted from the action of dimethyl and diethyl amines, N-ethyl aniline, pyrrolidine, piperidine, and morpholine upon 2,5-bis-(chloro-methyl) furan (I): 2,5-bis-(dimethyl-

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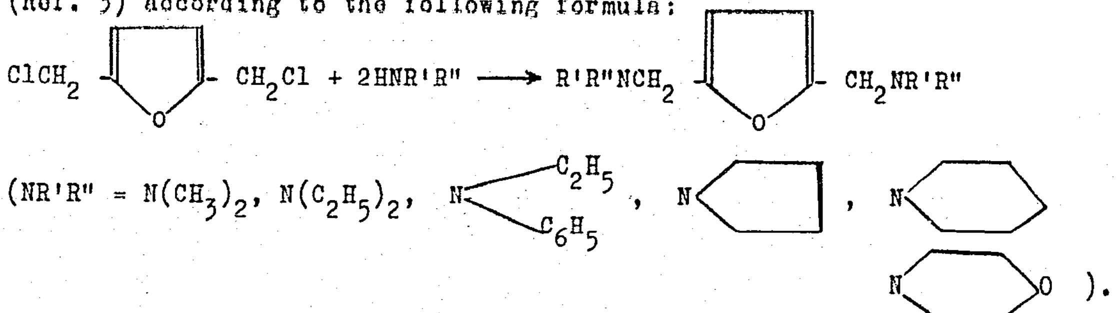
Investigation of the Furan Series. IX. Synthesis of 2,5-Bis-(amino-methyl) Furans

S/079/60/030/010/009/030
B001/B075

amino-methyl) furan (75.5%) (Ref. 3); 2,5-bis-(diethyl-amino-methyl) furan (61%) (Ref. 3); 2,5-bis-(N-ethyl-N-phenyl-amino-methyl) furan (40%); 2,5-bis-(N-pyrrolidino-methyl) furan (62%); 2,5-bis-(N-piperidino-methyl) furan (76.5%) (Ref. 3); and 2,5-bis-(N-morpholino-methyl) furan (62%) (Ref. 3) according to the following formula:



In order to synthesize a doubly primary diamine of the furan series, 2,5-bis-(chloro-methyl) furan was reacted with phthalimide potassium. The resulting diphthalide was reacted with hydrazine hydrate (Ref. 4) to give 2,5-bis-(amino-methyl) furan in a 40% yield. There are 4 references: 2 Soviet and 2 British.

Card 2/3

84873

Investigation of the Furan Series. IX. Synthesis of 2,5-Bis-(amino-methyl) Furans

S/079/60/030/010/009/030
B001/B075

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: November 1959

X

Card 3/3

86505

5.3700 2209, 1282, 1273

S/079/60/030/011/011/026
B001/B066

AUTHORS: Yur'yev, Yu. K., Zaytseva, Ye. L., and Rozantsev, G. G.

TITLE: Chemistry of Selenophene. XXXI. Reactions of the Acid Chloride of 5-Nitro-selenophene-2-carboxylic Acid and of 5-Nitro-2-diazoacetoselenophene

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 11, pp.3672-3675

TEXT: In the present paper, the above acid chloride (I), from which 5-nitro-2-acetoselenophene had been obtained previously (Ref.1), was used in the synthesis of a number of substituted amides of 5-nitro-selenophene-2-carboxylic acid (II), as well as of ω-derivatives of 5-nitro-2-acetoselenophene. On reaction of this acid chloride with dimethyl amine, pyrrolidine, piperidine, morpholine, the dimethyl amide of 5-nitro-selenophene-2-carboxylic acid; 1-(5'-nitro-selenenoyl-2')-pyrrolidine; 1-(5'-nitro-selenenoyl-2')-piperidine; N-(5-nitro-selenenoyl-2)-morpholine were synthesized accordingly. Compound (I) was also allowed to react with diazomethane which gave 5-nitro-2-diazoaceto-selenophene (III) in a yield of 70.5%. The ether solution of diazomethane

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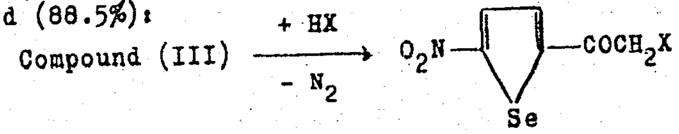
86505

Chemistry of Selenophene. XXXI. Reactions of the Acid Chloride of 5-Nitro-selenophene-2-carboxylic Acid and of 5-Nitro-2-diazoacetoselenophene

S/079/60/030/011/011/026
B001/B066

obtained from nitroso-methyl urea (Ref.2) had to be first distilled since also traces of alkali lye cause a resinification and decrease the yield. According to the US patent (Ref.3), 5-nitro-2-diazoacetofuran was obtained in a yield of 83.5% by this method in the nitrofuran series by reaction of the acid chloride of the corresponding acid with diazomethane; in the thiophene series, this reaction has so far not been investigated. On hydrolysis of 5-nitro-2-diazoacetoselenophene with dilute sulfuric acid, the authors obtained 5-nitro-2-hydroxy-acetoselenophene (IV) in good yield (96%). By treating diazo ketone with HCl or HBr, 5-nitro-2-chloro-acetoselenophenes (V) is formed (92.5%), or, accordingly, the bromine product (VI) (84%); on treatment with acetic acid, the compound (VII) was obtained (88.5%):



(IV) X = OH, (V) X = Cl, (VI) X = Br, (VII) X = $OCOCH_3$.

Card 2/3

86505

Chemistry of Selenophene. XXXI. Reactions of the Acid Chloride of 5-Nitro-selenophene-2-carboxylic Acid and of 5-Nitro-2-diazoacetoselenophene

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In the same way, 5-nitro-2-chloro-acetofuran (96%) and 5-nitro-2-bromo-acetofuran (85.5%) (Ref.4) were synthesized. There are 5 references: 4 Soviet and 1 US.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet (Moscow State University)

SUBMITTED: January 1, 1960

X

Card 3/3

YUR'YEV, Yu.K.; ZEFIROV, N.S.; SHTEYNMAN, A.A.; RYBOYEDOV, V.I.

Furan series. Part 10: 2-Methylfuran in a reaction of substitutive addition with α,β-unsaturated aliphatic ketones. Zhur. ob. khim. 30 no.11:3755-3759 N'60. (MIRA 13:11)

1. Moskovskiy gosudarstvennyy universitet.
(Furan) (Ketones)

S/661/61/000/006/017/081
D205/D302

AUTHOR: Yur'yev, Yu. K.

TITLE: Catalytic synthesis and transformations of tetramethylene silane

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, 94-95

TEXT: The catalytic transformation of heterocyclics was attempted by subjecting tetrahydrofuran to the action of silane in the presence of Al_2O_3 at 375°C. Tetramethylene silane (4% yield) and ditetramethylene silane (2% yield) were obtained. Analogously, tetrahydropyran was converted by silane at 375°C in the presence of Al_2O_3 into pentamethylene silane with a yield of 9.5%. Pentamethylene silane is converted at 375°C by H_2S in the presence of Al_2O_3

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Catalytic synthesis and ... S/661/61/000/006/017/081
D205/D302

into pentathiophane with a yield of 24%. The attempt to convert tetrahydrofuran into diethyl tetramethylene silane by the action of diethyl silane was unsuccessful, owing to the decomposition of diethyl silane on contact with Al_2O_3. Thus, the conversion of oxygen-containing five- and six-membered heterocyclics into silicon-containing heterocyclics was achieved. There is 1 non-Soviet-bloc reference. The reference to the English-language publication reads as follows: J. Am. Chem. Soc., 80, 5418, (1958).

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University im. M. V. Lomonosov)

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Card 2/2

S/661/61/000/006/028/081
D205/D302

AUTHORS: Belyakova, Z. V., Yur'yev, Yu. K. and Yelyakov, G. B.

TITLE: Tetraacycloxysilanes in organic synthesis

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedeniy; 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, 127-134

TEXT: A direct proof of the structure of the silicoanhydrides of the dibasic acids was obtained. It was previously stated that these compounds have carboxylic groups in each of the acid remnants bonded to the silicon atom. The reaction of the silico-succinic anhydride with diethylamine

Card 1/5

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Tetraacycloxysilanes in ...

S/661/61/000/006/028/081
D205/D302

$$Si(OCOCH_2CH_2COOH)_4 + 4NH(C_2H_5)_2 \longrightarrow$$

$$\xrightarrow[-Si(OH)_4]{} 4HOOCCH_2CH_2CON(C_2H_5)_2$$

is a direct proof of this structure. In the reaction of excess $SiCl_4$ with organic monobasic acids (of the fatty, aromatic and heterocyclic series) acyloxy-trichlorosilanes are obtained in quantitative yields; $SiCl_4 + RCOOH \rightarrow RCOOSiCl_3 + HCL$ where R = CH_3, C_2H_5, n-C_3H_7, iso-C_3H_7, n-C_4H_9, iso-C_4H_9, tert.-C_4H_9, n-C_5H_{11}, n-C_7H_{15}, C_6H_5, o-,p-$CH_3C_6H_4$, o-,p-ClC_6H_4, o-,m-,p-$NO_2C_6H_4$, α-$C_{10}H_7$, ✓ α-C_4H_3S. The excess $SiCl_4$ is distilled off in vacuum. Acids insoluble in $SiCl_4$ give, under these conditions, only tetraacyloxysi-

Card 2/5

lanes, a solvent being indispensable for preparing acyloxy-trichlorosilanes. Acyloxy-trichlorosilanes are mobile, fuming, easily hydrolysable liquids. They disproportionate easily according to $4RCOOSiCl_3 \longrightarrow Si(OCOR)_4 + 3SiCl_4$. The stronger the organic acid, the higher is the tendency towards disproportionation. In the fatty acid series, this tendency decreases with the length and degree of branching of the organic chain. The known process of the preparation of chloroanhydrides from acids and $SiCl_4$ which supposedly takes place in two stages, is shown to take place in four stages with possible 3 alternative chemical routes. Two of the alternative routes were tested:

$$2RCOOSiCl_3 + Si(OCOR)_4 \longrightarrow 6RCOCl + 3SiO_2 \qquad (6)$$

$$RCOOSiCl_3 + 3(RCO)_2O \longrightarrow 3RCOCl + Si(OCOR)_4 \qquad (7)$$

✓

Thus, benzoyloxy trichlorosilane reacted with silicobenzoic and benzoic anhydrides yielding 64.5 and 74% of benzoyl chloride respectively. Acyloxy-trichlorosilanes are good acylation agents. The acyloxy group is more readily substituted for the alkoxy-group by the action of ethyl alcohol at -30°C than the chlorine atoms. The acyloxy trichlorosilanes may serve for the formation of new mixed anhydrides: $RCOOSiCl_3 + 3CH_3COOH \longrightarrow RCOOSi(OCOCH_3)_3$

$$R = \alpha - X_4H_3O \quad \text{or} \quad \alpha - C_4H_3S$$

By the action of Grignard reagents the acyloxy-trichlorosilanes form tetraalkyl silanes and tertiary alcohols. S. A. Golubtsov (Moscow), V. P. Davydova (IKhS AN SSSR, Leningrad), N. S. Leznov (Moscow), A. L. Klebanskiy (VNIISK, Leningrad), Yu. Ya. Fialkov (KPI, Kiyev), P. V. Davydov (Moscow), R. Kh. Freydlina (INEOS AN SSSR, Moscow) and M. G. Voronkov (IKhS AN SSSR, Leningrad) took part in the discussion. There are 2 tables.

Tetraacyloxysilanes in ...

S/661/61/000/006/028/081
D205/D302

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow State University im. M. V. Lomonosov)

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Card 5/5

YUR'YEV, Yuriy Konstantinovich; GOL'DENBERG, G.S., red.; YERMAKOV, M.S., ~~tekhn. red.~~

[Practical work in organic chemistry] Prakticheskie raboty po organicheskoi khimii. Moskva, Izd-vo Mosk. univ. Pt.3. 1961. 251 p. (MIRA 14:8)

(Chemistry, Organic—Synthesis)

YUR'YEV, Yuriy Konstantinovich; GOL'DENBERG, G.S., red.; LAZAREVA, L.V., tekhn. red.

[Laboratory work in organic chemistry] Prakticheskie raboty po organicheskoi khimii. 2., dop. izd. Moskva, Izd-vo Mosk. univ. No.1-2. 1961. 418 p. (MIRA 15:2)
(Chemistry, Organic—Laboratory manuals)

YUR'YEV, Yu.K.; PENTIN, Yu.A.; REVENKO, O.M.

Infrared absorption spectra of γ-alkylpentamethylene sulfides.
Neftekhimiia 1 no.2:163-168 Mr-Ap '61. (MIRA 15:2)

1. Moskovskiy gosudarstvennyy universitet.
(Thiopyran--Spectra)

YUR'YEV, Yu.K.; LEVINA, R.Ya.

N.D.Zelinskii and the scientific trends in his students' schools; on the one hundredth anniversary of his birth. Vest. Mosk. un. Ser. 2: Khim. 16 no.1:3-10 Ja-F '61. (MIRA 14:4)
(Zelinskii, Nikolai Dmitrievich, 1861-1953)

YUR'YEV, Yu.K.; ZEFIROV, N.S.; MINACHEVA, M.Kh.

Furan series. Part 8: Tetramethylfuran in the reaction of diene synthesis. Zhur.ob.khim. 30 no.10:3214-3217 O '61. (MIRA 14:4)

1. Moskovskiy gosudarstvennyy universitet.
(Furan)

NOVITSKIY, K.Yu.; YUR'YEV, Yu.K.; ZHINGAREVA, V.N.

Furna series. Part 9: Synthesis of 2,5-bis(aminomethyl) furans. (MIRA 14:4)
Zhur.ob.khim. 30 no.10:3218-3220 O '61.

1. Moskovskiy gosudarstvennyy universitet.
(Furan)