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INVESTIGATION OF FUNGICIDAL PROPERTIES OF HEXYL BROMIDE COMPLEX OF N'-(2,2-BITCYCLO[2.2.1]HEPT-5-EN-2-YL-4,5-DIHYDRO-1-H-IMIDAZOLIN-1-YLETHYL)ETHANE-1,2-DIAMINE AS ANTIMICROBIAL ADDITIVE TO LUBRICANT-COOLANT FLUIDS

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Abstract: *The results of a study of the antifungal properties of an imidazoline hexyl bromide complex obtained on the basis of bicyclo[2.2.1]hept-5-ene-2-carboxylic acid and triethylenetetraamine as an additive to cutting fluids are presented. First, imidazoline was obtained by reacting norbornene carboxylic acid with triethylene tetraamine. In the next step, imidazoline was reacted with hexyl bromide to obtain its alkyl halide complex. The physicochemical properties of the resulting complex were determined, and its composition and structure were confirmed by IR spectroscopy. The complex has been tested as an antimicrobial additive in cutting fluids. It was found that the resulting complex has a destruction zone of 1.3-1.5 cm at a concentration of 0.25% and 2.4-2.6 cm at a concentration of 0.5%. N'-(2,2-bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine hexyl bromide complex as an antimicrobial additive exhibiting high fungicidal properties, destroys fungi even at a concentration of 0.25% - the affected area is 1.3-1.5 cm.*

Keywords: *bicyclo[2.2.1]hept-5-ene-2-carboxylic acid, triethylenetetraamine, imidazoline, additive, cutting fluids.*

Introduction

Lubricating-cooling fluids (LCFs) are fluids of various compositions used during the cutting or pressure processing of metals. The most common LCFs are petroleum oils and their 3-10% water emulsions. The main functions of LCFs are cooling and lubrication. LCF plays an important role in the protection of metal equipment used in cutting, pressing, rolling, drilling, polishing and other intensive processes. High voltage, high static and dynamic loads cause heating of equipment subject to deformation, which in turn leads to a decrease in the quality of processing, and damage to equipment and tools. The application of LCFs allows for the reduction of the temperature in the processing zone to a suitable level due to heat exchange and more steam generation. The presence of lubricating properties in LCFs significantly reduces the possibility of friction in the machining zone, tool wear, scraping, and damage to the surfaces of the machined parts and tools. In general, the use of LCFs makes it possible to increase the intensity of technological processes and the productivity of equipment. Antimicrobial additives are added to LCFs to improve their quality. When additives such as antimicrobial additives are used, factors such as toxicity, environmental efficiency and cost of these additives must be considered.

Currently, there is a greater demand for additives to lubricating-cooling fluids. The most effective way to protect the equipment and tools used in the processing process from damage is the chemical method, in which biocide and fungicidal additives are added to the lubricating-coolant fluids. Additives not only stop the development of living cells, but also completely destroy them. Their correct use creates conditions for increasing the intensity of technological processes and the productivity of equipment [1, 2]. Since microorganisms are more easily adapted to the environment, the inclusion of fungicidal additives in lubricating and cooling fluids is a very urgent issue [3].

The development of convenient synthesis methods for the practical application of S- and N-retaining additives with biocide properties, determining the effect of structural parameters on reactivity and the effectiveness of additives in lubricants, as well as researching new areas of application are of great importance [4-7].

The basis of the problem of improving the operational properties of oil products is the application of additives with different functional groups. Nitrogen and sulfur-containing compounds occupy a special place among multifunctional additives used to improve the quality of petroleum products. In addition to having a high effect against oxidation, eating, peeling, and corrosion, these types of additives are also applied as biocidal compounds [8].

Currently, imidazolines are widely used as corrosion inhibitors, surface-active agents, as well as fuel and oil additives in various fields of industry [9]. Thus, an imidazoline-based detergent additive obtained from the reaction of carbonic acid with polyamine with a suitable composition for reducing deposits in internal combustion engines, fuels or lubricants of internal combustion engines was synthesized. In addition, imidazoline has been combined with sulfonates to obtain an improved detergent for cleaning fuels [10].

Ethoxylated alkylimidazolines with an appropriate degree of ethoxylation can be used as antioxidants. These ethoxylated imidazolines are more effective than other antioxidants in hydrocarbon media [11].

In order to obtain a new type of antimicrobial additive, 2-allyoxy-3-(aminomethoxy)bicycloheptane compound was synthesized and applied as an antimicrobial additive in lubricant-coolant fluids [12].

Employees of the Additives Chemistry Institute have also conducted extensive research in the direction of improving the quality of lubricants and coolants [13-16].

This study is aimed at the synthesis of imidazoline (NTI) complex with hexylbromide (NTI+$C_6H_{13}Br$) based on bicyclo[2.2.1]hept-5-ene-2-carboxylic acid and triethylenetetraamine (TETA), a new compound with the above-mentioned properties, as an antimicrobial additive, and study of its properties.

Experimental part

Physico-chemical parameters of synthesized bicyclo[2.2.1]hept-5-ene-2-carboxylic acid, imidazoline and its inorganic anionic complex were determined. The IR spectra of the obtained compounds were recorded on diamond crystals at a wavelength of 400-4000 cm^{-1} in "BRUKER" brand spectrometers belonging to the German company "ALPHA IR FOURYE". The refractive index of the compounds (n_D^{20}) was determined in the "ABBEMAT" 350/500 brand refractometer, and the density (ρ) was determined in the "ДМА" 4500 M device. The specific resistance and specific electrical conductivity of the obtained substances were determined. The specific resistance was determined using the E6-13A Theraohmmeter device.

N'-(2,2-Bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1yl-ethyl)ethane-1,2-diamine hexyl bromide complex antimicrobial tested as an additive in lubricant-coolant liquids at concentrations of 0.25 and 0.5% [17]. The study of the complex as an antimicrobial additive in lubricants and coolants was conducted at the Institute of Chemical Additives named after Ali Musa oglu Guliyev. The antimicrobial properties of the investigated complex were determined by the zonal diffusion method based on GOST 9.052-88. The following laboratory strains were used as test cultures:

Fungi: *Aspergillus niger, Cladosporium resiane, Penicillium chrysogenum, Trichoderma viride.*

Malt water agar (SA) medium was selected for the cultivation of fungi.

Inoculation of microorganisms was carried out on the surface of the nutrient medium. The studied sample was added to the lubricant-coolant liquids by mass percentages. Laboratory tests were performed as follows: 20-25 ml of nutrient medium was filtered into a Petri dish, and after it cooled down a little, microorganisms were spread on the surface of the nutrient medium. In order to determine the diameter of the destruction zone of microorganisms, 4-5 mm deep grooves with a diameter of 10 mm were opened on the surface of the medium, and 0.3-0.5 ml of a percentage solution of the lubricant-cooling liquid without biocide and with biocide was added to these grooves. The Petri dish was placed in a thermostat and kept at 29 ± 2°C for 3-4 days for the development of fungi.

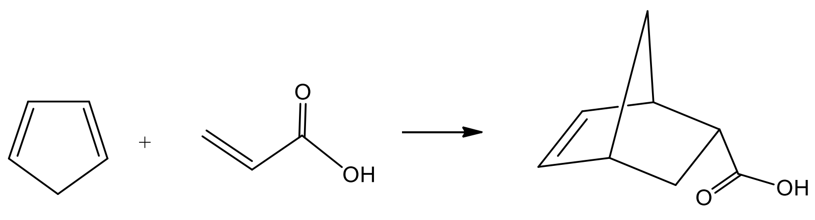
The effectiveness of the studied samples as an additive against microorganisms is determined by the size of the diameter (in cm) of the destruction zone of microorganisms (with or without additives): the larger the diameter of the destruction zone, the higher the antimicrobial property of this additive is considered.

The physico-chemical parameters of the initial substances used during the experiment are listed in **Table 1**.

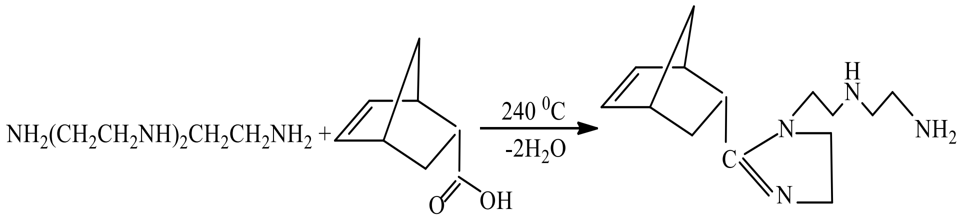
Table 1. Physico-chemical parameters of primary substances

Substances name	**Density, (ρ_4^{20}) g/cm^3**	**Refraction index, (n_D^{20})**	**Boiling temperature , °C**
Dicyclopentadiene	0.973	1.5110	166
Cyclopentadiene	0.802	1.4400	41
Acrylic acid	1.051	1.4224	141
5-Norbornene-2-carboxylic acid	1.129	1.4941	136-138 (10 mm/ Hg.)
Triethylenetetraamine	0.9810	1.496	276.5
Hexyl bromide	1.176	1.448	154-158

At the initial stage, bicyclo[2.2.1]hept-5-ene-2-carboxylic acid (NKT) was obtained. The purchase of acid was carried out in two stages. In the first step, cyclopentadiene was synthesized by monomerization of dicyclopentadiene. Dicyclopentadiene In the "EP-300" unit in Sumgayit city, valuable connected dienes (dicyclopentadiene, isoprene, piperylene) are obtained in the C_5 fraction of liquid pyrolysis products, which are chemically highly reactive, received as a multi-tonnage by-product [18]. Monomerization was carried out by heating dicyclopentadiene over iron scraps at a temperature of 140-150 °C. Bicyclo[2.2.1]hept-5-ene-2-carboxylic acid was obtained based on the reaction of cyclopentadiene obtained in the second step with acrylic acid. For this purpose, the required amount of acrylic acid benzene solution is first placed in a 3-necked flask. A benzene solution of cyclopentadiene was then added dropwise. The yield of NKT obtained during the process was 94%. The reaction scheme is below:



Later, imidazoline (NTI) was synthesized based on bicyclo[2.2.1]hept-5-ene-2-carboxylic acid and triethylenetetraamine (TETA). To synthesize NTI, 0.5 mol of bicyclo[2.2.1]hept-5-ene-2-carbonic acid is poured into the flask and heated to 80-100ºC with stirring. At this temperature, 0.5 mol of amine (TETA) is gradually added to the acid through a drop funnel. During the synthesis of imidazoline, the temperature is raised to 240°C and the reaction is continued with intensive stirring for 3-3.5 hours. The yield of obtained imidazoline was 84.28%. The N'-(2,2-Bicyclo[2.2.1]hept-5-ene-2-yl-4)-5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine is obtained from the interaction of bicyclo[2.2.1]hept-5-ene-2-carboxylic acid with triethylenetetraamine in a 1:1 ratio at 240°C [19]. The synthesis of imidazoline based on NKT and TETA was carried out according to the following scheme:



The IR spectrum of imidazoline (NTI) synthesized on the basis of NKT and TETA is shown in **Figure 1** and has the following absorption bands:

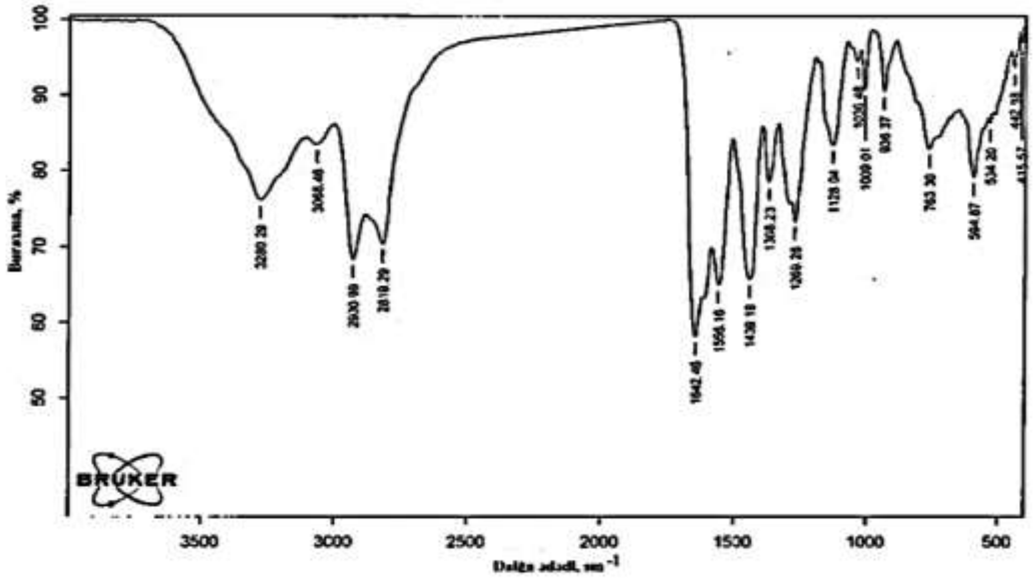
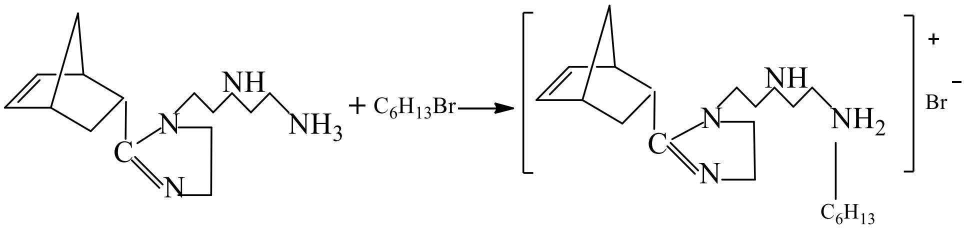


Fig. 1. IR-spectra of N'-(2,2-Bitsiklo[2.2.1]hept-5-en-2-il-4,5-dihidro-1-H-imidazolin-1-iletil)etane-1,2-diamine compound

The IR spectrum of NTI: absorption bands at 1556 and 1642 cm^{-1} belong to the C=N, N-H and NH_2 groups. The absorption maxima at 1036 and 1128 cm^{-1} reflect the valence vibration of the C-N bond of the triple amine. The absorption band at 1269 cm^{-1} belong to the valence vibration of the C=N bond. A peak at 3280 cm^{-1} reflects the valence vibration of the N-H bond of single and double amine. Absorption bands at 1368, 1439, 2819, and 2930 cm^{-1} correspond to deformation and valence oscillations of C-H bond of CH and CH_2 groups. The absorption band at 3068 cm^{-1} demostrates the valence vibration of the C-H bond of the =CH group. The absorption band characteristic of the C=C bond of the C=CH group of an unsaturated hydrocarbon coincides with the absorption bands of nitrogen compounds at 1642 cm^{-1}.

At the last stage, 5.2 grams of imidazoline was dissolved in 25 ml of isopropyl alcohol (GOST 9805-84), poured into a flask, and 8.3 grams of hexyl bromide were added to it. The reaction was carried out for 2.5-3 hours at a temperature of 50-60°C with continuous stirring. As a result of the reaction, the yield according to the reagents was 92% [20]. The synthesis of the complex was carried out according to the following scheme:



The obtained N'-(2,2-bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-yl-ethyl)ethane-1,2-diamine hexyl bromide complex physico-chemical indicators were determined and the obtained results are listed in **Tables 2** and **3**, respectively:

Table 2. N'-(2,2-Bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine hexyl bromide complex physical and chemical properties

Compound and its code name	Agregate state	Color	Density, (ρ_4^{20}) q/cm^3	Refractive index (n_D^{20})
Solution of hexyl bromide complex of N'-(2,2-Bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine in izopropil alcohol	Liquid	Brown	0.9816	1.4334

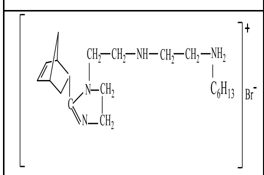
Table 3. Specific resistance and specific electrical conductivity of substances

Examples	Specific resistance, ρ, Ohm·m	Specific electrical conductivity, σ, S/cm
NKT	$4.95\cdot10^{7}$	$2.02\cdot10^{-8}$
NTI	$1.8\cdot10^{5}$	$5.6\cdot10^{-6}$
NTI+$C_6H_{13}Br$	$2.6\cdot10^{2}$	$4\cdot10^{-3}$

As can be seen from **Table 3**, the specific resistance of NTI ($1.8•10^5$ Ohm•m) is smaller than the specific resistance of NKT ($4.95•10^7$ Ohm•m). As a result, it can be seen that the specific electrical conductivity of NTI was higher than that of NKT. The specific resistance of the hexylbromide complex of NTI is low and is ($r=2.6•10^2$ Ohm•m). Accordingly, the hexylbromide complex has a high specific electrical conductivity.

Table 4 represents elemental composition of the obtained complex.

Table 4. Elemental composition of the obtained complex

Chemical formula	Brutto formula	Element composition, %								Mol wt.
		Calculated				Founded				
		C	H	N	Br	C	H	N	Br	
	$C_{20}H_{37}N_4Br$	58.11	8.95	13.55	19.37	57.83	8.79	12.92	19.43	413

The structure and composition of the synthesized complex was confirmed by IR spectroscopy (**Figure 2**).

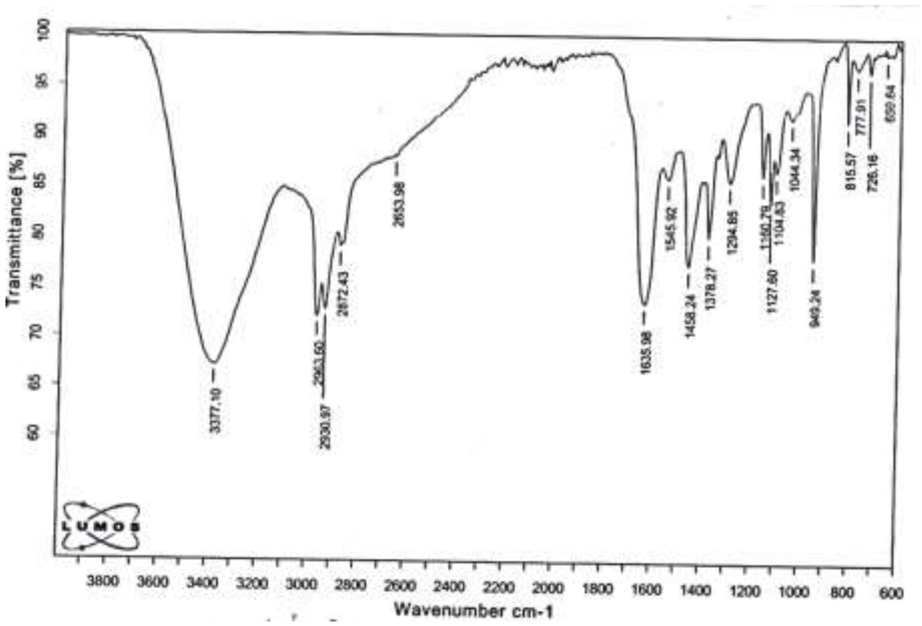


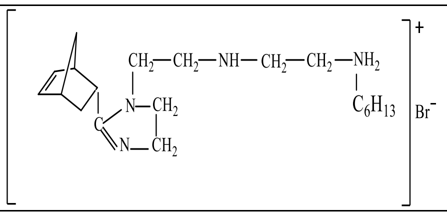
Fig. 2. IR-spectra of hexyl bromide complex of N'-(2,2-Bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine

Description of IR spectrum of the NTI+$C_6H_{13}Br$ complex: absorption band at 815 cm^{-1} corresponds to the deformation vibration of the N-H bond; at 3377 cm^{-1} is the valence vibration of the N-H bond. Absorption band 949 cm^{-1} is the C–N bond in the cyclic ring, at 1127 cm^{-1} is the valence vibration of the C–N bond. A peak at 1294 cm^{-1} is the C–N bond. Absorption bands at 1378, 1458, 2872, 2930, and 2963 cm^{-1} are deformation and valence vibrations of C-H bond of CH_3 and CH_2 groups. Ones at 1545, 1635 cm^{-1} are C-N bonds. Absorption band at 2653 cm^{-1} characterizes the "Ammonium band".

Results and Discussion

The fungicidal properties of the synthesized hexylbromide complex as an antimicrobial additive were determined. The results of the tests and the activity index of the studied substances are shown in **Table 5**.

Table 5. Determination of antimicrobial properties of hexylbromide complex of N'-(2,2-Bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine in water-based LCFs

Compound formula	**Concentration of biocide, %**	**Zone of destruction of microorganisms (cm)**
		Fungi (*Aspergillus niger, Cladosporium resiane, Penicillium chrysogenum, Trichoderma viride*)
	*0.5*	*2.4-2.6*
	0.25	*1.3-1.5*
Control – water based LCF	**-**	+++

**Note: (+++) – development of microorganisms.*

The obtained complex was tested in a comparative form with lubricating-cooling fluids taken as a benchmark. Based on the test results, it can be noted that the studied compound has a high antifungal properties. While the LCF sample taken under the same conditions developed fungi, the growth of fungi stopped in the sample treated with the investigated additive. Thus, the synthesized complex destroyed fungi by showing a destruction zone of 1.3-1.5 cm in 0.25% concentration, and 2.4-2.6 cm in 0.5% concentration. N'-(2,2-bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine hexyl bromide complex as an antimicrobial additive showing a high fungicidal property, even at a concentration of 0.25% - it showed a destruction zone of 1.3-1.5 cm. The presence of bicycloheptene fragments, amino groups and halogen ions in the composition of the compounds further increases the effectiveness of this property [21].

Conclusion

1. Synthesis of imidazoline was carried out based on bicyclo[2.2.1]hept-5-ene-2-carboxylic acid and triethylenetetramine. The hexylbromide complex of the synthesized compound was obtained, its physicochemical properties were determined, composition and structure were confirmed by infrared spectroscopic method.
2. The fungicidal properties of the received alkylhalide complex as an antimicrobial additive in lubricating-coolant fluids were tested. N'-(2,2-Bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-H-imidazolin-1-ylethyl)ethane-1,2-diamine hexyl bromide complex as an antimicrobial additive showing high fungicidal properties, it destroyed fungi by showing a destruction zone of 1.3-1.5 cm at 0.25% concentration and 2.4-2.6 cm at 0.5% concentration. Based on the results of the research, it can be said that the synthesized bicyclo[2.2.1]hept-5-ene-2-carboxylic acid and triethylenetetramine-based imidazoline hexylbromide complex shows high results in lubricating-coolant fluids and can be recommended as a fungicidal additive.

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N'-(2,2-BİTSİKLO[2.2.1]HEPT-5-EN-2-İL-4,5-DİHİDRO-1-H-İMİDAZOLİN-1İLETİL)ETAN-1,2-DİAMİNİN HEKSİLBROMİD KOMPLEKSİNİN YAĞLAYICI-SOYUDUCU MAYELƏRƏ ANTİMİKROB AŞQAR KİMİ FUNQİSİD XASSƏLƏRİNİN TƏDQİQİ

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Xülasə: Məqalədə bitsiklo[2.2.1]hept-5-en-2-karbon turşusu və trietilentetraamin əsasında alınmış imidazolinin heksilbromid kompleksinin yağlayıcı-soyuducu mayelərdə aşqar kimi antifungisid xassələrinin nəticələri göstərilmişdir. İlk əvvəl norbornen karbon turşusunun trietilentetraaminlə qarşılıqlı təsirindən imidazolin alınmışdır. Sonrakı mərhələdə imidazolinin heksilbromidlə

reaksiyasından onun alkilhalogendli kompleksi alınmışdır. Alınan kompleksin fiziki-kimyəvi xassələri təyin edilmiş, tərkib və quruluşu İQ spektroskopik üsulla təsdiq edilmişdir. Kompleks yağlayıcı-soyuducu mayelərdə antimikrob aşqar kimi sınaqdan keçirilmişdir. Müəyyən edilmişdir ki, alınmış kompleks 0.25% qatılıqda – 1.3-1.5 sm, 0.5% qatılıqda – 2.4-2.6 sm məhvolma zonası göstərir. N'-(2,2-bitsiklo[2.2.1]hept-5-en-2-il-4,5-dihidro-1-H-imidazolin-1iletil)etan-1,2-diaminin heksilbromid kompleksi antimikrob aşqar kimi yüksək fungisid xassə göstərməklə, hətta 0.25% qatılıqda – 1.3-1.5 sm məhvolma zonası göstərməklə göbələkləri məhv edir.
Açar sözlər: bitsiklo[2.2.1]hept-5-en-2-karbon turşusu, trietilentetraamin, imidazolin, aşqar, yağlayıcı-soyuducu mayelər

ИССЛЕДОВАНИЕ ФУНГИЦИДНЫХ СВОЙСТВ ГЕКСИЛБРОМИДНОГО КОМПЛЕКСА N'-(2,2-БИТЦИКЛ[2.2.1]ГЕПТ-5-ЕН-2-ИЛ-4,5-ДИГИДРО-1-Н-ИМИДАЗОЛИН-1ИЛЭТИЛ)ЭТАН-1,2-ДИАМИНА КАК АНТИМИКРОБНОЙ ДОБАВКИ ДЛЯ СМАЗОЧНО-ОХЛАЖДАЮЩИХ ЖИДКОСТЕЙ

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Аннотация: Представлены результаты изучения противогрибковых свойств имидазолингексилбромидного комплекса, полученного на основе бицикло[2.2.1]гепт-5-ен-2-карбоновой кислоты и триэтилентетрамина в качестве добавки в смазочно-охлаждающие жидкости. Впервые имидазолин был получен взаимодействием норборненугольной кислоты с триэтилентетраамином. На следующем этапе имидазолин подвергали взаимодействию с гексилбромидом с получением его алкилгалогенидного комплекса. Определены физико-химические свойства полученного комплекса, а также подтверждены его состав и структура методом ИК-спектроскопии. Комплекс апробирован в качестве антимикробной добавки в смазочно-охлаждающие жидкости. Установлено, что полученный комплекс имеет зону деструкции 1,3-1,5 см при концентрации 0,25% и 2,4-2,6 см при концентрации 0,5%. N'-(2,2-бицикло[2.2.1]гепт-5-ен-2-ил-4,5-дигидро-1-Н-имидазолин-1-илэтил)этан-1,2-диамин гексилбромидный комплекс как антимикробная добавка, проявляющая высокие фунгицидные свойства, уничтожает грибы даже при концентрации 0,25% - зона поражения 1,3-1,5 см.
Ключевые слова: бицикло[2.2.1]гепт-5-ен-2-карбоновая кислота, триэтилентетрамин, имидазолин, присадка, смазочно-охлаждающие жидкости.