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RESEARCH ARTICLE

Synthesis of alkyl-*gem*-dichlorocyclopropanes based on isoamylene fraction

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Objectives. *The study aims to analyze the dichlorocarbenation of the isoamylene fraction, which is a mixture of 2-methyl-butene-1 and 2-methyl-butene-2, in order to obtain the corresponding alkyl-gem-dichlorocyclopropanes in quantitative yield, and also to determine their structure.*
Methods. *In order to determine the qualitative and quantitative composition of the reaction masses, the following analysis methods were used: gas-liquid chromatography (using the Crystal 2000 hardware complex), mass spectrometry (using a Chromatec-Crystal 5000M device with NIST 2012 database), and nuclear magnetic resonance (NMR) spectroscopy (using a Bruker AM-500 device at operating frequencies of 500 and 125 MHz).*
Results. *Alkyl-gem-dichlorocyclopropanes were synthesized from an isoamylene fraction in the presence of catamine AB as a catalyst. Alternatively, isomeric alkenyl-gem-dichlorocyclopropanes were obtained on the basis of isoprene, and by reduction, the corresponding alkyl-gem-dichlorocyclopropanes were synthesized. The synthesized substances were analyzed by gas-liquid chromatography, mass spectrometry, and NMR spectroscopy, as previously mentioned above.*
Conclusions. *The results show that the dichlorocyclopropanation of the isoamylene fraction proceeds quantitatively with the formation of a mixture of 2-methyl-2-ethyl-1,1-dichlorocyclopropane and 2,3,3-trimethyl-1,1-dichlorocyclopropane. Using isoprene, counter-synthesis through successive dichlorocarbenation and hydrogenation was used to synthesize 2-methyl-2-ethyl-1,1-dichlorocyclopropane, one of the products of dichlorocarbenation of the isoamylene fraction.*

Keywords: *alkenyl-gem-dichlorocyclopropane, isoamylene fraction, hydrogenation, Pd/C*

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НАУЧНАЯ СТАТЬЯ

Синтез алкил-*гем*-дихлорциклопропанов на основе изоамиленовой фракции

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Цели. *Изучить дихлоркарбенирование изоамиленовой фракции, представляющей собой смесь 2-метил-бутена-1 и 2-метил-бутена-2, получить соответствующие алкил-гем-дихлорциклопропаны с количественным выходом и установить их строение.*
Методы. *Для определения качественного и количественного состава реакционных масс были использованы газожидкостная хроматография (на аппаратно-программном комплексе «Кристалл 2000»), хроматомасс-спектрометрия (на приборе «Хроматэк-Кристалл 5000М» с базой NIST 2012), и спектроскопия ядерного магнитного резонанса (ЯМР-спектроскопия) (на приборе «Bruker AM-500» с рабочими частотами 500 и 125 МГц).*
Результаты. *Алкил-гем-дихлорциклопропаны синтезированы из изоамиленовой фракции в присутствии катализатора катамина АВ. Альтернативным путем на основе изопрена получены изомерные алкенил-гем-дихлорциклопропаны, восстановлением которых синтезированы соответствующие алкил-гем-дихлорциклопропаны. Вещества проанализированы и доказаны методами газожидкостной хроматографии, хроматомасс-спектрометрии и ЯМР-спектроскопии.*
Выводы. *Установлено, что дихлорциклопропанирование изоамиленовой фракции протекает количественно с образованием смеси 2-метил-2-этил-1,1-дихлорциклопропана и 2,3,3-триметил-1,1-дихлорциклопропана. С использованием изопрена встречным синтезом через последовательное дихлоркарбенирование и гидрирование был синтезирован 2-метил-2-этил-1,1-дихлорциклопропан – один из продуктов дихлоркарбенирования изоамиленовой фракции.*

Ключевые слова: *алкенил-гем-дихлорциклопропаны, изоамиленовая фракция, гидрирование, Pd/C*

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INTRODUCTION

In the industrial production of isoprene by dehydrogenation of pentanes (at the first stage), pentenes (isoamylene fraction) with a total volume of 2-methylbut-1-ene **1** and 2-methylbut-2-ene **2** (of at least 80%) are formed. This mixture, along with the formation of isoprene, is used in the alkylation, the epoxidation of the Prince reaction, and others as well [1–4].

At an earlier point in time [5–8], it was shown that alkenyl- and alkyl-*gem*-dichlorocyclopropanes are used as solvents, plasticizers, and intermediate products in organic synthesis. In this regard, it was of interest to study the preparation of substituted alkyl-*gem*-dichlorocyclopropanes by dichlorocarbenation of an industrial isoamylene fraction.

MATERIALS AND METHODS

The analysis of the reaction masses was carried out using gas-liquid chromatography (on a Crystal 2000 hardware-software complex, *NPF Meta-khrom*, Russia). Mass spectra were obtained using a Chromatec-Cristall 5000M instrument (*Chromatec*, Russia) with the NIST 2012 database (*National Institute of Standards and Technology*, USA). Nuclear magnetic resonance (NMR) spectra of ^{1}H and ^{13}C were recorded on a Bruker AM-500 spectrometer (*Bruker Corporation*, USA) with operating frequencies of 500 and 125 MHz, respectively, in a $CDCl_3$ solvent. Chemical shifts are reported on a δ (ppm) scale relative to tetramethylsilane as the internal standard. Spin-spin coupling constants (J) are given in Hz.

We used substituted vinyl-*gem*-dichlorocyclopropanes obtained by the known method [9] using chloroform, a 50% alkali solution, and a catamine AB phase transfer catalyst (*NPK Isomer*, Russia).

Using the same technique, we obtained the following:

2-methyl-2-ethyl-1,1-dichlorocyclopropane 3. Yield (**3** + **4**) 95%, T_{boil} = 52°C (35 mm Hg). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm. (J, Hz): 1.07 t (3H, CH_3, J = 7.9), 1.23 d (1H, 1 CH_a, J = 7.1), 1.28 d (1H, CH_b, J = 7), 1.36 d (3H, CH_3), 1.59–1.73 m (2H, CH_2). ^{13}C NMR, δC, ppm: 10.74 (CH_3), 19.65 (CH_3), 29.75 (C), 31.56 (CH_2), 33.03 (CH_2), 68.03 (C). Mass spectrum, *m/e* (I_{rel}, %): 152/154/156 (≤3) $[M]^+$, (137/139/141)/(22/10/3), (123/125)/(30/15), (117/119)/(50/20), (101/103)/(10/3), (79/77)/(30/22), 56/100.

2,3,3-trimethyl-1,1-dichlorocyclopropane 4. Yield (**3** + **4**) 95%, T_{boil} = 52°C (35 mm Hg). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm. (J, Hz): 1.16 t (6H, 2 CH_3, J = 3.9, 4.0), 1.36 t (3H, CH_3, J = 6.6), 1.16 t (1H, CH, J = 7.2). ^{13}C NMR, δC, ppm: 9.55 (CH_3), 16.86 (CH_3), 24.73 (CH_3), 28.06 (C), 32.64 (CH), 71.46 (C). Mass spectrum, *m/e* (I_{rel}, %): 152/154/156 (≤3) $[M]^+$, (137/139/141)/(50/25/5), (123/125)/(≤5), (117/119)/(100/30), (101/103)/(22/8), (79/77)/(50/30), 56/(≤5).

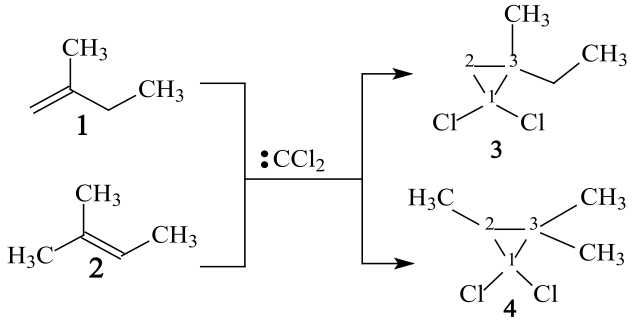
The hydrogenation procedure for unsaturated compounds **6a**, **6b** is similar to that previously published [10].

The catalyst is palladium-carbon, Pd/C granular[1], TU 2170-300-29131036-97. Before use, it was ground in a mortar, sieved, and stored in a desiccator.

By this method, a mixture of substances **3**, **3a** was obtained. The physicochemical constants of compound **3** are presented above. For compound **3a** they correspond to the literature data [11].

RESULTS AND DISCUSSION

As a result of our studies, we found that dichlorocarbenation by the Makoshi method [12, 13] of an isoamylene fraction containing olefins **1**, **2** in a ratio of 1 : 5 gives a mixture (T_{boil} = 156–158°C) 2-methyl-2-ethyl-1,1-dichlorocyclopropane **3** and 2,3,3-trimethyl-1,1-dichlorocyclopropane **4** in the same ratio (Scheme 1, Fig. 1).



Scheme 1. Carbenation of the amylene fraction **1**, **2**.

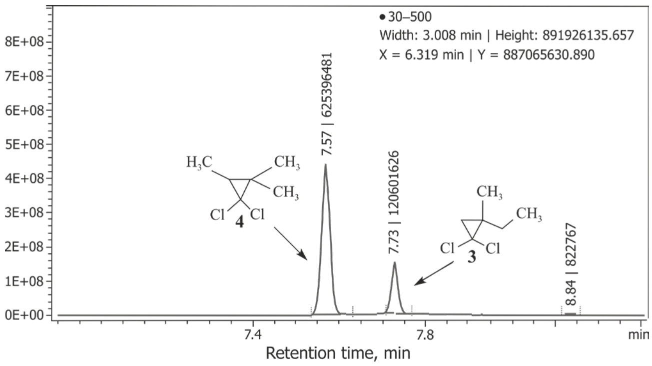
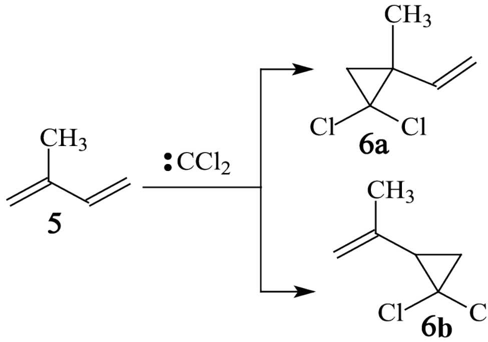


Fig. 1. Chromatogram of a mixture of alkyl-*gem*-dichlorocyclopropanes **3**, **4**.

Earlier [9], we carried out selective monocarbenation of isoprene **5** to vinyl- and isopropenyl-*gem*-dichlorocyclopropanes **6a,b** (Scheme 2), which are formed in a ratio of 95 : 5 (Fig. 2).



Scheme 2. Carbenation of 2-methyl-2-vinyl-butadiene-1,3 **5**.

Hydrogenation (Scheme 3) of a mixture of alkenyl-*gem*-dichlorocyclopropanes **6a,b**, according to the known method [14, 15], was carried out on an industrial catalyst Pd/C [14, 15] at a temperature of 22–24°C and atmospheric pressure for 4–5 h. A mixture of products **3**, **7** was obtained in a quantitative yield, in which their ratio corresponded to the content of olefins **6a,b** (Fig. 3).

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[1] Information about the catalyst is available on the manufacturer's website https://www.kazanorgsintez.ru.

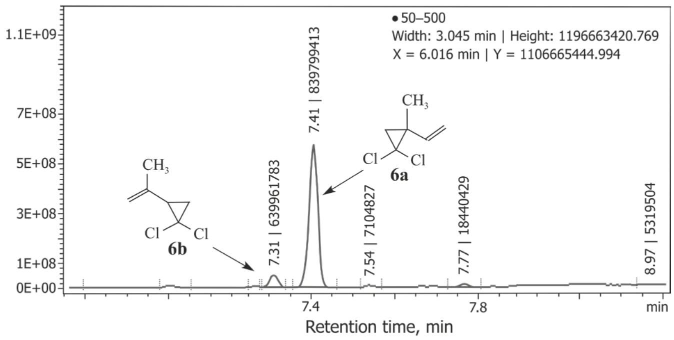


Fig. 2. Chromatogram of a mixture of alkenyl-*gem*-dichlorocyclopropanes **6a,b**.

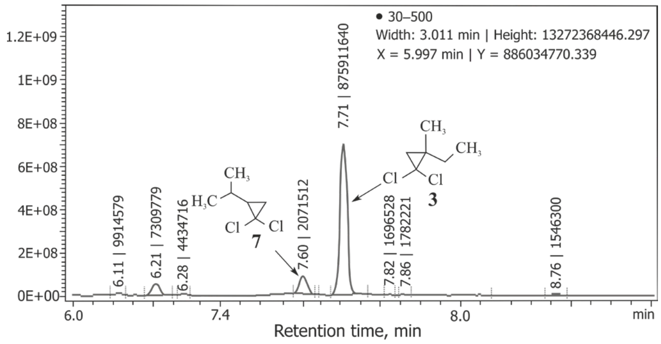
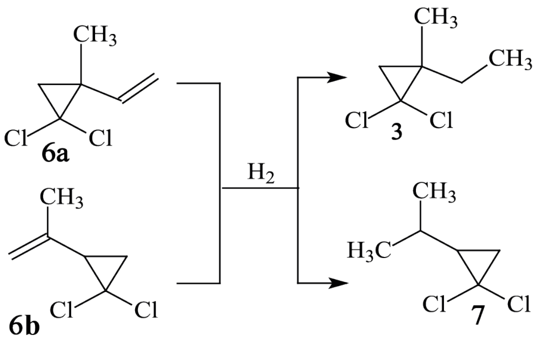


Fig. 3. Chromatogram of a mixture of hydrogenation products **3**, **7**.



Scheme 3. Hydrogenation of a mixture of alkenyl-*gem*-dichlorocyclopropanes **6a,b**.

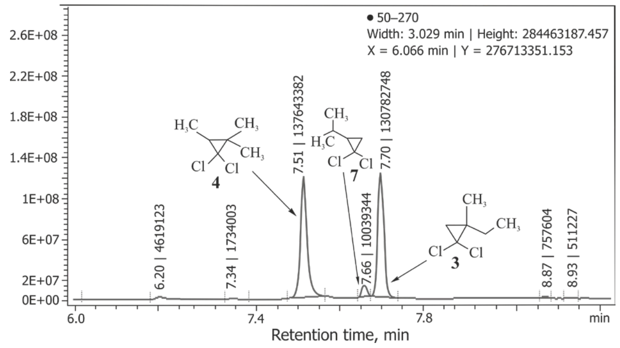


Fig. 4. Chromatogram of an artificial mixture of alkenyl-*gem*-dichlorocyclopanes hydrogenation products **3**, **7** and a mixture of dichlorocarbenation products of the amylene fraction **3**, **4**.

The chromatogram of an artificial mixture of the carbeneation products of the isoamylene fraction **3**, **4**, and the hydrogenation products of alkenyl-*gem*-dichlorocyclopane **3**, **7** indicates the identity of 2-methyl-2-ethyl-*gem*-dichlorocyclopropane **3**, which is obtained by different methods (Fig. 4).

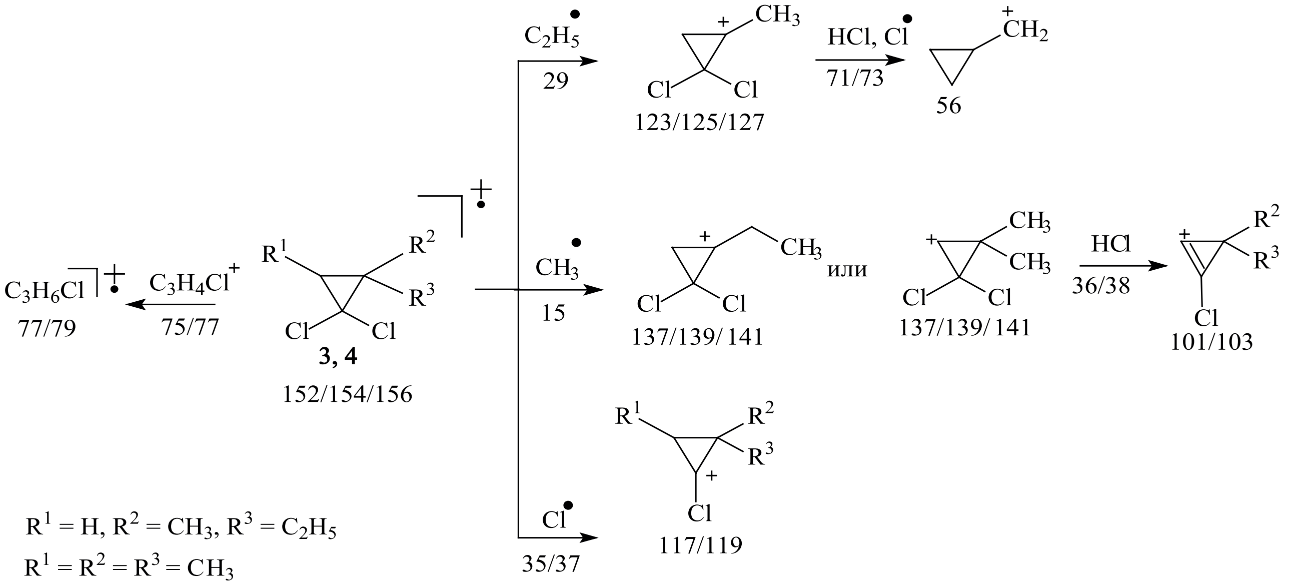
The NMR spectrum of the obtained mixture of alkenyl-*gem*-dichlorocyclopanes **3**, **4** contains signals of protons of the cyclopropane ring at δ C^2 and $C^{2''}$ carbon atoms. For molecule **3**, protons at δ C^2 carbon atom appear as two doublets at 1.23 ppm ($^2J = 7.1$ Hz) and 1.28 ppm ($^2J = 7$ Hz), while for molecule **4** the proton at δ $C^{2''}$ carbon atom is a triplet in the region of 1.16 ppm ($^3J = 7.2$ Hz). Methyl groups are registered with a triplet for 2-methyl-2-ethyl-1,1-dichlorocyclopropane **3** at 1.07 ppm ($^3J = 7.9$ Hz) and a doublet at 1.36 ppm ($^2J = 6.2$ Hz), for 2,3,3-trimethyl-1,1-dichlorocyclopropane **4**—at 1.16 ppm ($^3J = 6.6$ Hz) triplet and 1.36 ppm ($^2J = 3.9$ Hz) doublet.

In the ^{13}C NMR spectra of the mixture of alkyl-*gem*-dichlorocyclopropanes **3**, **4**, the common signal is the C^1 signal of the carbon atom at 68.03 ppm and 71.46 ppm, respectively. For isomer **3** δ C^2 and C^3, the carbon atoms of cyclopropane are recorded in the region of 33.03 ppm and 29.75 ppm, respectively, and for isomer **4** δ $C^{2''}$ and $C^{3''}$ carbon atoms—at 32.64 ppm and 28.06 ppm, respectively. The presence of high-field signals of carbons for molecule **3** in the region of 10.74 ppm and 19.65 ppm corresponds to methyl groups, for molecule **4** the same groups appear at 9.55 ppm and 24.76 ppm.

Mass spectra of a mixture of *gem*-dichlorocyclopropanes **3**, **4** contain peaks of molecular ions *m* = 152/154/156 of low intensity (≤3%). Dissociative ionization of *gem*-dichlorocyclopropanes **3**, **4** can proceed in different ways. The molecule decomposes into a dichlorocyclopropane fragment and a substituent R, or the molecule loses chlorine atoms, but the main carbon skeleton is retained (Scheme 4).

The table shows the values of fission ion mass *m* and the relative intensity of the ion peaks *e* (% of the maximum) for compounds **3**, **4**.

Therefore, based on the frequency of occurrence and intensity of the peaks (see table), the most stable fragment in the molecule is the cyclopropane fragment, which forms a fragmented ion upon the decomposition of all compounds **3**, **4** with a *gem*-dichlorocyclopropane substituent.



Scheme 4. Dissociative ionization of *gem*-dichlorocyclopropanes **3**, **4**.

Values of fission ion mass *m* and relative intensity of ion peaks *e* (% of maximum) for compounds **3** and **4**

Compounds	*m/e* (%)						
	M^+ 152/154/156	56	77/79	101/103	117/119	123/125	137/139/141
3	≤3	100	30/22	10/3	50/20	30/15	22/10/3
4	≤3	≤3	55/30	22/8	100/30	≤5	50/25/5

CONCLUSIONS

It was found that dichlorocarbenation of the isoamylene fraction, by the Makoshi method, proceeds with the formation of isomeric alkyl-*gem*-dichlorocyclopropanes: 2-methyl-2-ethyl-1,1-dichlorocyclopropane and 2,3,3-trimethyl-1,1-dichlorocyclopropane. Using isoprene, counter-synthesis through sequential dichlorocarbenation and hydrogenation was used to synthesize 2-methyl-2-ethyl-1,1-dichlorocyclopropane, one of the products of dichlorocarbenation of the isoamylene fraction.

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Authors' contribution

A.I. Musin – conducting research, literature analysis;

Yu.G. Borisova – collection and processing of the material, writing the text of the article;

G.Z. Raskil'dina – collection and processing of the material, statistical processing;

R.R. Daminev – consultation on planning, methodology and implementation of the study;

R.U. Rabaev – consultation on conducting individual stages of the study;

S.S. Zlotskii – development of the concept of scientific work, critical revision with the introduction of valuable intellectual content.

The authors declare no conflicts of interest.

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