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RESEARCH ARTICLE

Dichlorocarbenation of polar olefins in conditions of microwave irradiation

Yulianna G. Borisova✉**, Airat I. Musin, Rimma M. Sultanova, Simon S. Zlotskii**
Ufa State Petroleum Technological University, Ufa, 450064 Russia
✉ *Corresponding author, e-mail: yulianna_borisova@mail.ru*

Abstract

Objectives. To evaluate the influence and efficiency of using microwave irradiation on the dichlorocarbenation of polar olefins. To determine the conditions (reaction time and process temperature) under which the maximum yield of target *gem*-dichlorocyclopropanes is achieved.

Methods. The target compounds were obtained by classical methods of organic synthesis—acetalization of polyols and dichlorocarbenation of unsaturated compounds. The preparation of *gem*-dichlorocyclopropanes was carried out using the microwave activation method on a Sineo device (microwave system for organic synthesis, made in China). In order to determine the qualitative and quantitative composition of the reaction masses, gas–liquid chromatography (using the Kristall 2000 hardware complex), mass-spectroscopy (using Chromatek-Kristall 5000M device with NIST 2012), and nuclear magnetic resonance spectroscopy (using Bruker AM-500 device with operating frequencies of 500 and 125 MHz) were carried out.

Results. Under microwave irradiation at 25°C for 2 h with the maximum yield (92–98%), the target substituted *gem*-dichlorocyclopropanes were obtained: 2-(2,2-dichloro-3-methylcyclopropyl)-1,3-dioxolane, 2-(2,2-dichloro-3-phenylcyclopropyl)-1,3-dioxolane, 8,8-dichloro-4-isopropyl-3,5-dioxabicyclooctane, diethyl-2,2-dichloro-3-phenylcyclopropane-1,1-dicarboxylate, and diethyl-2,2-dichloro-3-isopropylcyclopropane-1,1-dicarboxylate.

Conclusions. Under the conditions herein proposed, the use of the microwave stimulation method in the dichlorocarbenation of double C=C bonds containing polar substituents allows the reduce the temperature and reaction time to be significantly reduced, and the yield of target *gem*-dichlorocyclopropanes to be increased.

Keywords
dichlorocarbenation, Mokosh method, microwave radiation, olefins, phase transfer catalysis

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НАУЧНАЯ СТАТЬЯ

Дихлоркарбенирование полярных олефинов в условиях микроволнового излучения

Ю.Г. Борисова✉**, А.И. Мусин, Р.М. Султанова, С.С. Злотский**

Уфимский государственный нефтяной технический университет, Уфа, 450064 Россия

✉*Автор для переписки, e-mail: yulianna_borisova@mail.ru*

Аннотация

Цели. Оценить влияние и эффективность использования микроволнового излучения на дихлоркарбенирование полярных олефинов; определить условия (продолжительность реакции и температуру проведения процесса), при которых достигается максимальный выход целевых *гем*-дихлорциклопропанов.

Методы. Целевые соединения были получены классическими методами органического синтеза — ацетализацией полиолов и дихлоркарбенированием непредельных соединений. *Гем*-дихлорциклопропаны были получены методом микроволновой активации с помощью микроволновой системы для проведения органических синтезов «Sineo» (Китай). Для определения качественного и количественного состава реакционных масс использовались газожидкостная хроматография (на аппаратно-программном комплексе «Кристалл 2000»), масс-спектроскопия (на приборе «Хроматэк-Кристалл 5000М» с базой NIST 2012) и спектроскопия ядерного магнитного резонанса (на приборе «BrukerAM-500» с рабочими частотами 500 и 125 МГц).

Результаты. В условиях микроволнового излучения при 25°С за 2 ч с максимальным выходом (92–98%) получены целевые замещенные *гем*-дихлорциклопропаны: 2-(2,2-дихлор-3-метилциклопропил)-1,3-диоксолан, 2-(2,2-дихлор-3-фенилциклопропил)-1,3-диоксолан, 8,8-дихлоро-4-изопропил-3,5-диоксабициклооктан, диэтил-2,2-дихлоро-3-фенилциклопропан-1,1-дикарбоксилат и диэтил-2,2-дихлоро-3-изопропилциклопропан-1,1-дикарбоксилат.

Выводы. В предложенных условиях использование метода микроволновой активации при дихлоркарбенировании двойных С=С связей, содержащих полярные заместители, позволяет существенно снизить температуру, уменьшить продолжительность реакции и повысить выход целевых *гем*-дихлорциклопропанов.

Ключевые слова

дихлоркарбенирование, метод Макоши, микроволновое излучение, олефины, межфазный катализ

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INTRODUCTION

Polyfunctional *gem*-dichlorocyclopropanes find application in the synthesis of low-tonnage products, reagents, and biologically active compounds [1–5]. In addition, molecules containing the *gem*-dichlorocyclopropane fragment are important intermediates which can be modified into more complex structures exhibiting a variety of properties [6]. The main method for the preparation of compounds of this class is dichlorocarbenation of double C=C bonds using the Mokosh method [7–10].

The aim of this work is to determine the effect of microwave radiation (MWR) on the multiple bond attachment of dichlorocarbenes, since under such conditions a high yield can be achieved in a short time [11].

MATERIALS AND METHODS

Using the hardware-software complex Chromatek-Crystal 5000M (*Chromatek*, Russia) with NIST 2012 database (*National Institute of Standards and Technology*, USA), reaction masses were analyzed and mass spectra of compounds were recorded. The conditions of analysis were as follows: capillary quartz column — 30-m long; analysis duration, 20 min; ion source temperature, 260°C; transition line temperature, 300°C; scanning range, 30–300 Da; pressure, 37–43 mTorr; and carrier gas—helium; heating rate, 20 deg/min). The electron impact ionization method, 70 eV, was used to obtain mass spectra of the compounds. ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AM-500 spectrometer (*Bruker Corporation*, USA) with operating frequencies of 500 and 125 MHz, respectively. The solvent used was $CDCl_3$ (Russia). Chemical shifts are given on the δ scale (ppm) relative to tetramethylsilane as internal standard. Spin-spin interaction constants (*J*) are given in Hz.

The basic methodology of dichlorocarbenization under thermal conditions is presented in [9].

Synthesis of compounds 2a-b and 4a,b under microwave radiation conditions

A mixture of 0.01 mol of the olefin 2-[(1E)-prop-1-en-1-yl]-1,3-dioxolane **1a** [9], 2-[(E)-2-phenylvinyl]-1,3-dioxolane **1b** [12], 2-isopropyl-4,7-dihydro-1,3-dioxepin **1c** [13], diethyl-(2-methylpropylidene)-malonate **3a** [13], diethylbenzylidene malonate **3b** [14], 30 mL of chloroform, 32 g of 50% sodium hydroxide solution and 1% by weight of triethylbenzylammonium chloride was stirred under MWR conditions at a given temperature. The progress of the reaction was monitored by gas–liquid chromatography. At the end of the reaction the reaction mixture was washed with water, extracted with chloroform (Russia), dried with calcium chloride (Russia) and evaporated. The target compounds were then isolated by means of vacuum distillation.

2-(2,2-Dichloro-3-methylcyclopropyl)-1,3-dioxolane (**2a**). Colorless liquid. Boiling point $T_{b.p.}$ = 98–99°C (5 mm Hg). 78% yield. Mass spectrum *m*/*z*, (I_{rel}, %): 195/197/199 $[M]^+$, (0.37/0.25/0.04), 123/125/127 (2.44/1.98/0.31), 109/111/113 (1.33/0.75/0.15), 75/77 (5.95/1.65), 73 (100), 45 (45), 43 (5.6), 39 (11.44). The spectral characteristics are in accordance with [9].

2-(2,2-Dichloro-3-phenylcyclopropyl)-1,3-dioxolane (**2b**). 92% yield. Colorless liquid. $T_{b.p.}$ = 103–104°C (2 mm Hg). The spectral characteristics are in accordance with [15]. Mass spectrum *m*/*z*, (I_{rel}, %): 260 (1) $[M]^+$, 252 (2), 219 (4), 147 (12), 114 (20), 101 (8), 77 (10), 73 (96), 63 (5), 46 (30).

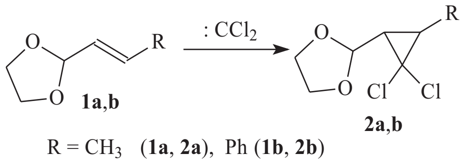
8,8-Dichloro-4-isopropyl-3,5-dioxabicyclooctane (**2c**). 98% yield. Colorless liquid. $T_{b.p.}$ = 103–104°C (2 mm Hg). The spectral characteristics are in accordance with [16]. Mass spectrum *m*/*z*, (I_{rel}, %): (188/190)/(20/7), (77/75)/(100/35), (109/111)/(45/17), (51/53)/(80/30).

Diethyl-2,2-dichloro-3-phenylcyclopropane-1,1-dicarboxylate (**4a**). 92% yield. Colorless liquid. $T_{b.p.}$ = 154–155°C (2 mm Hg). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.36 t (3H, CH_3, ^3J 7.0), 3.45 s (CH_3), 3.89 qu. (2H, CH_2, ^3J 6.9), 7.20–7.40 (Ph-). ^{13}C NMR, δ_C, ppm: 15.30 (CH_3), 43.34 (CH), 52.00 (C), 62.49 (CH_2), 74.12 (C), 127.16–131.61 (Ph-), 162.02 (C=O). The spectral characteristics are in accordance with [14].

Diethyl-2,2-dichloro-3-isopropylcyclopropane-1,1-dicarboxylate (**4b**). 92% yield. Colorless liquid. $T_{b.p.}$ = 154–155°C (2 mm Hg). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.32 t (3H, CH_3, ^3J 7.1), 3.45 s (1H, CH, ^3J 7.1), 3.48 s (3H, CH_3), 4.32 qu. (4H, CH_2, ^3J 7.2), 7.40–7.49 (Ph-). ^{13}C NMR, δ_C, ppm: 14.33 (CH_3), 46.73 (CH), 55.92 (C), 58.45 (CH_3), 65.48 (CH_2), 78.10 (C), 127.15–130.61 (Ph-), 201.08 (C=O). The spectral characteristics are in accordance with [14].

RESULTS AND DISCUSSION

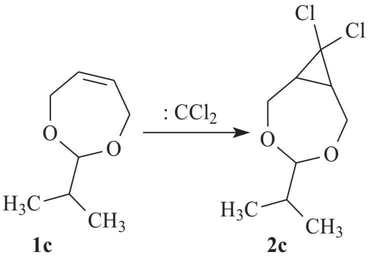
Dichlorocarbenation of cyclic acetals of unsaturated aldehydes **1a**,**b** under microwave irradiation enabled *gem*-dichlorocyclopropanes **2a**,**b** to be obtained at room temperature (25°C) in 1–2 h in quantitative yield (Scheme 1).



Scheme 1. Dichlorocarbenation of cyclic acetals of unsaturated aldehydes

It took 4–5 h to achieve similar results under thermal heating conditions (40°C) (Table 1). It should be noted that under the conditions being studied *trans*-**1a**,**b** form *trans*-*gem*-dichlorocyclopropanes **2a**,**b**.

The use of MBI for dichlorocarbenization of the endocyclic C=C bond in 2-isopropyl-4,7-dihydro-1,3-dioxepin **1c** was successful (Scheme 2).



Scheme 2. Dichlorocarbenation of 2-isopropyl-4,7-dihydro-1,3-dioxepine

The corresponding bicyclic product **2c** was obtained at room temperature in 2 h in quantitative yield, whereas thermal heating (40°C) required 5 h (Table 1).

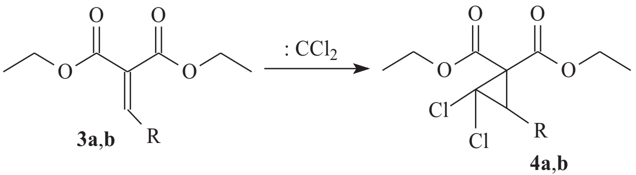
In dichlorocarbenation under MWR conditions, it was found that the endocyclic double C=C bond in 2-isopropyl-4,7-dihydro-1,3-dioxepin **1c** is 2 times more active than the exocyclic double C=C bond in 1,3-dioxolane **1a** (method of competitive reactions, conversion of initial olefins **1a**,**c** is not more than 30%).

1,1,2-Trisubstituted double C=C bonds in aryl- and alkylidenmalonates **3a**,**b** obtained according to the method [15] under thermal conditions show a low level of activity towards dichlorocarbenes (Scheme 3).

The yield of phenyl-substituted *gem*-dichlorocyclopropane **4a** using thermal heating (40°C) for 5 h is no more than ≤5%. With an increase in temperature the destruction of ether groups and intensive osmosis is observed. Using MWR at 25°C for 5 h the corresponding 1,1,2-trisubstituted *gem*-dichlorocyclopropane **4a** was obtained in 40% yield (Table 2).

Table 1. Synthesis conditions and yield of dichlorocarbenation products of compounds **2a-c**

No.	Initial compounds	Reaction conditions		Reaction products	Yield, %	Heating method
		T, °C	Reaction time, h			
1	**1a**	40	4	**2a**	35	Thermal heating
		40	5		40	
		25	1		55	MWR
		25	2		92	
2	**1b**	40	4	**2b**	70	Thermal heating
		40	5		90	
		25	1		70	MWR
		25	2		98	
3	**1c**	40	4	**2c**	70	Thermal heating
		40	5		93	
		25	1		60	MWR
		25	2		98	



R = Ph (**3a**, **4a**), *i*-C_3H_7 (**3b, 4b**)

Scheme 3. Dichlorocarbenation of aryl- and alkylidenemalonates **3a**,**b**

Isopropylidene malonate **3b** was more active in the dichlorocarbenation reaction. Under thermal conditions (40°C, 5 h) the target product **4b** is formed in 30% yield. When using MWR (25°C, 2 h), there is quantitative formation of the product (Table 2).

Table 2. Synthesis conditions and yield of dichlorocarbenation products of compounds **4a**,**b**

No.	Initial compounds	Reaction conditions		Reaction products	Yield, %	Heating method
		T, °C	Reaction time, h			
1	**3a**	40	4	**4a**	≤1	Thermal heating
		40	5		≤3	
		25	1		28	MWR
		25	2		40	
2	**3b**	40	4	**4b**	25	Thermal heating
		40	5		30	
		25	1		70	MWR
		25	2		98	

CONCLUSIONS

Based on the results obtained, the use of MWR in the dichlorocarbenylation of double C=C bonds containing polar substituents allows the temperature and reaction time to be significantly reduced, and the yield of target *gem*-dichlorocyclopropanes to be increased.

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Authors' contributions

Yu.G. Borisova—collecting and processing the material, writing the text of the article.

A.I. Musin—conducting research.

R.M. Sultanova—planning consultations.

S.S. Zlotskii—conceptualization of the research paper, critical revision with the introduction of valuable intellectual content.

The authors declare no conflict of interest.

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About the authors

Yulianna G. Borisova, Cand. Sci. (Chem.), Teacher, Department of General, Analytical and Applied Chemistry, Ufa State Petroleum Technological University (1, Kosmonavtov ul., Ufa, 450064, Russia). E-mail: yulianna_borisova@mail.ru. Scopus Author ID 56526865000, Researcher ID P-9744-2017, RSCI SPIN-code 3777-0375, https://orcid.org/0000-0001-6452-9454

Airat I. Musin, Postgraduate Student, Department of General, Analytical and Applied Chemistry, Ufa State Petroleum Technological University (1, Kosmonavtov ul., Ufa, 450064, Russia). E-mail: musin_1995@list.ru. ResearcherID R-9142-2016, RSCI SPIN-code 9573-4624, https://orcid.org/0000-0002-8662-9680

Rimma M. Sultanova, Dr. Sci. (Chem.), Professor, Department of General, Analytical and Applied Chemistry, Ufa State Petroleum Technological University (1, Kosmonavtov ul., Ufa, 450064, Russia). E-mail: rimmams@yandex.ru. Scopus Author ID 6602738038, RSCI SPIN-code 8208-6060, https://orcid.org/0000-0001-6719-2359

Simon S. Zlotskii, Dr. Sci. (Chem.), Professor, Head of the Department of General, Analytical and Applied Chemistry, Ufa State Petroleum Technological University (1, Kosmonavtov ul., Ufa, 450064, Russia). E-mail: nocturne@mail.ru. Scopus Author ID 6701508202, ResearcherID W-6564-2018, RSCI SPIN-code 6529-3323, https://orcid.org/0000-0001-6365-5010

Об авторах

Борисова Юлианна Геннадьевна, к.х.н., преподаватель кафедры общей, аналитической и прикладной химии, ФГБОУ ВО «Уфимский государственный нефтяной технический университет» (450064, Россия, г. Уфа, ул. Космонавтов, д. 1). E-mail: yulianna_borisova@mail.ru. Scopus Author ID 56526865000, Researcher ID P-9744-2017, SPIN-код РИНЦ 3777-0375, https://orcid.org/0000-0001-6452-9454

Мусин Айрат Ильдарович, аспирант кафедры общей, аналитической и прикладной химии, ФГБОУ ВО «Уфимский государственный нефтяной технический университет» (450064, Россия, г. Уфа, ул. Космонавтов, д. 1). E-mail: musin_1995@list.ru. ResearcherID R-9142-2016, SPIN-код РИНЦ 9573-4624, https://orcid.org/0000-0002-8662-9680

Султанова Римма Марсельевна, д.х.н., профессор кафедры общей, аналитической и прикладной химии, ФГБОУ ВО «Уфимский государственный нефтяной технический университет» (450064, Россия, г. Уфа, ул. Космонавтов, д. 1). E-mail: rimmams@yandex.ru. Scopus Author ID 6602738038, SPIN-код РИНЦ 8208-6060, https://orcid.org/0000-0001-6719-2359

Злотский Семен Соломонович, д.х.н., заведующий кафедрой общей, аналитической и прикладной химии, ФГБОУ ВО «Уфимский государственный нефтяной технический университет» (450064, Россия, г. Уфа, ул. Космонавтов, д. 1). E-mail: nocturne@mail.ru. Scopus Author ID 6701508202, ResearcherID W-6564-2018, SPIN-код РИНЦ 6529-3323, https://orcid.org/0000-0001-6365-5010

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