MAKIN, S. M.

"Esters of 4-Phenyl-4-Piperidols With Functional Bubstitutes in the Presence of Nitrogen and in Acyl Groups." Cana Chem Sci, Moscow Inst of Fine Chemical Technology imeni M. V. Lomonosov, 11 Oct 54. (VM, 29 Sep 54)

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MAKIN, S.M.

459

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AUTHORS:

Nazarov, I. N., and Makin, S. M.

TITLE:

Heterocyclic Compounds, Part 50. Synthetic Anesthetics. Chap. 13. Reaction of Primary Amines with beta, beta-dimethyldivinylketone and 1-methoxy-5 methyl-4-hexene-3-on. Esters of 1,2,2-trimethyl-4-phenyl-4-piperidol (Geterotsiklicheskiye soyedineniya. 50. Sinteticheskiye obexbolivayushchiye veshchestva. XIII. Deystviye pervichnykh aminov na beta, beta-dimetildivinilketon i 1 metoksi-5 metil-4-geksen-3-on. Slozhnyye efiry 1,2,2-trimetil-4-fenil-4-niperidola)

piperidola)

PERIODICAL:

Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 94-100 (U.S.S.R.)

ABSTRACT:

A study of the reaction between primary amines (methylamine, ethylamine, isopropylamine) and 1-methoxy-5-methyl-4-hexene-3-on taking place at room temperature showed only the addition of the amines according to the double bonds and the formation of unstable methoxyketoamines which during the distillation in vacuum decomposed almost entirely into initial components. The reaction of 1-methoxy-5-methyl-4-hexene-3-on with aqueous solutions of primary amines (methylamine, isopropylamine) at 60-65° for 4-6 hrs resulted in the formation of 1-21kyl-2,2-dimethyl-4-piperidones. The reaction of methylamine though smoother than that with isopropylamine, showed

Card 1/2

Heterocyclic Compounds, Part 50

459

considerable tar formation (resinification). Efforts to eliminate tar formation by reducing the reaction temperature or by decreasing the heating period produced no positive results because of the formation of almost inseparable mixtures of piperidones and isomeric amines. Cyclization into gamma-piperidones during the reaction of primary amines with beta-beta-dimethyldivinylketone is less smooth but with much smaller yields than in the case of other divinylketones having substitutes in both vinyl groups. The reaction of 1,2,2-trimethyl-4-piperidon with phenyl lithium yielded about 60% of 1,2,2-trimethyl-4-phenyl-4-piperidole the esters of which were obtained for pharmacological experiments.

ASSOCIATION:

Moscow Institute of Fine Chemical Technology im. M. V. Lomonosov (Moskovskiy Institut Tonkoy Khimicheskoy Tekhnologii im. M. V. Romonosova)

PRESENTED BY:

SUBMITTED:

November 9, 1955

AVAILABLE: Card 2/2

MAKIN, SM.

460

AUTHORS:

Nazarov, I. N.; Makin, S. M.; and Grapov, A. F.

TITLE:

Heterocyclic Compounds. Part 51. Synthetic Anesthetics. Chap. 14. Esters of 1,2,5-trimethyl-4-phenyl-4-piperidole with Functional Groups at Acyl radical. Analogues of promedol and Isopromedol. 1. (Geterotsiklicheskiye Soyedineniya 51. Sinteticheskiye obezbolivayushchiye veshchestva. XIV. Slozhniye efiry 1,2,5-crimetil-4fenil-4-piperidola s funktsional'nymi gruppami pri atsil'nom

ostatke. Analogi promedola i izopromedola. 1)

PERIODICAL:

Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 101-110 (U.S.S.R.)

ABSTRACT:

Studying the physiological activities of various esters, the authors synthesized numerous 1,2,5-trimethyl-4-phenyl-4-piperidole esters containing (in the acyl radical) alkoxy, phenoxy and alkylsulfide groups as well as halides, nitrogen and double bonds. It was found that the introduction of these groups into the acyl radical of acetic and propionic esters of 1,2,5-trimethyl-4-phenyl-4-piperidole sharply reduces or completely destroys the analgetic activity of these substances. Esterification of 1,2.5-trimethyl-4-phenyl-4piperidole with chlorides of analogous acids by the Spasov (4) method led to the formation of methoxyacetate and ethoxyacetate.

Card 1/2

Heterocyclic Compounds. Part 51.

460

· CONTROL CONTROL OF THE STREET

Esterification with chloride of beta-phenoxypropionic acid gave beta-phenoxypropionic ester. Results showed that, of the many esters synthesized, only acrylate and methacrylate showed a considerable anesthetic effect equal to 35-50% of the promedole effect, all other esters were practically void of any morphine-like activity. Propionates were found to have maximum analgetic activity of all the 4-phenyl-4-piperidole esters investigated. There are 14 references, of which 4 are Slavic.

ASSOCIATION:

Moscow Institute of Fine Chemical Technology im. M. V. Lomonosov (Moskovskiy Institut Tonkoy Khimicheskoy Tekhnologii im. M. V. Lomonosova)

PRESENTED BY:

SUBMITTED:

November 9, 1955

AVAILABLE:

Card 2/2

AUTHORS:

Nezarov, I. N. and Makin, S. M.

79-2-50/58

TITLE:

Heterocyclic Compounds. Part 52. Synthesis of 1-gamma-Alkoxypropyl-4-Piperidones and 1-gamma-Dialkylaminopropyl-4-Piperidones (Geterotsik-licheskiye soyedineniya. 52. Sintez 1-gamma-alkoksipropil-4-piperidonov i 1-gamma-dialkilaminopropil-4-piperidonov).

PERIODICAL:

Zhurnal Obshchey Khimii, 1957, vol. 27, No 2, pp. 499-509 (U.S.S.R.)

ABSTRACT:

Considering the great importance of gamma-piperidones for the synthesis of new physiologically active compounds possessing strong pain killing, anesthetic and spasmolytic properties, the authors investigated the reaction between vinylpropenyl ketones with gemma-dialkylaminopropylamines, gamma-alkoxypropylamines and gamma-ethylmercaptopropylamine which are easily obtainable from acrylonitrile. It was found that different vinylpropenyl ketones react with the above amines much easier than with ordinary alkyl amines, giving high yields of gamma-piperidones. Instead of vinylpropenyl ketones, it is quite possible to utilize homologous beta-methoxy ketones which form as a result of addition of methanol to the vinylpropenyl ketones. The authors synthesized more than twenty new gamma-

Card 1/2

Heterocyclic Compounds. Part 52.

79-2-50/58

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piperidones and 2-methyl-4-ketodecahydroquinolines of three different types.

Some of the gamma-piperidones derived were reduced with the aid of hydrazinehydrate by employing the N. M. Kizhner method (10), into homologous piperidine bases.

There are 11 references, of which 8 are Slavic

ASSOCIATION:

Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov

PRESENTED BY:

SUBMITTED:

November 9, 1955

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ATTHORS:

Nazarov, I. N. and Makin, S. M.

79-2-51/58

TITLE:

Heterocyclic Compounds. Part 53. Synthetic Anesthetics. Chapt. 15. Synthesis of 2,5-Dimethyl-4-Phenyl-4-Piperidoles and Their Esters Containing, in Presence of Nitrogen, Gamma-Substituted Propyl Radicals. Analogues of Promedol and Isopromedol II. (Geterotsiklicheskiye soyedineniya.

53. Sinteticheskiye obezbolivayushchiye veshchestva. XV. Sintez 2,5-dimetil-4-fenil-4-piperidolov i ikh slozhnykh efirov soderzhashchikh pri azote gamma-zameshchennyye propil'nyyeradikaly. Analogi promedola i izopromedola.

II.)

PERIODICAL:

Zhurnal Obshchey Khimii, 1957, vol 27, No 2, pp. 510-518 (U.S.S.R.)

ABSTRACT:

Some of the gamma-piperidones described in report 53 were used for the synthesis of 4-phenyl-4-piperidoles and their esters (acetates and propionates) which belong to the group of highly actibe anesthetics. Synthesized were numerous 1-propyl-2,5-dimethyl-4-phenyl-4-piperidoles and their esters having in the propyl radical at the nitrogen (in gamma position) alkoxy, alkylsufide and dialkylamine groups. The pripionates of 1-(gamma-methoxy)-and 1-(gamma-ethoxy)-propyl-2,5-dimethyl-4-phenyl-4-piperidoles were found

Card 1/2

Heterocyclic Compounds. Part 53. Synthetic Anesthetics. Chapt. 15. Synthesis of 2,5-Dimethyl-4-Phenyl-4-Piperidoles and Their Esters Containing, in Presence of Nitrogen, Gamma-Substituted Propyl Radicels. Analogues of Promedol and Isopromedol II.

to have a high anesthetic (analgetic) and locally-anesthetic characteristic. The analgetic activity of all the esters described was still much lower than the activity of promedol and Isopromedol. It was established that a methyl radical in presence of nitrogen, as compared with other known substitutes, has a much greater effect on the analgetic properties of 4-phenyl-4-piperidole esters.

There are 4 references, of which 2 are Slavic

ASSOCIATION: Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov PRESENTED BY:

Card 2/2

SUBMITTED: November 9, 1955

AVAILABLE: Library of Congress

20-114-3-28/60

AUTHORS:

Nazarov, I. N., Member of the AS USSR, Krasnaya, Zh. A.,

Makin, S. M.

TITLE:

Anionotropic Regrouping of 2,6-Dimethylocta-2,4,7-Triene-6-ol (Anionotropnaya peregruppirovka 2,6-dimetilokta-2,4,7-triyen-

6-ola)

PERIODICAL:

Doklady Akademii Nauk SSSR, 1957, Vol 114,Nr 3, pp 553-556(USSR)

ABSTRACT:

Polyene alcohols have an interesting characteristic, namely that they are easily subject to anionotropic regrouping under the influence of deluted acids, on which occasion hydroxyl is shifted and the number of conjugated double bonds is increased. The investigation of this regrouping is interesting from the point of view of the production of primary polyene alcohols, especially of those of an isoprenoidal type. The authors investigated the anionotropic regrouping of the substance mentioned in the title, which, as regards the structure of the carbon chain, is similar to linalcol Linalcool with an additional double bond. By the influence of lithium acetylide in a solution of liquid summonia of 2-methyl-hepta-2, 4-diene-6-on the

hitherto not described 2,6-dimethylocta-2,4-diehe-7-In-6-ol

Card 1/3

20-114-3-28/60

Anionotropic Regrouping of 2,6-Dimethylocta-2,4,7-Triene-6-01

was produced with a high output. By absorbing 1 mol of hydrogen in the presence of the Pd-catalyzer carbinol was changed into 2,6-dimethylocta-2,4,7-triene-6-ol. It is a liquid with the pleasant smell of flowers. There are two possibilities for the anionotropic regrouping of caroinol III: either by the formation of a primary alcohol (dehydrogeraniol IV) with the shifting of a double bond, or by the formation of a tertiary alcohol with a parallel shifting of 2 double bonds. The authors stated that only a tertiary alcohol V and no primary alcohol IV develop on the occasion of a 1.5-hour-long action of 0.05 % sulfuric acid on carbinol III. In order to prove this direction of reaction the obtained alcohol V was subjected on complete hydration. Then 2,6-dimethyloctane-2-ol(VI) developed. The same alcohol VI was obtained by the following reactions: 2,6-dimethylocta-2,4-diene-7-In-2-ol, for which an anionotropic regrouping is possible only in the direction of the formation of a tertiary alcohol, isomerizes under the influence of 1 % sulfuric acid into 2,6-dimethylocta-3,5-diene-7-In-2-ol VII. This again changes under partial hydration in the presence of Pd-catalyzers into V, and in the case of a complete hydration into VI. The saturated alcohols VI, obtained by either

Card 2/3

20-114 3-28/60

Anionotropic Regrouping of 2,6-Dimethylocta-2,4,7-Triene-6-ol

of the above methods, produce 3,5-dinitrobenzoates (molecular compounds with or naphthylamine), which melt at the same temperature (100 - 101°), which is unchanged by joint melting. The methylhoptadienone I, ne-

cessary for the synthesis of carbinol II, was obtained by two different ways: 1) By pyrolysis of the acetoacetate of dimethylethynylcarbinol and 2) by the condensation of dimethylacryl-aldehyde with acetone under the influence of isopropylate and of tertiary aluminum-amylate as well as of sodium hydrate (production 47.5%). The experimental part with constants, methods, and production of the substances Ascussed follows. There are 4 references, 1 of which is Soviet.

Moscow Institute for Fine Chemical Technology imeni M. V. ASSOCIATION:

Lomonosov (Moskovskiy institut tonkoy khimicheskoy tekhnologii

im. M. V. Lomonosova)

January 9, 1957 SUBMITTED:

Card 3/3

CIA-RDP86-00513R001031610005-4" APPROVED FOR RELEASE: 06/20/2000

AUTHORS:

Nazarov, I. N. Member, Academy of Sciences, 20-114-4-32/63 USSR, Gusev, B. P., Makin, S. M., Mochalin, V. B., Nazarova, I.I., Vinogradov, V. P., Kruptsov, B. K., Shavrygina, O. A., Nazarova, D. V.

CONTRACTOR OF THE PROPERTY OF

TITLE:

The Condensation of Acetylene With Methylheptanone and Its Analogues (Kondensatsiya atsetilena s metilgeptanonom i yego analogami) The Synthesis of Linalool and Its Analogues (Sintez linaloola i yego analogov)

PERIODICAL:

Doklady Akademii Nauk SSSR, 1957, Vol. 114, Nr 4, pp. 796-799

ABSTRACT:

Several years ago a simple method of synthesis of different acetylene alcohols was worked out in the laboratory of the authors by means of condensation of aldehydes and ketones under the influence of powdery caustic potash with acetylene at high pressure (5-10 at superpressure). It was of interest to employ this method in the condensation of acetylene with methylheptanone and similar ketones, in order to obtain the corresponding acetylenealcohols. Linalcol and some analogues may then be obtained easily by partial hydrogenation with a Pd-catalyst. Hitherto such condensations have usually been carried out under the influence of metallic sodium in a solution of liquid ammonia.

Card 1/4

The Condensation of Acetylene With Methylheptanone and Its 20-114-4-32/63 Analogues. The Synthesis of Linalcol and Its Analogues

It was found that methylheptanone and its various analogues may be condensed very easily with caustic potash and acetylene at the above-mentioned pressure. At o-2000 they give as a result the corresponding tertiary acetylene alcohols with an almost quantitative yield (more than 90%). This reaction may also be carried out without acetylene pressure, however, somewhat more slowly and with a yield of only 60-80%. It has been previously shown in the same laboratory that acetylene alcohols which contain a non-substituted acetylenehydrogen may be hydrated highly selectively in the presence of palladium over calcium carbonate or copper coated zinc powder. Thereby vinylalcohols with an almost theoretical yield are obtained. The acetylene alcohols may not be selectively hydrated with other catalysts (Ni, Pt) and are therefore useless in the production of pure vinyl alcohols. An analogous picture may also be noticed with the hydrogenation of the above-described acetylene alcohols which are obtained by condensation of acetylene with methylheptanone and its analogues. These acetylene alcohols may also be highly selectively hydrated in the presence of a Pd-catalyst. They form linalcol and its analogues

Card 2/4

The Condensation of Acetylene With Methylheptanone and Its 2q-114-4-32/63 Analogues. The Synthesis of Linalool and Its Analogues

with an almost theoretical yield. The purity control of the winylalcohols (linalcol and its analogues) was carried out by means of the acetylene test (with ammonia solution of silver or copper oxide), whose sensitiveness was determined by special tests and amounted to 0,2-0,3%. At the hydrogenation of the acetylene alcohols with a Pd-catalyst the acetylene test always disappears at the theoretical point, that is, as only one hydrogen molecule is strongly attached. The acetylene alcohols obtained in the course of this work are summarized in table 1. Linalcol and its analogues (table 2) were obtained by a partial hydrogenation of the above-mentioned acetylene alcohols with Pd-catelysts. In the experimental part the methods and yields of the said substances are described in detail. There are 2 tables and 5 references, 3 of which are Soviet.

ASSOCIATION:

Institute for Organic Chemistry imeni N.D. Zelinskiy of the AN USSR and Moscow Institute for Refined Chemical Technology imeni M.V. Lomonosov (Institut organicheskoy khimii im. N.D. Zelinskogo Akademii nauk SSSR i Moskovskiy institut tonkoy khimiches-

Card 3/4

CIA-RDP86-00513R001031610005-4"

APPROVED FOR RELEASE: 06/20/2000

The Condensation of Acetylene With Methylheptanone and Its 2. 114-4-32/63 Analogues. The Synthesis of Linalcol and Its Analogues

koy tekhnologii im. M.V. Lomonosova)

SUBMITTED: March 12, 1957

Card 4/4

MAKIN, S.M.

20-5-31/60

MOHTUA

HAZAROV, I.N., member of the Academy,

YAHOVSKAYA, L.A., GUSEV, B.P., MAKIN, S.M. and

NAZAROVA, I.I.

TITLE

The Synthesis of Geranylacetone, 3-Methylgerenylacetone,

Pseudoionon and Pseudoiron.

(Sintez geranilatsetona, 3-metilgerarilatsetona,

psericionona i psevdoirona.- kuasian)

PERIODICAL

Doklady Akademy Nauk SSSR 1957, Vol 114 Hr 5,

pp 1029-1032 (U.S.S.R.)

ABSTRACT

In the course of systematic work on the synthesis of isoprenoidcompounds a paper by Carrol attracted the attention of the author. That author reports that with the heating of linalool with acetoacetic ether, in the presence of a small amount of sodium alcoholate, geranylacetone with a 40-45 % yield develops. Kimel and Cope obtained 61 % linalylacetoacetate from interaction of linalool with diketone, in the presence of metallic sodium. Its pyrolysis at 170-235 % yielded 78 % geranylacetone. The authors thoroughly examined both synthesis varieties on linalool and 3-methyllinalool, in order to work out a production method for geranylacetone and 3-methylgeranylacetone. It was found out that reaction proceeds best at 160-180°C without any catalysts.

CARD 1/4

20-5-31/60

The Synthesis of Geranylacetone, 5-Methylgeranylacetone, Pseudelonon and Pseudoiron.

90-95% of the theoretical CO2-amount precipitate, and an almost theoretical amount of alcohol (with admixture of acetono) is distilled. Since the latter process is terminated considerably earlier than the CO2-elimination, it may be assumed that the reaction passes the acetoacetete-stage. A system is given. This mechanism was proved by the authors for the reaction between dimethylvinylcarbinol and acetoacetic ether. From a study of the second variety of synthesis the authors found that the acylation of linelocl and methylinalocl with diketone may best be performed in the presence of pyridine or triethylamine, and not of metallic sodium. Based upon the results obtained, the authors decided to investigate both varieties in dehydrolinalcol and 3-methyl dehydrolinalool, in order to work out synthesis methods for pseudopionon and pseudoiron. Sometime during the beginning of this work a paper by Lacey was published demonstrating the possibility of a synthesis of dienones on most simple ethinylcarbinols according to a general system mentioned here. The second half of the reaction the pyrolysis of acetoacetate, was performed by Lacey in the presence of small amounts of p-toluclsulfonic acid. This kind of dienous synthesis was thoroughly

CARD 2/4

20-5-31/60

The Synthesis of Geranylacetone, 3-Methylgeranylacetone, Pseudoionon and Pseudoiron.

studied in the laboratory of the authors. During the work the paper by Naves of the same topic was published. He also described a production method for 3-methylpseudoiron from 3-methyldehydrolinalool and a-methyl acetoacetic ether. Recently Preobrazhenskiy and collaborators wrote on this topic. The authors studied both synthesis varieties and found that the reaction between dehydrolinalcol or 3-methyl dehydrolinalool and acetoacetic ether proceeds best at 180-180°C. The yield of pseudoionon and pseudoiron amounts to about 55 %. The acylation of dehydrolinalcol or 3-methyldehydrolinalcol by diketone proceeds best in the presence of pyridine or triethylamine. The pyrolysis of the two mentioned substances thus obtained was performed at 185-195° C. In this connection about 80 % of the theoretical CC,-amount.is obtained. The yield of pseudoionon and pseudoiron amounted to 50-55 %, of the initial materials. The cyclization of the thus obtained pseudionon with a mixture of sulfur- and ice-acetic acid gave ionon

CARD 3/4

20-5-31/60

The Synthesis of Geranylacetone, 3-Nethylgeranylacetone, Pseudoionon and Pseudoiron.

with a 65 % yield. Experimental part with the usual data. (2 Slavic references)

ASSOCIATION: "N.D. ZELZINSKY" Institute for organic chemistry of the

Academy of Sciences of the U-SSR.

(Institut organicheskoy khimii im.N.D. Zelinskogo Akademii

nauk SSSR)

PRESENTED BY: -

SUBMITTED: 7.2.57

AVAILABLE: Library of Congress.

CARD 4/4

AUTHORS:

Nazarov, I. N., Academician, Makin, S. M., 20-114-6-29/54

Mochalin, V. B., Nazarova, D. V., Vinogradov, V. P.,

Kruptsov, B. K., Nazarova I. I. and Shavrygina, O. A.

TITLE:

The Synthesis of Methylheptenone and Methylheptadienone

Analogues (Sintez analogov metilgeptenona i metilgeptadiyenona)

PERIODICAL:

Doklady AN SSSR, 1957, Vol. 114, Nr 6, pp. 1242-1245 (USSR)

ABSTRACT:

This synthesis is of interest for the production of a number of corresponding analogues of natural isoprenoid compounds. The initial acetylene-alcohols for this purpose were produced according to the authors' method (reference 1).

By a selective hydrogenation in the presence of palladium on calcium-carbonate acetylene alcohols are almost quantitatively converted to analogous vinyl alcohols (reference 2). These latter yield the corresponding analogues of methylheptenone in three different ways (reference 3). Method A. By the influence of gaseous

hydrogen chloride or hydrogen bromide upon tertiary vinyl alcohols at 0 - 20°C primary haloid-derivatives of an allyl-

Card 1/3

-type easily form (reference 4). Their condensation with sodium-acetate-acetic-ether with a subsequent saponification

The Synthesis of Methylheptenone and Methylheptadienone Analogues

6 o. which are Slavic.

20-114-6-29/54

leads to methylheptenone analogues. Method B. At 140 -190°C tertiary vinyl alcohols directly react with the same ether. An almost theoretical quantity of ethanol and CO2 is separated and the same analogues as in A) are obtained. Method V. By the action of diketene upon tertiary vinyl alcohols in the presence of small amounts of triethylamine or piperidine, acetonacetic ethers of these alcohols are obtained (table 2). Their pyrolysis also leads to the above--mentioned analogues (reference 6). The 2,3-dimethy1-2--heptene-6-on (IV) necessary for the synthesis of irone was produced all three ways mentioned. Dimethylisopropenyl-carbinol (initial substance) was produced by the influence of methyl-lithium upon methyl-metacrylate. All methylheptenone analogues produced are comprised in table 1. The authors further produced: allyl- (I) (reference 9), crotyl-(II) and chlorocrotyl-acetone (III) (reference 8), dimethylisopropenyl-carbinol-acetoacetate, dimethylheptenone (IV), cyclohexylidenpentanone (IX) and tertiary butylheptadienone (XIII). The production methods and constants of these substances are given. There are 2 tables and 12 references,

card 2/3

The Synthesis of Methylheptenone and Methylheptadienone 20-114-6-29/54 Analogues

ASSOCIATION: Institute of Organic Chemistry AN USSR imeni N. D. Zelinskiy

AS USSR and Institute for Fine-Chemical Technology imeni M. V. Lomonoscv, Moscow (Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR i Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonoscova)

SUBMITTED: March 12, 1957

Card 3/3

AUTHORS: Nazarov, I. N., Member of the Academy, 20-117-5-27/54

(Deceased), Makin, S. M., and Kruptsov, B. K.

TITLE: The Synthesis of Ethoxyisoprene (Sintez etoksiizoprena).

PERIODICAL: Doklady AN SSSR, 1957, Vol. 117, Nr 5, pp. 823-825 (USSR)

ABSTRACT: The alkoxydienes are interesting for the organic chemistry by the presence of 2 double conjugated formations since they can

be used furthermore for the synthesis of various substances in consequence of a alkoxyl group capable of reaction. Especially intersting is the ethoxy-isoprene (1-ethoxy-3-methyl-butadiene-1,3) since it can be used for the synthesis of important isoprenoids (zitral, vitamin A, carrotine and others). It has a methyl-branching in the third position and has therefore an isoprenoid structure. Reactions (I) - (IV) are given by means of which the authors have obtained the synthesis mentioned in the title. By action of tetraethoxysilane on acetone the acetone-diethyl-ketal (II) was obtained. The syntheses according to former references

(reference 1) led to only small yields (10-15%). The authors obtained yields of 92% of acetone-ketal (in presence of

0,5 mol ethanol pro 1 mol tetra-ethoxysilane) by the

Card 1/3 application of phosphorous acid of 85 % as catalyst. Acetone-

The Synthesis of Ethoxyisoprene

20-117-5-27/54

diethylketal was then introduced into the condensation reaction with vinyl-ethyl-ether. The addition of ketals to these ethers was hitherto scarcely researched. The authors succeeded in carrying out this reaction at 0° and in presence of catalytic quantities of tri-fluorboron-ethyrate with a yield of 85% of 3-methyl-1, 1,3 triethoxybutane, with reference to the acetone-diethylketal which entered into the reaction. In order to prevent a further condensation a ketal excess has to be used which is higher by 1,5 times. In the case of passage of vapors of the 1,1,3-triethoxy-3-methylbutane by MgHPO,-catalyst at 350° in a 15-20 mm-vacuum 2 alcohol molecules are splitted off and ethoxyisoprene is formed with a yield of 77%. Furthermore this latter substance was obtained with a good yield in the passage of vapors of the β -methylcroton-aldehyde-diethylacetal over the contact catalyst NaH, PO,. The initial substance in question was produced from iso-valerian-aldehyde (reference 6). In the experimental part the usual data are given with the preparation of the catalyst.

Card 2/3

CIA-RDP86-00513R001031610005-4 "APPROVED FOR RELEASE: 06/20/2000

AUTHORS:

Nazarov, I. N. (Deceased), Makin, S. M., SOV/79-29-1-25/74

Kruptsov, B. K.,

Mironov, V. A.

TITLE:

Synthesis of Acetals and Ketals by Means of Tetraalkoxy-

Silanes (Sintez atsetaley i ketaley s pomoshch'yu tetraalkoksi-

silanov)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 1, pp 106-111 (USSR)

ABSTRACT:

It is known that the most suitable acetylating agents are the esters of the ortho-formic acid. Owing to their high costs they can however not be used as initial substance for the synthesis of acetals. As a substitute for the above esters the authors chose the easily available esters of the ortho--silicic acid (tetraalkoxy-silanes). Helferich and Hansen (Ref 1) found that the tetraalkoxy-silenes are able to acetylate aldehydes and ketones in alcoholic medium in the presence of hydrogen chloride. The authors met however with many difficulties at the attempt to obtain some acetals and ketals according to this method and the yields were also small. For this reason, they investigated thoroughly the reaction of the tetraelkoxy-silanes with aldehydes and ketones under different conditions. On the reaction of acetone with

Card 1/3

Synthesis of Acetals and Ketals by Means of Tetraalkoxy-Silanes

sov/79-29-1-25/74

tetraalkoxy-silane they used HCl, concentrated H_2SO_A ,

p-toluenesulfonic acid, phosphoric acid, etc. as catalysts for the acetylation. Phosphoric acid proved to be the most suitable catalyst. In contrast with the general opinion, tetraalkoxy--silanes react well with aldehydes and ketones in the presence of some alcohol. Thus the diethyl ketal of acetone with tetraethoxy-silane was obtained in 93 % yield, on addition of 0.5 % alcohol only. Many other acetals of various aldehydes, ketones, etc. were synthesized also with good yields and very limited use of alcohol. The small alcohol quantity is important in the synthesis of low-boiling acetals and ketals: It is thus possible to obtain in the distillation the acetal and ketal with minute alcohol quantities which can easily be removed by water, which is rather difficult at higher quantities of alcohol. In the synthesis of high-boiling acetals the amount of the easily separable alcohol is of no importance. In the acetylation of the croton aldehyde the alcohol quantity is of particular importance: At 0.1-0.2 mol alcohol with 1 mol aldehyde dimethyl and diethyl acetal were resulting in a yield

Card 2/3

Synthesis of Acetals and Ketals by Means of Tetraalkoxy-Silanes

SOV/79-29-1-25/74

up to 86 %. On addition of more than 1 mol alcohol 1,1,3-trialkoxy-butanes are formed as main products (Ref 2). Without any alcohol the reaction yields 47 % only. For the removal of the acetals and ketals two methods were applied according to whether they are lower or higher boiling than the tetraalkoxy-silanes used in the reaction (see experimental part). The diethyl acetals of the croton aldehyde and methyl heptenone were obtained in good yield by the esters of ortho-formic acid as well. Both tables present all acetals and ketals synthesized. There are 2 tables and 3 references, 1 of which is Soviet.

ASSOCIATION:

Moskovskiy institut tonkoy khimicheskoy tekhnologii (Moscow

Institute of Fine Chemical Technology)

SUBMITTED:

July 19, 1957

Card 3/3

SOV/79-29-1-26/74 Nazarov, I. N. (Decensed), AUTHORS:

Makin, S. M., Kruptsov, B. K., Mironov, V. A.

Synthesis of Vinyl and Diene Ethers (Sintez prostykh vinilo-TITLE:

vykh i diyenovykh efirov)

Zhurnal obshchey khimii, 1959, Vol 29, Nr 1, pp 111-117 (USSR) PERIODICAL:

In the passage of the vapors of dimethyl and diethyl acetals ABSTRACT: of the acetone of acetic acid, propione, butyric acid, isobutyric acid, as well as of the dimethyl and diethyl ketals

of acetone and cyclohexanone over NaH2PO4 and MgHPO4 (as cata-

lysts) at 300-3750 the authors obtained the substituted vinyl ethers listed in table 1. The simple diene ethers are considerably interesting in organic chemistry since they possess two conjugated double bonds and a reactive alkoxy group. Furthermore, methoxy and ethoxy isoprenes were synthesized and the methoxy and ethoxy butadienes previously described (Refs 7,8,9)

were investigated. The alkoxy dienes specified were obtained by catalytic cleavage of the acetals of croton and $oldsymbol{eta}$ -methyl--croton aldehyde, as well as of the 1,1,3-trialkoxy-butanes and

1,1,3-trialkoxy-3-methyl butanes. The dimethyl and diethyl Card 1/3

Synthesis of Vinyl and Diene Ethers

SOV/79-29-1-26/74

scetals of β -methyl-croton aldehyde were synthesized according to scheme 1 in the presence of small quantity of ${\rm BF_3.0(C_2H_5)_2}$, the butanes mentioned according to reference 7 and scheme 2 (Refs 11,14,15,16). The catalytic cleavage of the acetals of croton and /3 -methyl-croton aldehyde, as well as of the trialkoxy-butanes into the simple diene ethers was thus carried out on the catalysts NaH2PO4 and MgHPO4. Active charcoal, silica gel, and glass were used as carriers. This process proceeded in vacuum 10-20 mm in the nitrogen current (Scheme 3). The maximum yield of ethoxy-isoprene was attained on the catalysts MgHPO 4 on active charcoal and MgHPO 4. NaSiO 3 (Table 2). As to durability, ${
m MgHPO}_4$ on chargoal proved to be the best catalyst. The activity of the catalysts produced from NaH_2PO_A decreases more rapidly. On the catalytic cleavage of the 3-methyl-1,3-dimethoxy-1-ethoxy-butane a mixture from methoxy- and ethoxy-isoprene resulted in about the same quentity. There are 2 tables and 16 references, 8 of which are Soviet.

Card 2/3

Synthesis of Vinyl and Diene Ethers

SOV/79-29-1-26/74

ASSOCIATION:

Moskovskiy institut tonkoy khimicheskoy tekhnologii (Moscow Institute of Fine Chemical Technology)

SUBMITTED:

September 10, 1957

Card 3/3

5 (3) AUTHORS:

SOV/79-29-3-4/61 Nazarov, I. H. (Deceased), Makin, S. M., Mochalin, V. B., Shavrygina , O. A., Nazarova, D. V., Kruptsov, B. K.

TITLE:

Synthesis of Analogues of Geranyl Acetone and Pseudoionone

(Sintez analogov geranilatsetona i psevdoionona)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 3, pp 744-753 (USSR)

ABSTRACT:

These analogues are initial products for the synthesis of the corresponding analogues of the most important natural isoprenoid compounds, of vitamin A, carotene, farnesol, as well as of phytol, a component of vitamins K and E. Recently, the authors reported on three syntheses of ketones of the isoprenoid type carried out by them: 1) By reaction of sodium acetoacetic ester with halogen derivatives of the allyl type (method A). 2) By reaction of vinyl- and ethynyl carbinols with acetoacetic ester (method B). 3) By pyrolysis of the acetoacetates of vinyl- and ethynyl carbinols (method V). This method was used to obtain methyl heptenone, methyl heptadienone and their analogues (Refs 1, 2). By condensation of methyl heptenone and its analogues with acetylene under pressure (5-10 atmospheres excess pressure), dehydrolinalool and its analogues resulted almost quantitatively. These com-

Card 1/3

SOV/79-29-3-4/61

Synthesis of Analogues of Geranyl Acetone and Pseudoionone

pounds were transformed by partial hydrogenation over a Pd-catalyst into linalool and its analogues (Ref 3). The three methods used for the synthesis of the ketones of the isoprenoid type were also employed for the synthesis of various analogues of geranyl acetone (Scheme 1). The synthesized analogues of geranyl acetone are shown in table 2. The synthesis of the pseudoionone analogues was carried out according to the methods B and V. In heating the analogues of dehydrolinalcol with acetoacetic ester the analogues of pseudoionone were formed (Table 3) (Scheme 2), in yields of 50-70%. The pyrolysis of acetoacetates of the dehydrolinalools preponderantly leads to one of the pseudoionone isomers; the other is obtained but in small quantities, which is not the case with the pseudoionone analogues (XVIII) and (XX), where two stereoisomeric forms (Table 3) were separated in form of their hydrazones. The compounds synthesized are characterized by absorption spectra in the ultraviolet range. There are 3 tables and 7 references, 5 of which are Soviet.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii (Moscow Institute of Fine Chemical Technology)

Card 2/3

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sov/79-29-3-4/61	
Synthesis of Analogues of Geranyl Acetone and Pseudoionone	
Synthesis of Analogues of Columbia modern and a series	
SUBMITTED: January 23, 1958	
SUBMITTED. Danuary 27, 1770	
Card 3/3	
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5 (3) SCY/79-29-1-29/77 Makin, S. M., Mochalin, V. B., AUTHORS: Shavrygina, O. A., Nazarova, D. V., Nazarov, I. . (Deceased) Synthesis of the Analogs of Merolidol, Farnesil Acetone and Geranyl Linaicol (Sintez analogov nerolidola, farnezilatsetona TITLE: i geranillinaloola) Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, PERIODICAL: pp 1176-1182 (USSR) In the present paper the authors investigated thoroughly the synthesis of isoprenoid alcohols and -ketones (of the ABSTRACT: analogs of the above-mentioned products) and of the intermediate products according to the given scheme. The condensation of the analogs of geranyl acetone (a) with acetylene was carried out in the steel reactor at 0-200 and at 3-10 atmospheres excess pressure in the presence of powdery caustic potas. The yield of tertiary acetylene alcohols (b) was 80-85 %. The condensations hitherto used (Refs 5, 6) are very complicated. All analogs of dehydronerolidol (b) synthesized in this investigation are presented in table 1. According to previous experiments (Ref 7) it was possible to carry out the hydrogenation of the Card 1/3

Synthesis of the Analogs of Merolidol, Fernesil Acetone and Geranyl Linelpöl

507/79-19-1-31/77

acetylene elcohols obtained, the analogs of ashpironarolilal (b), in the presence of the Pa/Caff -cetalyes. The analogs of nerolidel (v) obtained by partial hylmagenati n of acctylene alcohols (b) with this catalyst are given in table 2. According to the reaction with catalysts described in reference 8 the authors were able earlier to carry out the reaction of tertiary vinyl alcohols with acetoacetic ester at 150-190° also without catalysts, and obtained in this way methyl heptene, geranyl acetone, and their analogs (Rofs 1, 2). In the present study they applied this method to the synthesis of the analogs of farnesil acetone (g). When heating the nerolidol analogs described above (Table 2) with acetoacetic ester at 180-200° the analogs of farnesil acetone (g) were obtained (Table 3). The analogs of farnesil acetone (g) synthesized were then condensed with acetylene. The resulting tertiary acetylene alcohols (d) were converted by partial hydrogenation on Pd/CaCO, into the analogs of gerenyl linalool

Card 2/3

(e) (Tables 4 and 5). The compounds obtained could be used in the synthesis of the corresponding analogs, the phytol, a

Synthesis of the Analogs of Nerolidol, Farnesil 30V/79-29-4-29/77

Acetone and Geranyl Linalool

vitamins K and E. There 5 tables and constituent of

8 references, 6 of which are Soviet.

Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni ASSOCIATION:

Lomonosova (Moscow Institute of Fine Chemical Technology

imeni Lomonosov)

SUBMITTED:

March 28, 1958

Card 3/3

5.3400

77868

SOV/79-30-2-19/78

AUTHORS:

Nazarov, I. N., Makin, S. M., Shavrygina, O. A.,

Smirnyagin, V. A.

TITLE:

Synthesis of Higher Fatty Acids and Alcohols From

Tertiary Vinylcarbinols

PERIODICAL:

Zhurnal obshchey khimii, 1960, Vol 30, Nr 2, pp

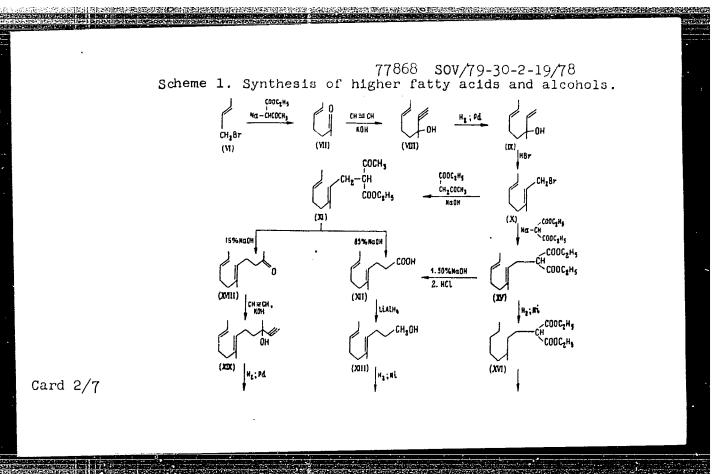
443-450 (USSR)

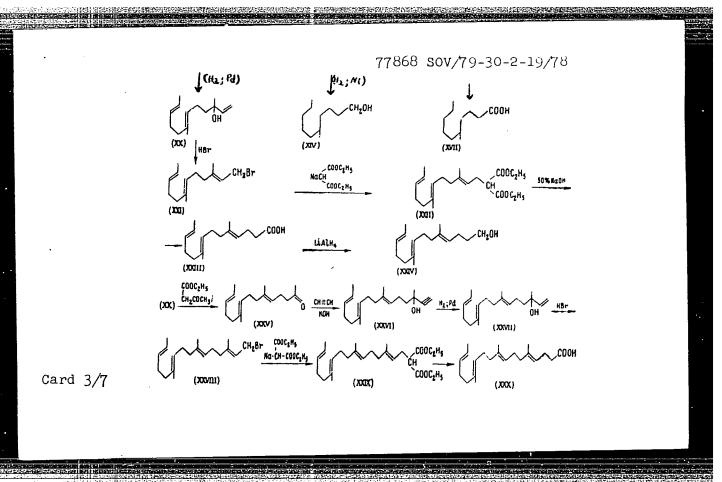
ABSTRACT:

The higher unsaturated acids were synthesized using acetoacetic and malonic esters. Diesters (III, R - $\text{CH}_2\text{R}^1\text{-OC}_2\text{H}_{\text{E}}$), (XV), (XXII) and (XXIX) were obtained by reacting sodium derivatives of malonic or acetoacetic esters with the respective bromides (see schemes 1 and 2 for designations

and for synthetic routes).

Card 1/7





Synthesis of Higher Fatty Acids and Alcohols From Tertiary Vinylcarbinols

77868 sov/79-30-2-19/78

Scheme 2. Synthesis of geranylacetic acid and of its analogues.

Card 4/7

The diesters were converted by successive saponification and decarboxylation into (2, 6-dimethyl-2,6-decadien--l0-oic (geranylacetic) acid (IV, R-CH $_3$), 6-methyl-

Synthesis of Higher Fatty Acids and Alcoh Is 77868
From Tertiary Vinylcarbinols 50V/79-30-2-19/70

-2,6-decadien-10-oic acid (XII), 6,10-dimethyl-2,6,16-tetradecatrien-14-oic acid (XXIII), and 6, 10, 14-tetradecatrien-14-oic acid (XXIII), and 6, 10, 14-trimethyl-2,6-10, 14-octadecatetrien-18-oic acid (XXX). The table below lists the constants of the synthesized acids with the constants of their S-benzylpseudothluronium salts. The higher alcohols were obtained by either reducing the respective acids with lithium aluminum hydride (2,6-dimethyl-2,6-decadien-10-oi (V), bp 72-74 (0.05 mm), 120 (0.05 mm), 120 (XIII), bp 120 (0.035 mm), 120 (1.4711, 120 (XIII), bp 120 (0.035 mm), 120 (1.4711, 120 (1.4711, 120 (1.4711), 120 (1.4

Card 5/ 7

SOV/79-30-2-19/78

77868

Synthesis of Higher Fatty Acids and Alcohols From Tertiary Vinylcarbinols

Table 3. Constants of higher fatty acids. Key to Table 3: (1) Acid; (2) boiling point (pressure in mm); (3) found; (4) calculated; (5) formula; (6) yield (in %); (7) S-benzylpseudothiuronium salt; (8) boiling point.

	:		MA,					13) (40) 1		(4)	(4) (h)		171					
(1)		(2)		7 D	d 40	(3)	(41	(5)	С	Н	د	Н	(6)	(8)	<i>(5)</i>	/3. N (%)	N	!
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(, - , ,,-		124-127				i —	_	C ₁₂ H _{2n} O ₃	-	-	-	-	.69	113-1149	$C_{20}H_{20}U_{1}N_{2}S$		7.72	
CMb		125-127				53.77	53.57	$C_{11}H_{14}O_{\tau}$	72.53,		72.52	9,89	58	114-115	UnaHasOaNeS	7.74 8.19	8.04	
(XXIII)		150 152		1.4842	0.932	76.61	76.29	$C_{16}H_{26}O_{2}$	72.85		76.84	10.16	į.		_	7.89	_	
(XXX)	,	154-162 (0.02)		1.4890	0,926	98,77	98.85	$C_{24}B_{24}O_2$		10,56 11,03 11,07	713.17	10.74	10	89-90	C29H44O3N2S	5.55.	3.77	
(IV R C		138 - 138		ŀ	i	1		.,		. 11,01	-	j -	11	117 118	Cat HagOz NaS	7.86	7.55	
(D) the O	CL	113141	(2)	1.4870	1.057	SH.96	58.17	$C_{11}H_{17}O_{3}CI$] -	-	38	116117	Callerorthis		7.41	į
				No	tes	to	Tat	10·*Th	e v	161	d i	s h	hase	On th	io tont !	7.10		

olerinic alcohol.

Card 6/7

Synthesis of Higher Fatty Acids and Alcohols From Tertiary Vinylcarbinols

77868 **SOV**/79**-**30-2**-**19/78

obtained from the previously hydrogenated diesters (e.g., 6-methyl-lo-decanoic acid (XVII), bp $128-129^{\circ}$ (3 mm), n_D^{20} 1.4548, d_4^{20} 0.9209). There are 1 table; and 9 references, 7 Soviet, 1 German, 1 U.K. The U.K. reference is: M. O. Forster, D. Cardwell, J. Chem. Soc., 103, 1346 (1913).

ASSOCIATION:

Moscow Institute of Fine Chemical Technology (Moskovskiy

institut tonkoy khimichskoy tekhnologii)

SUBMITTED:

⊕

February 4, 1959

Card 7/7

s/079/60/030/05/15/074 B005/B126

AUTHORS :

Makin, S. M., Mochalin, V. B., Nazarova, D. V.

TITLE:

Ring Closure of Analogs of Pseudoionone and Citral

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 5, pp. 1471-1476

TEXT: The authors examined the ring closure of five previously (Ref. 11) synthesized analogs of pseudoionone with different gem-substituents in position 1. The reaction scheme of this ring closure is given. The ring closure was carried out with the help of two standard methods: 1) Ring closure under the effect of boron trifluoride in a benzene solution at -5°. With this method the relevant α -ionone is produced. 2) Ring closure by the effect of a mixture of concentrated sulfuric acid and acetic acid at $10-15^{\circ}$. A mixture of α - and β -ionone is produced by this method. The products were identified by the analysis of their ultraviolet absorption spectra. Table 1 shows the results obtained by ring closure by method 1). The analogs of pseudoionone with the following gem.-substituents R in position 1 were examined: R = H; C2H5; iso-C3H7; tert.-C4H9; Cl. The

Card 1/3

CIA-RDP86-00513R001031610005-4" **APPROVED FOR RELEASE: 06/20/2000**

Ring Closure of Analogs of Pseudoionone and Citral

S/079/60/030/05/15/074 B005/B126

above table shows boiling range, refractive index, λ_{\max} , ϵ_{\max} , yield and results of the C, H-determination for each of the products obtained by ring closure. 2,4-dinitrophenylhydrazones were produced from the resulting analogs of α -ionone. The table also gives melting point, λ_{max} , and nitrogen content of these derivatives. Table 2 gives the results obtained by ring closure by method 2). Both analogs of pseudoionone with the substituents R = H and R = Cl gave no ring closure by either method, since the activating energy necessary to form the carbonium ion, which is an important intermediate, is in both cases too high. The authors also examined ring closure of some analogs of citral, which were previously (Ref. 13) synthesized. In this case ring closure was brought about by the effect of sulfuric acid on the Schiff's base of the citral analog (Ref. 14). Mixtures of α - and β -cyclocitral were thus formed; the reaction scheme is given. The analog with R = H gave no ring closure here either, while the Schiff's base of the compound with R = Cl split off under the effect of sulfuric acid HCl, and changed into a cyclic product, whose structure was not determined. Table 3 gives the results obtained from the ring closure of the citral analogs. All the ring closures

Card 2/3

Ring Closure of Analogs of Pseudoionone and Citral

S/079/60/030/05/15/074 B005/B126

carried out are fully described in the experimental part. There are 3 tables and 16 references: 4 Soviet, 6 English, 1 German, 4 Swiss, and 1 Czechoslovakian.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii (Moscow Institute for Fine Chemical Technology)

SUBMITTED: May 21, 1959

Card 3/3

84876

158104

S/079/60/030/010/015/030 B001/B066

AUTHORS:

Makin, S. M. and Kruptsov, B. K.

TITLE:

Chemistry of Unsaturated Ethers. V. Acetals of Vinyl Acetaldehyde: A New Method of Synthesizing 1-Alkoxy-dienes-1,3

arenes

PERIODICAL:

Zhurnal obshchey khimii, 1960, Vol. 30, No. 10,

pp. 3276 - 3280

TEXT: The authors of the present paper investigated the autocondensation of vinyl ethyl, vinyl butyl, and vinyl isoamyl ethers in the presence of $\overline{\mathrm{BF}_3} \cdot \mathrm{O(C_2H_5)_2}$, in the complex with HgO, Hg(OCOCH₃)₂, HgSO₄, HgCl₂, of FeCl₃ in the complex with HgO and Hg(OCOCH₃)₂, and of ZnCl₂ in the complex with HgO. Acetone, dimethyl formamide, diethyl ether, nitromethane, and acetophenone were used as solvents. The autocondensation was most efficient in acetone or diethyl ether when using $\overline{\mathrm{BF}_3} \cdot \mathrm{O(C_2H_5)_2}$ in the complex with HgO or Hg(OCOCH₃)₂. The acetal of vinyl acetaldehyde

Card 1/3

84876

Chemistry of Unsaturated Ethers. V. Acetals S/079/60/030/010/016/030 of Vinyl Acetaldehyde. A New Method of B001/B066 Synthesizing 1-Alkoxy-dienes-1,3

and a small amount of condensation products of this acetal with vinyl alkyl ethers was thus obtained. In the absence of $BF_{3} \cdot O(C_{2}H_{5})_{2}$, HgO and mercury acetate are ineffective. On the other hand, vinyl alkyl ethers are known to polymerize readily in the presence of $\mathrm{BF_3} \cdot \mathrm{O(C_2H_5)_2}$, so that the autocondensation was assumed to take place in two stages, first under the formation of an acetal acylal (III) (Ref.4) which is added in the second stage to the other molecule of the vinyl alkyl ether by means of the catalyst (Refs.5-7) (Scheme 2). The autocondensation of vinyl alkyl ethers by means of $HgO + BF_3 \cdot O(C_2H_5)_2$ obviously takes place according to Scheme 3. The resultant acetals of vinyl acetaldehyde are added to a molecule of the vinyl alkyl ether, thus forming 1,1,3-trialkoxy compounds (VI) (Scheme 4). This reaction, however, proceeds slowly and with low yields of the compounds (VI). The highest yield was 24%. The infrared spectra of the autocondensation products of vinyl ethyl and vinyl butyl ethers showed frequencies characteristic of the vinyl group $(CH_2 = CH-)$ (Ref.9). The spectrum of the diethyl acetal

Card 2/3

84876

Chemistry of Unsaturated Ethers. V. Acetals S/079/60/030/010/016/030 of Vinyl Acetaldehyde. A New Method of B001/B066 Synthesizing 1-Alkoxy-dienes-1,3

of croton aldehyde shows an absorption band characteristic of a substituted vinyl group (Ref. 9). When passing acetal vapors of vinyl acetaldehyde in vacuo at 350°C over the acid catalyst MgHPO₄, 1-alkoxy-

butadiene-1,3 (VII) resulted (Scheme 5). With maleic aldehyde, the 1-alkoxy-dienes-1,3 gave the adducts (VIII). Their hydrolysis (Scheme 6) yields crystalline alkoxy-tetrahydrophthalic acids (IX). There are 12 references: 9 Soviet, 2 US, and 1 Japanese.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii

(Moscow Institute of Fine Chemical Technology)

SUBMITTED: December 7, 1959

Card 3/3

MAKIN, S.M.; ROZHKOV, I.N.

Chemistry of unsaturated ethers. Part 6: Condensation of acetals with 1-alkoxyddenss. Zhur.ob.khim. 31 no.10:3214-3220 0 '61. (MIRA 14:10)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V.Lomonosova. (Acetals) (Olefins)

MAKIN, S.M.; ROZHKOV, I.N.

Chemistry of unsaturated others. Part 7: Addition of -aldehyde acetal to 1-ethoxy-2-methyl-1,3-butadiene. Zhur.ob. khim. 31 no.10:3319-3323 0 '61. (MIRA 14:10)

l. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V.Lomonosova. (Aldehydes) (Butadience) (Ethers)

MAKIN, S.M.

New method of producing polyene aldehydes. Dokl.AN SSSR 138 no.2: 387-389 My '61. (MIFA 14:5)

la Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova. Predstavleno akademikom I.V.Kminyantsem. (Aldehydes)

MAKIN, S.M.: TELEGINA, N.I.

Chemistry of unsaturated ethers. Part 9: Alkoxylation of furan. Synthesis of dialdehydes and glycols. Zhur.ob.khim. 32 no.4: 1104-1111 Ap '62. (MIRA 15:4)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii.
(Furan) (Alkoxy groups) (Glycols)

MAKIN, S.M.; NAZAROVA, D.V.; KIRSANOVA, E.A.; SMIRNOVA, L.N.

Chemistry of unsaturated ethers. Part 10: Addition reactions of 1-alkoxy-1,3-dienes. Zhur.ob.khim. 32 no.4:1111-1116 Ap '62. (MIRA 15:4)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii.
(Unsaturated compounds) (Alkoxy groups)

MAKIN, S.M.; NAZAROVA, D.V.

Chemistry of unsaturated ethers. Part 11: Synthesis of polyene ethers based on 1-alkoxy-1,3-dienes. Zhur.ob.khim. 32 no.4: 1117-1119 Ap '62. (MIRA 15:4)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii. (Unsaturated compounds) (Alkoxy groups)

MAKIN, S.M.; KRUPTSOV, B.K.

Chemistry of unsaturated ethers. Part 12: Structural orientation of diene condensations of 1-alkoxydienes with asymmetrical dienophyls. Zhur.ob.khim. 32 no.8:2521-2527 Ag '62.

(MIRA 15:9)

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

(Butadiene) (Ethers)

MAKIN, S.M.; KRUPTSOV, B.K.; MEDVEDEVA, V.M.; SMIRNOVA, L.N.

Chemistry of unsaturated ethers. Part 13: Reaction of acetalization of 1,1,3-trialkoxyalkanes and the synthesis of 1-alkoxy-1,3-dienes with heavy alkoxy groups. Ultraviolet spectra and Raman spectra of 1-alkoxy-1,3-dienes. Zhur.ob.khim. 32 no.8:2527-2535 Ag 162.

(MIRA 15:9)

l. Moskovskiy institut to koy khimicheskoy tekhnologii imeni M.V. Lomonosova.

(Butadiene-Spectra) (Alkany groups)

MAKIN, S.M.	_		
prepari	ry of unsaturated ethers ng retinens (aldehyde of 0:3159-3161 0 '62.	Part 14: New method of Vitamin A). Zhur.ob.khim. (MIRA 15:11)	
1. Mos	kovskiy institut tonkoy	khimicheskoy tekhnologii	
imeni M	(Retinal)	(Ethers)	

APPROVED FOR RELEASE: 06/20/2000 CIA-RDP86-00513R001031610005-4"

MAKIN, S.M.; SUDAKOVA, V.S.

Chemistry of unsaturated ethers. Part 15: Telomerization of vinyl ethyl ether with acetaldehyde acetal. Synthesis of 1-alkoxypolyenes. Zhur.ob.khim. 32 no.10:3161-3166 0 '62. (MIRA 15:11)

l. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V. Lomonosova. (Ethers) (Acetaldehyde) (Polymerization)

MAKIN, S.M.; ROZHKOV, I.N.; SUDAKOVA, V.S.

Chemistry of unsaturated ethers. Part 16: Telomerization of 1-alkoxy-1,3-dienes with acetals of unsaturated aldehydes. Zhur.ob.khim. 32 no.10:3166-3170 0 '62. (MIRA 15:11)

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

(Unsaturated compounds)

(Acetals)

(Polymerization)

<u>l 27182-65</u> EPA(s)-2/EVI(m)/EPF(c)/EPR/EVP(j)/T Pc-L/Pr-L/Ps-E/Pt-10 WW/RM ACCESSION NR: AP4009481 S/0063/63/00B/006/0708/0709

SOURCE: Vsesoyuznoye khimicheskoye obshchestvo. Zhurnal, v.8, no. 6, 1963. 708-709

TOPIC TAGS: polymethine polymer, dialdehydic polymer, polymethine dialdehydic polymer, dialdehyde phosphonium, salt polycondensate, thermal stability, magnetic susceptibility, polyene polymer

ABSTRACT: 1,4-bis-(triphenylphosphonium bromide)-butene-2(I) or p-zylylene-bis-(triphenylphosphonium chloride) (II) were reacted with octatriene-2,4,6-dial (III) or with terephthalic aldehyde (IV) (equations shown in the enclosure) to form polymers consisting exclusively of (CH=CH)_n and/or -C₆H₄-CH=CH- linkages. The polycondensation is carried out in absolute ether in the presence of sodium ethylate without separating the bis-alkylidenephosphorane. There is no halide in the product, and IR spectra show the terminal groups are aldehydic. All the products are powders, which melt above 4000

Card 1/j

L 27182-65

ACCESSION NR: AP4009481

end are insoluble in organic solvents. The oxygen content and the polymer weight (by 10%) of compound V increased on storage in air. With prolonged boiling in water, oxygen content increases 35%. EPR signal intensity decreases with increase in oxygen content. These polymers are thermaily stable have catalytic activity and increased magnetic susceptibility. Abstractor's note: author apparently calls the (OH=OH), gouping "methine", this work does not relate to the trivalent HO=radical 7. Orig. art. has: 1 table and 1 set of equations.

ASSOCIATION: None

SUBMITTED: 20Sep63

ENOL: 02

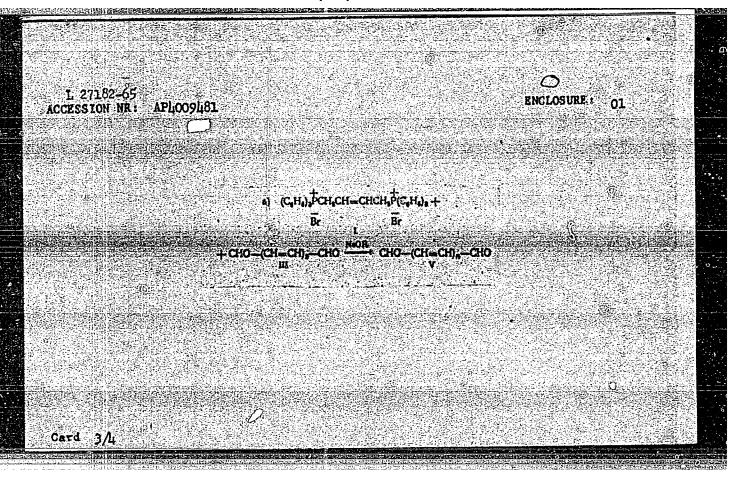
SUB CODE: 10,60

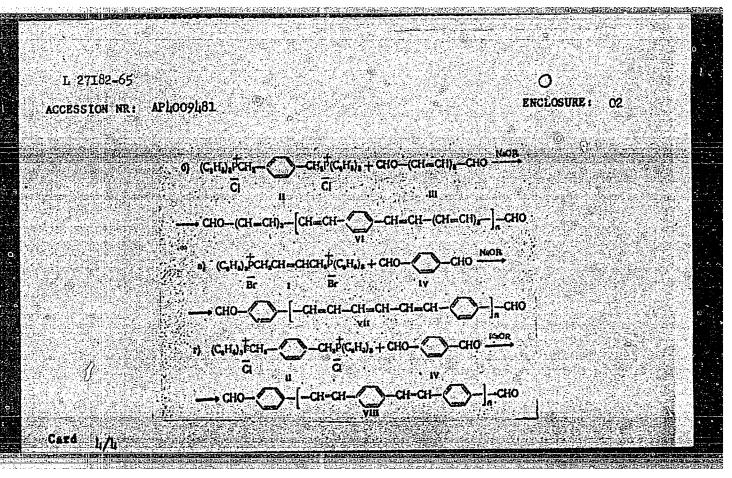
NR REF SOV: 005

OTHER: 007-

card 2/4

"APPROVED FOR RELEASE: 06/20/2000 CIA-RDP86-00513R001031610005-4





SHAVRYGINA, O.A.; MAKIN, S.M.

Chemistry of unsaturated ethers. Part 17: Synthesis of acetals of aryl polyene aldehydes. Zhur.ob.khim. 33 no.10:3176-3180 0 163. (MIRA 16:11)

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

TO THE TAXABLE PROPERTY OF THE PROPERTY OF THE

ROZHKOV, I.N.; MAKIN, S.M.

Interaction of A, 3 -unsaturated ketones and esters with metalloorganic compounds. Zhur.ob.khim. 34 no.1:59-65 Ja '64. (MIRA 17:3)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova.

MAKIN, S.M.; LAPITSKIY, G.A.; STREL'TSOV, R.V.

Chemistry of unsaturated ethers. Part 18: Synthesis of unsaturated dialdehydes and their derivatives. Zhur.ob.khim. 34 no.1:65-70 Ja 164. (MIRA 17:3)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova.

LAPITSKIY, G.A.; MAKIN, S.M.

"JAN

Synthesis of polymeric Schiff bases with a system of conjugate bonds. Zhur.VKHO 9 no.1:116-117 '64. (MIRA 17:3)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova.

MAKIN, 3. M.: LIKHOCHE.STOW, V. M.; SHEDEXINA, M. I.

Synthesis of 2,6-i alkoxy-4-dicydropyrans (cyclic acetals of glutaronaldehyde). Zhur. ob. knim. 34 no.6:1809-1915 de ica.

(MINA 17:7)

1. Meskewskiy inst tut tonkoy khimicheskoy tekhnologii imeni Lomonosova.

L 21733-65 EWT(m)/EPF(c)/EPR/EWP(1)/T Po-4/Pr-4/Pa-4 RPL RM/WW/JW

ACCESSION NR: AP4044192 S/0079/64/034/008/2564/2566

AUTHOR: Lapitskiy, G. A.; Makin, S. M.; Dy*mshakova, G. M.

TITLE: Chemistry of unsaturated esters. XIX, Synthesis of octatriene-2, 4, 6-

dioic-1,8 acid and some of its derivatives

SOURCE: Zhurhal obshchey khimii, v. 34, no. 8, 1964, 2564-2566

TOPIC TAGS: octatrienedioic acid, derivative, dinitrile, dichloranhydride, bifunctional polyene compound

ABSTRACT: Octatriene-2, 4, 6-dioic-1, 8 acid, HOOC-(CH=CH)₃-COOH (I) was synthesized from octatriene-2, 4, 6-dial-1, 8 (II), either by direct oxidation with silver oxide or by heating the corresponding dinitrile N=C-(CH=CH)₃-C=N (III) with 75% sulfuric acid. III was synthesized directly from II with hydroxylamine and acetic anhydride, or from the dioxime HO-N=CH-(CH=CH)₃-CH=N-OH (IV) with acetic anhydride. IV was prepared by reacting II and hydroxylamine hydrochloride in pyridine solution. I treated with thionyl chloride gave the dichloran-

Card 1/2

L 21733-65

ACCESSION NR: AP4044192

 hydride of octatriene-2, 4, 6-dioic-1, 8 acid. The latter compound entered in polycondensation reaction with diamines to form polymers! The bifunctional polyene compounds are high melting and are relatively highly stable in comparison to the corresponding monofunctional polyene compounds. Orig. art. has: 2 equations.

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

SUBMITTED: 15Jun63

ENCL: 00

SUB CODE: GC

NO REF SOV: 002 OTHER: 005

Card 2/2

CIA-RDP86-00513R001031610005-4" APPROVED FOR RELEASE: 06/20/2000

MAKIN, S.M.; LIKHOSHERSTOV, V.M.

Synthesis of dianils of glutaconic aldehyde from 2,6-dialkoxy

\$\times^2\$-dihydropyrans. Zhur. org. khim. 1 no.4:640-643 Ap '65.

(MIRA 18:11)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova.

Indiviral activity of 2,6-minlkoxymyron and 2-alkoxy- \$\Delta 5-dibutropyrans (cyclic acetal: of clutaric aldebyda derivatives). Farm. 1 toko. 28 no.1:66-69 Ja-F 165.

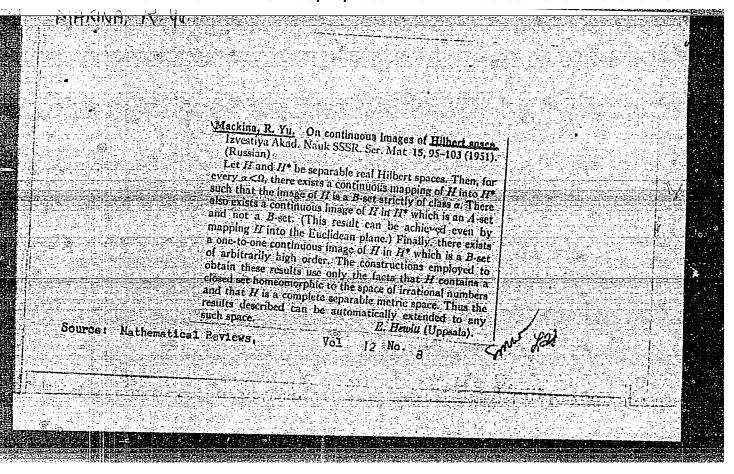
(MIRA 18:12)

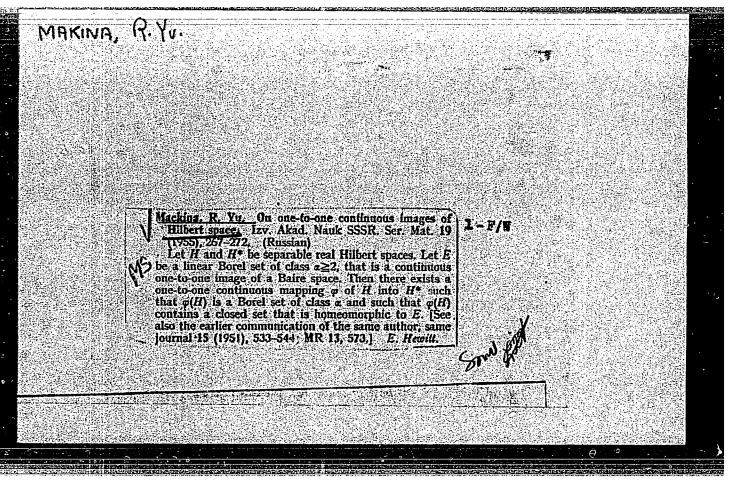
1. Vsesoyuznyy nauthno issledovateliskiy khimiko-farmatsevti-cheskiy institut imeni 3.0cdzhonjkidze i Moakovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V.Lomonosova, Modern. Submitted September 5, 1963.

en en er er en mensker år han men kom kylle (i fyrings), det g

ACC NR: AP6031299 SOURCE CODE: UR/0366/66/002/009/1586/1589 AUTHOR: Shavrygina, O. A.; Nazarova, D. V.; Makin, S. M. ORG: Moscow Institute of Fine Chemical Technology im. M. V. Lomonosov (Moskovskiy institut tonkoy khimicheskoy tekhnologii) TITLE: Chemistry of unsaturated ethers. XXIV. Preparation of vitamin A ethers SOURCE: Zhurnal organicheskoy khimii, v. 2, no. 9, 1966, 1586-1589 TOPIC TAGS: vitamin, ether, CHEMICAL REACTION Ethyl ether of vitamin A (bp 144—147°C, n_D^{20} 1.5735) was obtained by the following reactions: $\begin{array}{cccc} \text{CH}_2 = \text{CH} - \text{C} = \text{CHOC}_2\text{H}_5 & \xrightarrow{\text{(CH,CO),NBr}} & \text{BrCH}_2\text{CH} = \text{C} - \text{CH}(\text{OC}_2\text{H}_5)_2 \\ & \text{(1)} & \text{(1)} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} \\ & & \text{(1)} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} \\ & & \text{(1)} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} \\ & & \text{(1)} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} & \text{(CH,CO),NBr} \\ & & \text{(1)} \\ & & \text{(1)} & \text{(1)}$ → H₅C₂OCH₂CH=C-CH(OC₂H₅)₂

H⁺
H₅C₂OCH₂CH=C-CHO HC=CLi Ċн, ĊН, (111) 1/2 UDC: 547.37





MAKINOVA, F. A.

1257. Ogranizatsiya ucheta raskhoda matekialov v bor'be za ikh ekonomiyu i snizheniye sebostoimosti promyshlennoy produktsii. (Na opyte predlpriyatiy mestnoy prom-sti g. Leningrada_/. L. 1954. 20s. 20sm. (M-vo Vyssh. obrazovaniya SSSR. Leningr. Fin-ekon. in-t). 100 ekz. B. ts. - \[\int 54-51634 \]

SO: Knizhnaya Letopis, Vol. 1, 1955

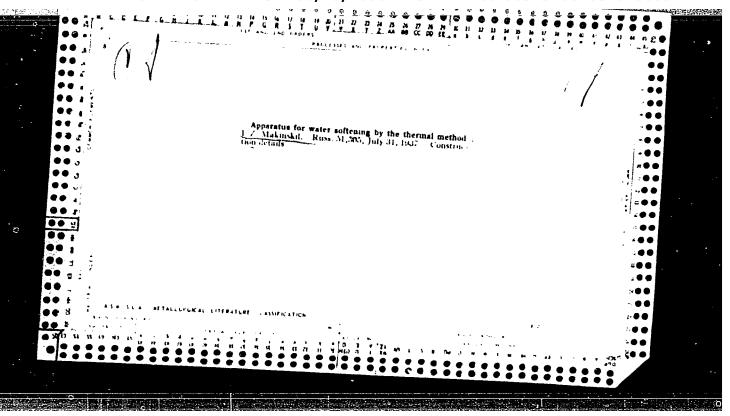
USER/Chemistry - Alcohols
Synthesis

"Derivation of Alcohols by the Grignard Method from
Olefin Oxides: FI, Synthesis of Propylisopropylcarbinol (I), Isopropylisobutylcarbinol, (II) and Isopropylbutylcarbinol (III) From Isoamylene Offide,"

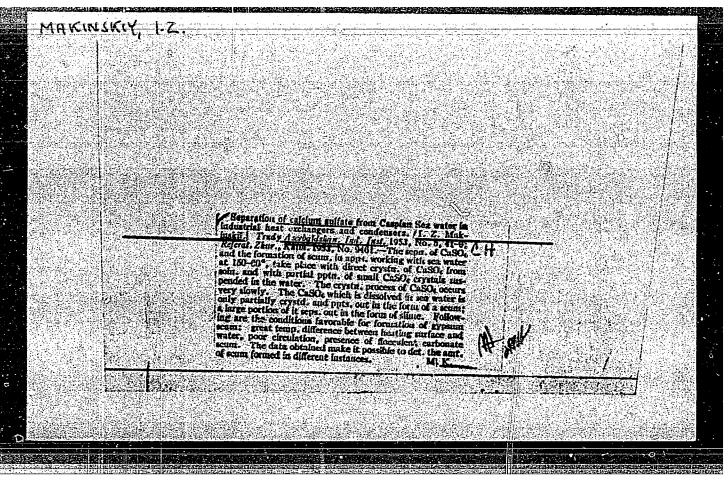
E. S. Mallnowskiy, B. N. Konevichev, Stud, 3 pp

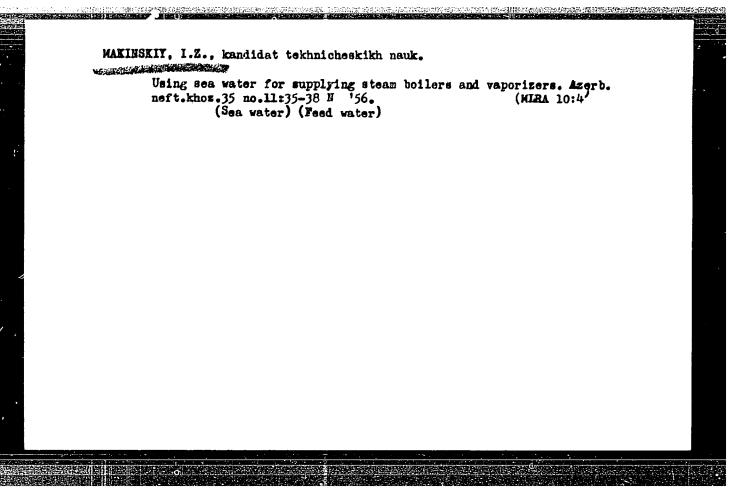
"Zhur Obshch Khim" Vol XVIII, No 10

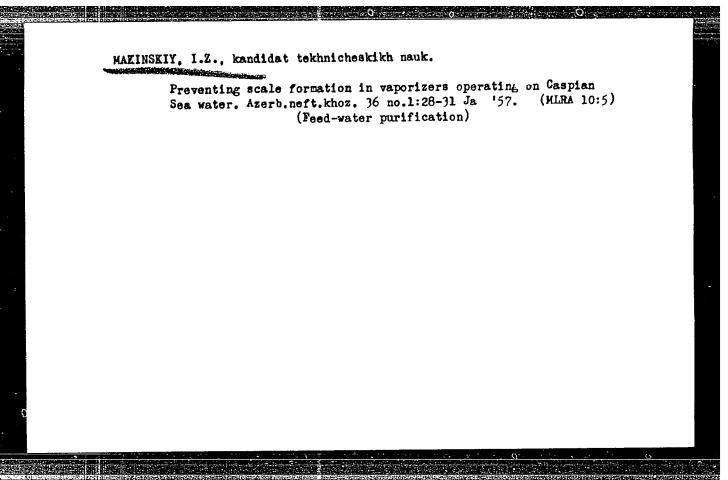
Isosmylene oxide reacted with ethylmagnesium bromide,
propylmagnesium bromide, and isopropylmagnesium bromide to give alcohols I, II, and III from isoamylene
oxide. Samitted 1 Oct 47.



"APPROVED FOR RELEASE: 06/20/2000 CIA-RDP86-00513R001031610005-4



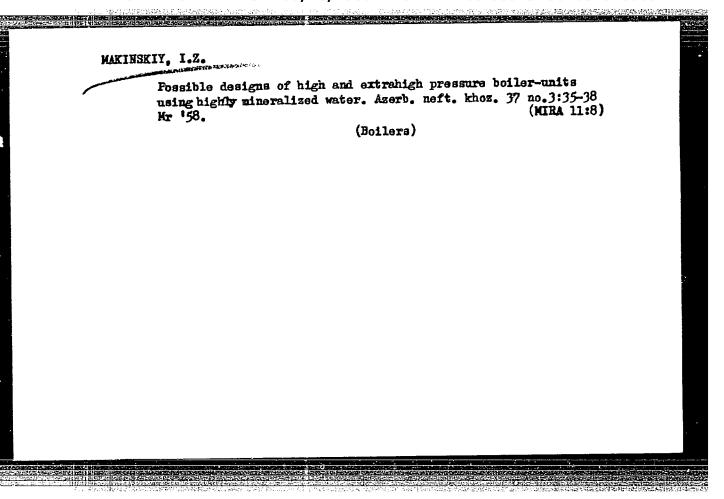




MAKINSKIY, I.Z.

Thermochemical method for softening sea water. Izv.vys. ucheb. zav.; neft' i gaz no.4:95-103 '58. (MIRA 11:9)

1.Azerbaydzhanskiy industrial'nyy institut im. M. Azizbekova. (Sea water)



10(4), 5(4)

AUTHORS:

Makinskiy, I. Z., Simonov, P. P.

SOV/152-59-3-19/25

TITLE:

On the Problem of the Influence of the Salt Content of the Liquid on the Ascending Velocity of Gas and Vapor Bubbles (K voprosu o vliyanii solesoderzhaniya zhidkosti na skorost'

pod"yema puzyr'kov gaza i para)

PERIODICAL:

Izvestiya vysshikh uchebnykh zavedeniy. Neft' i gaz, 1959, Nr 3, pp 83-90 (USSR)

ABSTRACT:

The experiments were partly carried out with single bubbles introduced from below from calibrated glass nozzles into a 1500 mm high glass tube filled with the liquid to be investigated, partly by bubbling up the surface of the liquid through boiling or pressing air through. Solutions with varying concentration of NaCl, Na₂SO₄, MgCl₂ and MgSO₄ were investigated. A number of

experiments was carried out under the addition of isoamyl alcohol for the purpose of investigating the effect of surfaceactive substances. In order to determine the influence of insoluble colloids, disperse Mg(OH)₂ was formed out of an

addition of NaOH and $MgSO_A$. With increasing NaCl-content

Card 1/4

On the Problem of the Influence of the Salt Content of the Liquid on the Ascending Velocity of Gas and Vapor Bubbles **SOV/152-59-3-19/25**

the capability of the air bubbles to unite decreases and ceases at approximately $5 \, \text{g/l}$ so that a large number of fine, slowly ascending air bubbles forms. At a salt content of more than $25 \, \text{g/l}$ the capability of uniting returns and becomes especially conspicuous at contents over $40 \, \text{g/l}$. For the ascending velocity wof a gas bubble the simplified formula

 $w = 3.63\sqrt{\frac{d}{k}}$ is set up (w = the ascending velocity in m/sec, d = the diameter of the gas bubble in m. V. Levich (Ref 3)

suggested another formula: $w \approx \frac{1}{36} \frac{d^2}{\sqrt{Fl}}$ (F_l = specific weight of the liquid, μ is not explained). Experiments showed that in the case of lower salt contents the ascending velocity follows the formula by Levich, in the case of higher salt contents, however, it passes over into the curve according to the first formula after a steep descend. The delay in velocity cannot only be explained by viscosity. Surface-active substances accumulate in the cover of the gas bubble and the velocity of

Card 2/4

On the Problem of the Influence of the Salt Content of the Liquid on the Ascending Velocity of Gas and Vapor Bubbles SOV/152-59-3-19/25

the bubble approaches that of a solid body. By proper dosing of isoamyl alcohol the velocity can be reduced to a constant value (~15 cm/sec), independent of the salt concentration; it will correspond to that of a solid ball. It is also possible to assume the solution to be a solution of water in salt. It becomes obvious that with increasing salt content the conditions of circumflowing deteriorate to begin with, later, however, as soon as the composition of the surface film approaches that of the liquid the effect of the salt content decreases. In the surface film a supersaturation occurs under the formation of insoluble colloids which reduce the velocity. In the case of bubbles of a diameter being more than 1 mm deviations occur as a result of deformation. On boiling, the bubbling also depends on the tendency of the vapor bubbles towards flowing together. NaCl shows with colloidal Mg(OH)₂

an intensified bubbling between 5-10 g/l, where flowing together ceases. The explanation for this: the colloidal parts form quasi-solid bubble covers which render flowing

Card 3/4

On the Problem of the Influence of the Salt Content of the Liquid on the Ascending Velocity of Gas and Vapor Bubbles sov/152-59-3-19/25

together more difficult at certain salt concentrations. At higher salt contents the colloids coagulate, no formation of covers takes place and the bubbles can flow together again. The same problem had already been investigated by A. Frumkin, V. Levich, B. Gorodetskaya and others. The effect of substances solved out of the wall of the vessel was detected by T. A. Kryukova (Ref 4) and S. A. Durov (Ref 5). There are 6 figures, 2 tables, and 7 Soviet references.

ASSOCIATION: Azerbaydzhanskiy industrial nyy institut im. M. Azizbekova (Azerbaydzha. Industrial Institute neni M. Azizbekov)

SUBMITTED: December 22, 1958

Card 4/4

MAKINSKIY, I.Z.; SINONOV, P.P.

Supplying steam to petroleum refineries. Izv.vys.ucheb.zav.; neft 1 gaz 3 no.2:87-94 '60. (HIRA 13:6)

1. Azerbaydzhanskiy institut nefti i khimii im. M.Azizbekova. (Steam)

MAKINSKIY, I. Z.

Doc Tech Sci - (diss) "Softening of sea-water and the problem of its utilization for supplying boilers operating at high and at superhigh pressures." Moscow, 1961. 34 pp with diagrams; (Academy of Sciences USSR, Power Inst imeni G. M. Krzhizhanovskiy); 200 copies; price not given; list of author's works at end of text (14 entries); (KL, 7-61 sup, 230)

MAKINSKIY, I.Z.; SIMONOV, P.P.

Supplying steam to chemical plants and petroleum refineries.

Izv. vys. ucheb. zav.; neft' i gaz 5 no.3:75-78 '62.

(MIRA 16:8)

1. Azerbaydzhanskiy institut nefti i khimii imeni M. Azizbekova.

L 45613-66

ACC NR. AP6025425

SOURCE CODE: UR/0143/66/000/007/0113/0115

AUTHOR: Makinskiy, I. Z. (Professor, Doctor of Technical Sciences); Abdullayev, K. M. (Engineer)

ORG: Azglavenergo

TITLE: Methods of selecting vaporization stages of optimum efficiency for boilers with stage vaporization

SOURCE: IVUZ. Energetika, no. 7, 1966, 113-115

(N)

TOPIC TAGS: steam boiler, vaporization, archose, industrial separator, distribution, water vapor, calculation

ABSTRACT: Four-stage vaporization stages with inside cylindrical cyclones in the first stage and outside cyclones in the other stages were studied because there is no common method for determining the load distribution among the vaporization stages. The solution is sought by the analytic method. A system of equations is set up and formulas for optimum throughput of the vaporization stages and for relative quality of the vapor are obtained. The calculation results show that the throughput of the first, second, third, and fourth vaporization stages is 56, 26.1, 12.2, and 5.7%, respectively. The principles presented in this study make it possible to analyze the effect of various factors on the load distribution among the vaporization stages of boilers. Orig. art. has: 1 figure and 17 formulas.

SUB CODE: 13/ SUBM DATE: 11Dec65/ ORIG REF: 002

Card 1/1 UDC 621.186.6+621.18

SOV/25-58-12-38/40

AUTHOR: Makinskiy, T.A., Learned Secretary

TITLE: The Epidemical Encephalitis (Epidemicheskiy entse-

falit)

PERIODICAL: Nauka i zhizn', 1958, Nr 12, pp 75-76 (USSR)

ABSTRACT: The symptoms of the encephalitis lethargica are

described, as well as the medicine used to fight

it.

ASSOCIATION: Institut nevrologii Akademii meditsinskikh nauk

SSSR (The Institute of Neurology of the Academy

of Medical Sciences of the USSR)

Card 1/1

SHMIDT, Ye.V., prof.; MAKINSKIY, T.A.

Studies on vascular diseases of the nervous system in the Soviet
Union in 1958; based on data from the first conference on research
planning and coordination conducted by the Institute of Neurology of
the Academy of Medical Sciences of the U.S.S.R. in November 1957]
Issledovania v oblasti sosudistykh porazhenii nervnoy sistemy v

Sovetskom Soiuze v 1958 g.; po materialam pervogo soveshchanila po planirovanilu i koordinatsii nauchnykh issledovanil, provedennogo Institutom nevrologii AMN SSSR v noiabre 1957 g. Vest. AMN SSSR 13 no.4:63-68 158. (HIRA 11:4)

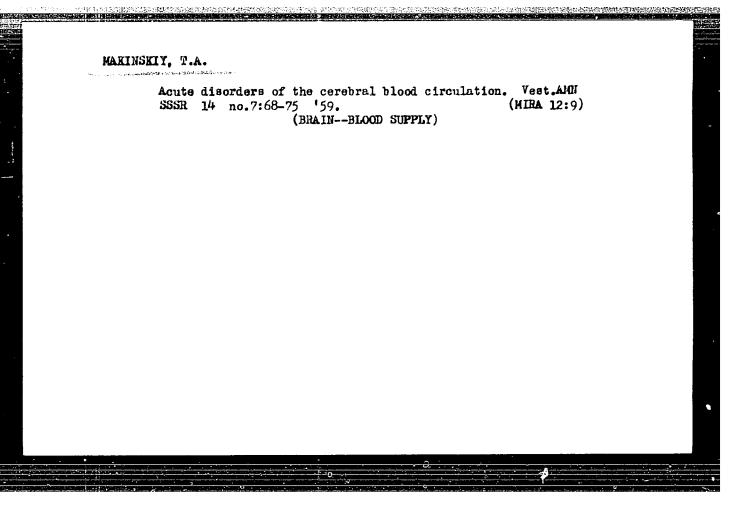
(BRAIN, blood supply, dis., review (Rus))



First conference on the coordination of Soviet research on vascular affections of the nervous system. Zhur.nevr. i psikh. 28 no.9:1154

[MIRA 11:11]

(MERVOUS SYSTEM_BLOOD SUPPLY)



MAKINSKIY, T.A.

Out-of-town session of the I stitute of Neurology of the Academy of Medical Sciences of the U.S.S.R. Zhur. nevr. i psikh 59 no.3:377-378 159.

(BRAIN-DISEASES) (POLICHYELITES)

MAKINSKIY, T.A.

Joint session of the Institute of Neurology of the Soviet Academy of Medical Sciences and the Kiev Medical Institute.

Zhur. nerv. i psikh. 60 no. 12:1695 '60. (MIRA 14:4)

(NERVOUS SYSTEM—DISEASES)