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RESEARCH ARTICLE

Synthesis and anticorrosive activity of *tert*-amines containing cycloacetal or *gem*-dichlorocyclopropane fragments and quaternary ammonium salts on their basis

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Abstract

Objectives. The work set out to synthesize tertiary amines comprising derivatives of morpholine and piperidine containing a 1,3-dioxolane or *gem*-dichlorocyclopropane fragment, as well as quaternary ammonium salts based on them. In order to determine the process conditions (duration and temperature of the reaction) under which the maximum possible yield of the target quaternary ammonium salts is achieved, the effect of the halide structure on the yield of *tert*-amines and their subsequent salts was evaluated. The study also aimed to establish the structural and spatial structure of the obtained carbo- and heterocyclic amines and salts based on them, as well as to evaluate the anticorrosive properties of the obtained products in a hydrogen sulfide medium.

Methods. The target compounds, such as tertiary amines and quaternary ammonium salts (QAS), were obtained by classical methods of organic synthesis consisting of alkylation and condensation of the corresponding amines of various structures. Preparation of QAS was carried out using a microwave system for organic synthesis via microwave activation on a Sineo device (China). The qualitative and quantitative composition of the reaction masses was determined using gas–liquid chromatography (Crystal 2000 hardware and software complex), while mass spectroscopy was carried out on a Chromatec-Crystal 5000M device with a NIST 2012 database). A Bruker AM-500 device having operating frequencies of 500 and 125 MHz was used to perform nuclear magnetic resonance spectroscopy.

Results. Tertiary amines containing a cycloacetal or *gem*-dichlorocyclopropane fragment were obtained under thermal heating conditions. By carrying out their condensation in excess halides using microwave radiation, new quaternary ammonium salts were synthesized with a yield close to quantitative. Anticorrosive activity was estimated for the obtained cyclic compounds. 4-Allyl-4-[2-(1,3-dioxolan-2-yl)-ethyl]morpholinium chloride was determined to have the maximum protective effect in a hydrogen sulfide medium with a protection level of 91%.

Conclusions. Tertiary amines containing a cycloacetal or *gem*-dichlorocyclopropane fragment were obtained under the proposed conditions. Such substances are in demand as intermediates in the synthesis of quaternary ammonium salts having anticorrosive activity.

Keywords

alkylation, microwave radiation, quaternary ammonium salt, corrosion

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НАУЧНАЯ СТАТЬЯ

Синтез и антикоррозионная активность *трет*-аминов, содержащих циклоацетальный или *гем*-дихлорциклопропановый фрагмент, и четвертичных аммониевых солей на их основе

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Аннотация

Цели. Синтезировать третичные амины — производные морфолина и пиперидина, содержащие 1,3-диоксолановый или *гем*-дихлорциклопропановый фрагмент, а также четвертичные аммониевые соли на их основе. Оценить влияние строения галогенидов на выход *трет*-аминов и их последующих солей. Определить условия (длительность и температуру реакции) проведения процесса, при которых достигается максимально возможный выход целевых четвертичных аммониевых солей. Установить структурное и пространственное строение полученных карбо- и гетероциклических аминов и солей на их основе, а также оценить антикоррозионные свойства полученных продуктов в сероводородной среде.

Методы. Целевые соединения, такие как третичные амины и четвертичные аммониевые соли (ЧАС), были получены классическими способами органического синтеза — алкилированием и конденсацией соответствующих аминов различного строения. Получение ЧАС было осуществлено с использованием микроволновой системы для проведения органических синтезов методом микроволновой активации на приборе «Sineo» (Китай). Качественный и количественный состав реакционных масс были определены газожидкостной хроматографией (на аппаратно-программном комплексе «Кристалл 2000»), масс-спектроскопией (на приборе «Хроматэк-Кристалл 5000М» с базой NIST 2012) и спектроскопией ядерного магнитного резонанса (на приборе «Bruker AM-500» с рабочими частотами 500 и 125 МГц).

Результаты. В условиях термического нагрева получены третичные амины, содержащие циклоацетальный или *гем*-дихлорциклопропановый фрагменты, конденсация которых в избытке галогенидов с использованием микроволнового излучения позволила синтезировать новые четвертичные аммониевые соли с выходом, близким к количественному. Для полученных циклических соединений была оценена антикоррозионная активность. Определено, что максимальным защитным эффектом в сероводородной среде обладает 4-аллил-4-[2-(1,3-диоксолан-2-ил)этил]морфолиниум хлорид, который имеет степень защиты, равную 91%.

Выводы. В предложенных условиях были получены третичные амины, содержащие циклоацетальный или *гем*-дихлорциклопропановый фрагменты. Третичные амины служат промежуточными продуктами в синтезе четвертичных аммониевых солей, обладающих антикоррозионной активностью.

Ключевые слова

алкилирование, микроволновое излучение, четвертичная аммониевая соль, коррозия

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INTRODUCTION

Cyclic amines such as morpholine, piperazine, and piperidine, which are widely used in the synthesis of a wide range of biologically active products, are produced on an industrial scale [1–3]. The tertiary amines and their derivatives containing 1,3-dioxacycloalkane or *gem*-dichlorocyclopropane fragments obtained on their basis exhibit various forms of biological activity, such as antifungal (fungicidal), bactericidal (biocidal), herbicidal, etc. [4, 5]. Nitrogen-containing heterocyclic compounds are additionally used in the design and synthesis of quaternary ammonium salts (QAS) with antimicrobial properties [6, 7]. It should be noted that QAS containing *gem*-dichlorocyclopropane fragment exhibit antibacterial activity against *Escherichia coli*,

Klebsiella pneumoniae, *Staphylococcus aureus*, *Acinobacter Baumanii* [8]. Prof. A. Vereshchagin, who synthesized QAS on the basis of cyclic acetals of pyridine aldehyde and hydroxypyridine esters, established their ability to inhibit the growth of gram-positive and gram-negative bacteria, fungi, and some viruses in low concentrations [9–11]. The series of works by Acad. A.L. Maksimov *et al.* demonstrate various applications of acetals in petrochemistry [12–14]. For example, cyclic acetals, which are well dispersed in lubricating compositions, can be used as active high-octane components of anti-wear additives in diesel fuel and other energy carriers to reduce the corrected diameter of the wear spot [15–17]. Heterocycles and their analogs or derivatives (esters, amides and salts) have antioxidant properties and inhibit acid corrosion of metals [18, 19].

Thus, the synthesis of new QAS containing 1,3-dioxacycloalkane and *gem*-dichlorocyclopropane structures seems to be important and relevant in terms of the creation of new petrochemical reagents and pharmacological preparations.

We previously showed that quaternary ammonium salts derived from 2-chloromethyl-*gem*-dichlorocyclopropane and 4-chloromethyl-1,3-dioxolane are catalytic in the *O*-alkylation reaction of 2,2-dimethyl-4-oxymethyl-1,3-dioxolane with allyl chloride [20].

In the present study, new *tert*-amines and salts based on them were prepared using 1,1-dichloro-2-(chloromethyl)-2-methylcyclopropane and 2-bromoethyl-1,3-dioxolane to evaluate the anticorrosion activity of the obtained compounds.

MATERIALS AND METHODS

During the process of analyzing reaction masses, the mass spectra of compounds were recorded on the Chromatek-Crystal 5000M hardware-software complex (*Chromatek*, Russia) with the NIST 2012 database (*National Institute of Standards and Technology*, USA). The analysis conditions were as described in the article [9]. The electron impact ionization method was used to obtain mass spectra of 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 used solvent was deuterated chloroform $CDCl_3$. Chemical shifts are given on the δ scale (ppm) relative to tetramethylsilane as internal standard. The spin-spin interaction constants (*J*) are given in Hz.

1,1-Dichloro-2-(chloromethyl)-2-methylcyclopropane **3** was prepared by dichlorocarbenylation of 2-methyl-3-chloropropene-1 (CAS 1563-47-3) under interfacial catalysis conditions following a similar procedure as presented in [9]. Morpholine (CAS 110-91-8), piperidine (CAS 110-89-4) and 2-bromoethyl-1,3-dioxolane **4** (CAS 4360-63-8) are commercially available reagents.

Synthesis of compounds 5–7 under thermal heating conditions

The flask was loaded with 0.002 mol of amine (0.17 g of morpholine or piperidine), 0.004 mol of halide (0.69 g of 1,1-dichloro-2-(chloromethyl)-2-methylcyclopropane **3** or 0.72 g of 2-bromoethyl-1,3-dioxolane **4**) and 20 mL of dimethylformamide. The reaction mixture was stirred at 100°C for 8–11 h until complete conversion of the amine (control by gas–liquid chromatography). The mixture was washed with water, extracted with methylene chloride, and evaporated. The target product was isolated by vacuum distillation.

4-[2,2-Dichloro-1-methylcyclopropyl)methyl]-morpholine **5**. Colorless liquid. $T_{boil.}$ = 98–99°C (5 mm Hg). Yield 90% (0.40 g). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.32 d (2H, CH_2, *J* 4.14), 1.67 s (3H, CH_3), 2.43 d (4H, $2CH_2$, *J* 4.14), 2.57 d (2H, CH_2, *J* 12.74), 3.71 d (4H, $2CH_2$, *J* 4.11). ^{13}C NMR, δ_C, ppm: 19.98 (CH_3), 28.61 (C), 31.23 (CH_2), 53.32 (2 CH_2), 62.79 (CH_2), 66.75 (C), 66.97 ($2CH_2$). Mass spectrum *m*/*z* (I_{rel}, %): 222.98/224.99/226.99 (15/10/4), 187.99/190.02 (13/4), 127.02/129.02 (18/5), 124.06 (30), 99.89/101.05 (100/27), 84.99/87.00 (30/10), 73.03/74.91 (18/5), 56.00 (60).

1-[2,2-Dichloro-1-methylcyclopropyl)methyl]-piperidine **6**. Colorless liquid. $T_{boil.}$ = 93–95°C (5 mm Hg). Yield 93% (0.41 g). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.10 d (2H, CH_2, *J* 6.03), 1.65 s (3H, CH_3), 1.64–1.75 m (6H, $3CH_2$), 2.33 dd (4H, $2CH_2$, *J* 8.8), 2.61 d (2H, CH_2, *J* 12.01). ^{13}C NMR, δ_C, ppm: 19.81 (CH_3), 23.01 (CH_2), 23.55 ($2CH_2$), 28.73 (C), 31.26 (CH_2), 57.39 ($2CH_2$), 62.81 (CH_2), 66.71 (C). Mass spectrum *m*/*z* (I_{rel}, %): 221.01/223.01/225.01 (10/6/2), 186.01/188.01 (10/5), 138.07 (30), 124.06 (30), 97.92/99.08 (100/31), 83.02/85.03 (30/6), 69.06/71.05 (28/8), 55.00 (55).

4-[2-(1,3-Dioxolan-2-yl)ethyl]morpholine **7**. Colorless liquid. $T_{boil.}$ = 101–102°C (5 mm Hg). Yield 95% (0.35 g). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.82–1.85 m (2H, CH_2), 2.39–2.44 m (6H, $3CH_2$), 3.65–3.68 m (4H, $2CH_2$), 3.80 d (2H, CH_2, *J* 3.67), 3.91 d (2H, CH_2, *J* 3.73), 4.88 t (1H, CH, *J* 9.56). ^{13}C NMR, δ_C, ppm: 30.99 (CH_2), 40.96 (CH_2), 53.64 ($2CH_2$), 64.80 ($2CH_2$), 66.84 ($2CH_2$), 103.13 (CH). Mass spectrum *m*/*z* (I_{rel}, %): 188.14 (2), 114.06 (100), 101.09 (25), 86.00 (22), 70.02 (52), 56.03 (44).

Synthesis of compounds 11–13 under microwave heating conditions

The flask was loaded with 0.002 mol of *tert*-amine (0.44 g of amine **5** or **6** or 0.37 g of amine **7**), 0.004 mol (0.68 g of benzyl bromide **8**, 0.58 g of amyl bromide **9** or 0.3 g of allyl chloride **10**) and 20 mL of methyl isobutyl ketone. The reaction mixture was stirred under microwave irradiation (MWI) conditions at 30°C for 2–4 h until precipitation. The mixture was filtered off, the residue on the filter was washed with hexane (2 × 100 mL) and dried under vacuum.

4-Benzyl-4-[-2,2-dichloro-1-methylcyclopropyl)-methyl]morpholinium bromide **11**. Brown powder. $T_{melt.}$ = 167°C. Yield 95% (0.75 g). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.67 s (3H, CH_3), 1.65 d (1H, CH_a, ^2J 7.7), 1.90 d (1H, CH_a, *J* 7.4), 3.22 d (4H, $2CH_2$, *J* 4.37), 3.49 d (2H, CH_2, *J* 10.87), 3.98 d (4H, $2CH_2$, *J* 9.98), 4.83 s (2H, CH_2), 7.55–7.61 m (5H, Ph–). ^{13}C NMR, δ_C, ppm: 19.60 (CH_3), 26.07 (C), 31.44 (CH_2), 60.00 (CH_2), 60.59 ($2CH_2$), 63.50 (CH_2), 65.99 ($2CH_2$), 129.32 (2CH), 127.07 (CH), 128.52 (2CH), 134.22 (C).

1-Butyl-1-[2,2-dichloro-1-methylcyclopropyl)-methyl]piperidinium bromide **12**. Brown powder. $T_{melt.}$ = 162°C. Yield 92% (0.66 g). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 0.93 s (3H, CH_3), 1.01 d (2H, CH_2, *J* 10.87), 1.23–1.29 m (10H, $5CH_2$), 3.57–3.61 m (4H, $2CH_2$), 3.41 d (2H, CH_2, *J* 4.77), 4.03 d (2H, CH_2, *J* 12.03). ^{13}C NMR, δ_C, ppm: 13.31 (CH_3), 19.82 (CH_2), 19.89 (CH_3), 21.56 (CH_2), 23.29 ($2CH_2$), 23.05 (CH_2), 29.34 (CH_2), 28.74 (C), 67.45 ($2CH_2$), 67.89 (CH_2), 68.41 (CH_2).

4-Allyl-4-[2-(1,3-dioxolan-2-yl)ethyl]morpholinium chloride **13**. Brown powder. $T_{melt.}$ = 145°C. Yield 98% (0.51 g). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.25–1.34 m (2H, CH_2), 3.03–3.12 m (6H, $3CH_2$), 3.34–3.41 m (4H, $2CH_2$), 3.80 d (2H, CH_2, *J* 5.79), 3.98 d (2H, CH_2, *J* 5.72), 5.55 d (1H, CH, *J* 6.16), 5.70 dd (2H, CH_2, *J* 10.55), 5.90–6.01 m (1H, CH). ^{13}C NMR, δ_C, ppm: 31.03 (CH_2), 43.28 (CH_2), 51.07 ($2CH_2$), 59.94 (CH_2), 63.73 ($2CH_2$), 67.56 ($2CH_2$), 103.55 (CH), 125.86 (CH_2), 127.32 (CH).

Methodology for determination of anticorrosive activity of substances in a hydrogen sulfide-containing environment

The electrochemical method was used to study the anticorrosive activity of substances. Electrochemical analysis was carried out on the Monikor-2M corrosion rate analyzer (*Akurs-M*, Russia). The device includes two electrodes made of St3 steel (properties of St3 steel, requirements to chemical composition, control methods, and data on international quality standard are listed in GOST 380-2005[1]). Prior to testing, the surface of steel electrodes was prepared using emery paper #180, followed by emery paper #240, in the direction of the electrode length. The prepared electrode samples were degreased with dimethyl ketone (*Vekton*, Russia) immediately before carrying out the test. Next, the electrodes were activated by means of three-stage washing. The test substance (volume of 0.25 mL) was dissolved in 25 mL of ethyl alcohol (*Ruskhim*, Russia). Cylindrical laboratory cells were filled with the calculated amount of 3% sodium chloride solution (*Lenreaktiv*, Russia) and purged for 30 min with nitrogen (Orenburg, Russia). After purging, the calculated amount of hydrogen sulfide water[2] and 1.25 mL of dissolved substance in alcohol were poured into the medium. Then the electrodes were immersed in an electrochemical cell pre-filled with the test medium and the corrosion rate was determined for a period of 60 min. To obtain convergent and reliable results, parallel tests of 2 cells with the same medium were performed and arithmetic mean values of the obtained corrosion rates were calculated.

The braking coefficient was calculated by the formula:

$$A = \frac{P_0}{P_1},$$ where P_0 is the depth corrosion rate of the sample in solution without corrosion inhibitor, mm/year; P_1 is the depth corrosion rate of the sample in solution with corrosion inhibitor, mm/year.

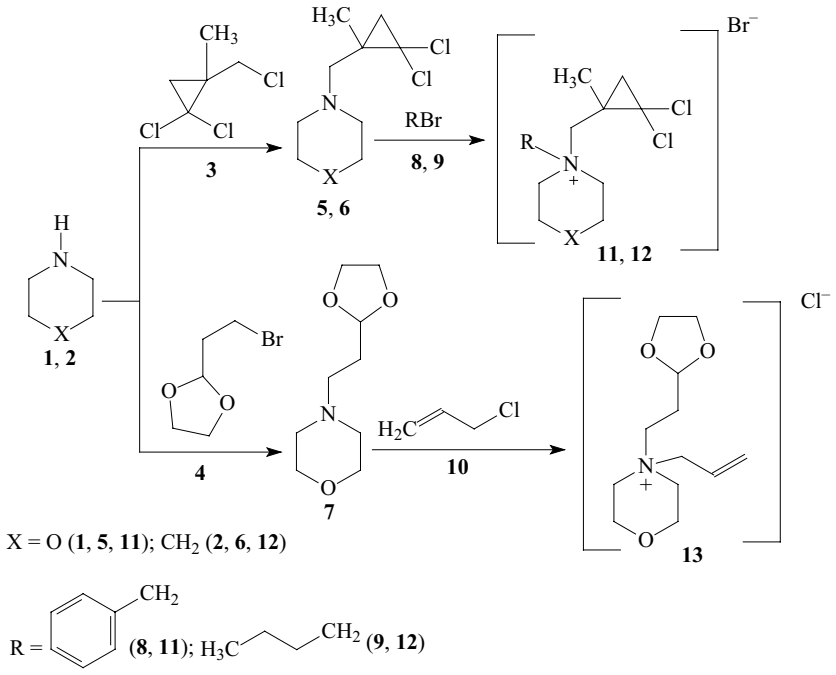
RESULTS AND DISCUSSION

Morpholine **1** and piperidine **2** were converted to the corresponding tertiary amines **5**–**7** by *N*-alkylation of 1,1-dichloro-2-(chloromethyl)-2-methylcyclopropane **3** and 2-β-bromoethyl-1,3-dioxolane **4**, which, upon action of benzyl bromide **8**, butyl **9**, or 3-chloro-propene-1 **10**, formed the corresponding quaternary ammonium salts **11**–**13** in quantitative yields (Scheme).

Under the selected conditions (100°C, 8–11 h), the yield of tertiary amines **5**–**7** was 90–95%. Dimethylformamide was used as a solvent since its use reduces the synthesis time by 2–4 times as compared to toluene without reducing the yield and selectivity of formation of target compounds **5**–**7**.

[1] GOST 380-2005. Interstate Standard. Common quality carbon steel. Grades. Moscow: Standartinform; 2007.

[2] Hydrogen sulfide water was prepared independently by mixing sodium sulfide and table salt solutions with hydrochloric acid. The concentration of hydrogen sulfide water was determined by the titration method.



Scheme. Obtaining target salts **11**–**13**

Quaternary ammonium salts containing acetal or *gem*-dichlorocyclopropane fragments **11**–**13** were obtained by condensation of tertiary amines **5**–**7** with an excess of halides **8**–**10** in 70–90% yield. MWI was successfully used for the synthesis of these QAS to prepare salts **11**–**13** at 30°C in 2–4 h in quantitative yields; conversely, thermal heating (40–100°C) required 6–8 h (Table 1).

Amines containing carbo- and heterocyclic fragments (acetal and cyclopropane groups) and the corresponding salts obtained on their basis are known to exhibit anticorrosion properties in acidic and hydrogen sulfide-containing media and can thus be effective inhibitors of corrosion of low-carbon and low-alloy steels [21]. While continuing to conduct studies in this field, we have investigated the anticorrosion activity of the obtained *tert*-amines **5**–**7** and salts **11**–**13** in hydrogen sulfide-containing medium, which is widespread in hydrocarbon production processes.

Table 1. Condensation of tertiary amines **5**–**7** with excess halides **8**–**10** and under the influence of thermal heating and microwave radiation **11**–**13**

Initial compounds		Reaction condition		Reaction product	Yield, %	Heating type
		T, °C	Reaction time, h			
5	**8**	100	7	**11**	80	Thermal
		40	3		95	MWI
6	**9**	90	8	**12**	70	Thermal
		60	4		92	MWI
7	**10**	40	6	**13**	90	Thermal
			2		98	MWI

Note: methyl isobutyl ketone solvent.

Table 2. Protection degree and inhibition coefficient of substances **5**–**7**, **11**–**13** in hydrogen sulfide-containing environment

Compound number	Corrosion, mm/year	Protection degree, %	Inhibition coefficient
5	0.78	23	1.31
6	0.29	71	3.51
7	0.23	78	4.47
11	0.18	82	5.58
12	0.11	89	9.23
13	0.08	91	11.61

Compound **13** showed the highest anticorrosion activity with a protection degree of 91% (inhibition coefficient = 11.61). Amines **5**–**7** showed protection efficiency in the range from 23 to 78% (inhibition coefficient = 1.31–4.47). We note that the degree of protection of salts **11**–**13** slightly exceeds the similar index (50%) of reagents used in oil production [22]. This may be assumed to be due to the presence of alkyl substituents in molecules **12** and **13**, leading to an increase in hydrophilicity and solubility in the corrosive environment, which in turn, according to [23], increases the inhibitory effect of organic compounds.

CONCLUSIONS

Thus, tertiary amines containing cycloacetal or *gem*-dichlorocyclopropane fragments were obtained under thermal heating conditions. Condensation of tertiary amines in an excess of halides using MWI allowed the synthesis of new quaternary ammonium salts in near quantitative yields. The anticorrosive activity was evaluated for the tertiary amines. 4-Allyl-4-[2-(1,3-dioxolan-2-yl)ethyl]morpholinium chloride is determined to exhibit anticorrosive properties in hydrogen sulfide environment, having a protection degree equal to 91%.

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Authors’ contributions

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

Sh.Sh. Dzhumaev—conducting research, reviewing publications on the topic of articles.

G.Z. Raskil’dina—collecting and processing material, statistical processing.

R.M. Sultanova—consultation on planning, methodology, and research implementation.

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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