

**ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)**

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ГЫЛЫМ АКАДЕМИЯСЫНЫҢ

Д.В.Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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NEWS

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN
JSC «D.V. Sokolsky institute of fuel, catalysis
and electrochemistry»

**SERIES
CHEMISTRY AND TECHNOLOGY**

4 (436)

JULY-AUGUST 2019

PUBLISHED SINCE JANUARY 1947

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруды. Web of Science зерттеушілер, авторлар, баспашилар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енүі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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ISSN 2518-1491 (Online),

ISSN 2224-5286 (Print)

Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» Республикалық қоғамдық бірлестігі (Алматы қ.)

Қазақстан республикасының Мәдениет пен ақпарат министрлігінің Ақпарат және мұрағат комитетінде 30.04.2010 ж. берілген №1089-Ж мерзімдік басылым тіркеуіне қойылу туралы куәлік

Мерзімділігі: жылына 6 рет.

Тиражы: 300 дана.

Редакцияның мекенжайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., 220, тел.: 272-13-19, 272-13-18,
<http://chemistry-technology.kz/index.php/en/archiv>

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Типографияның мекенжайы: «Аруна» ЖК, Алматы қ., Муратбаева көш., 75.

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«Известия НАН РК. Серия химии и технологии».

ISSN 2518-1491 (Online),

ISSN 2224-5286 (Print)

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы)

Свидетельство о постановке на учет периодического печатного издания в Комитете информации и архивов Министерства культуры и информации Республики Казахстан №10893-Ж, выданное 30.04.2010 г.

Периодичность: 6 раз в год

Тираж: 300 экземпляров

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, ком. 219, 220, тел. 272-13-19, 272-13-18,

<http://chemistry-technology.kz/index.php/en/arhiv>

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News of the National Academy of Sciences of the Republic of Kazakhstan. Series of chemistry and technology.
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty)
The certificate of registration of a periodic printed publication in the Committee of Information and Archives of the Ministry of Culture and Information of the Republic of Kazakhstan N 10893-Ж, issued 30.04.2010

Periodicity: 6 times a year

Circulation: 300 copies

Editorial address: 28, Shevchenko str., of. 219, 220, Almaty, 050010, tel. 272-13-19, 272-13-18,
<http://chemistry-technology.kz/index.php/en/arhiv>

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Editorial address: Institute of Organic Catalysis and Electrochemistry named after D. V. Sokolsky
142, Kunayev str., of. 310, Almaty, 050100, tel. 291-62-80, fax 291-57-22,
e-mail: orgcat@nursat.kz

Address of printing house: ST "Aruna", 75, Muratbayev str, Almaty

N E W S

OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES CHEMISTRY AND TECHNOLOGY

ISSN 2224-5286

<https://doi.org/10.32014/2019.2518-1491.41>

Volume 4, Number 436 (2019), 43 – 48

UDK 547.37:632.954

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SYNTHESIS AND MICROBIOLOGICAL EVALUATION OF ACETYLENIC AMINO ALCOHOLS N-PHENYL CARBAMATE DERIVATIVES

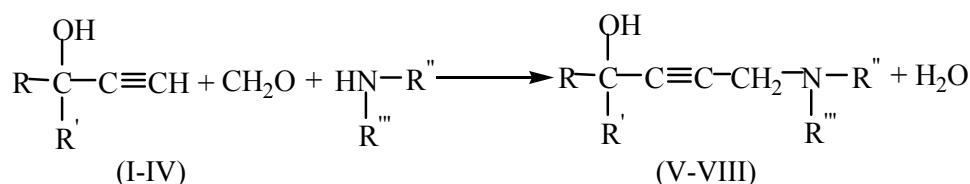
Abstract. The paper presents the results of synthesizing of acetylenic amino alcohols N-phenyl carbamate derivatives and the microbiological evaluation of the latter has been studied. The following microorganisms were considered as objects of microbiological research: *Bacillus subtilis*, *Botrytis cinerea*, *Echerichia coli*, *Erwinia caratovorum*, *Candida albicans*, *Fusarium solani* u *Helminthosporium*. There were researched physicochemical and bactericidal properties of acetylenic amino alcohols N-phenyl carbamate derivatives. The output of carbamates depends on the content and structure of acetylenic amino alcohols and is 68-88%. The purity, identification and structure of the compounds obtained have been determined by thin-layer and gas-liquid chromatography, elemental analysis, IR (Infrared) and PMR (Proton Magnetic Resonance) spectroscopy. It was established that these compounds exhibit high antimicrobial activity against the causative agents of certain animal and plant diseases. The relatively high microbiological activity of the phenylcarbamates studied is associated with the presence of various functional groups in the composition of the molecules.

Key words: carbamates, acetylene amino alcohols, bactericidal properties, microorganisms, microbiological activity.

Introduction. Obviously, the search for biologically active compounds is an actual problem of organic chemistry, biological chemistry and pharmacology. Because the efficiency and environmental safety of currently used compounds do not fully meet modern requirements [1-3].

Continuing our work in this direction [4,5], we synthesized a number of acetylenic amino alcohols, obtained N-phenyl carbamate derivatives and studied the antimicrobial activity of the latter.

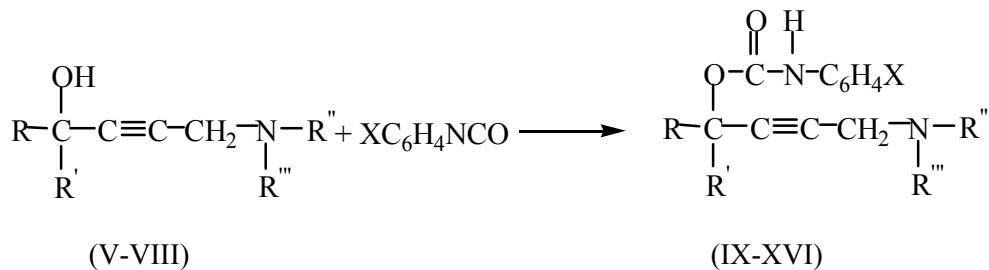
Methods. Acetylenic alcohols were synthesized by the well-known Favorsky reaction [6] and obtained a series of amino acids by aminomethylation with dibenzylamine under the reaction conditions of Mannich [7] in the presence of formaldehyde:



where:

$R = R' = -CH_3, R'' = -CH_2C_6H_5$	(I, V)
$R = -CH_3, R' = -C_2H_5, R'' = -CH_2C_6H_5$	(II, VI)
$R = -CH_3, R' = -C_3H_7-H, R'' = -CH_2C_6H_5$	(III, VII)
$R + R' = -(CH_2)_5-, R'' = -CH_2C_6H_5$	(IV, VIII)

The obtained amino acids reacted with phenyl and p-chlorophenyl isocyanate for 3-5 hours in an environment of benzene (between 70-80°C) or in an environment of acetone (boiling point). The reaction proceeds as follows:



where:

$R = R' = -CH_3, R'' = -CH_2C_6H_5, X = -H$	(V, IX)
$R = -CH_3, R' = -C_2H_5, R'' = -CH_2C_6H_5, X = -H$	(VI, X)
$R = -CH_3, R' = -C_3H_7-H, R'' = -CH_2C_6H_5, X = -H$	(VII, XI)
$R + R' = -(CH_2)_5-, R'' = -CH_2C_6H_5, X = -H$	(VIII, XII)
$R = R' = -CH_3, R'' = -CH_2C_6H_5, X = -Cl-n$	(V, XIII)
$R = -CH_3, R' = -C_2H_5, R'' = -CH_2C_6H_5, X = -Cl-n$	(VI, XIV)
$R = -CH_3, R' = -C_3H_7-H, R'' = -CH_2C_6H_5, X = -Cl-n$	(VII, XV)
$R + R' = -(CH_2)_5-, R'' = -CH_2C_6H_5, X = -Cl-n$	(VIII, XVI)

The yield of carbamates varied from 68 to 88%, depending on the structure and composition of acetylenic amino alcohols. The purity, individuality and structure of the obtained compounds were determined by thin layer and gas chromatography, as well as by elemental analysis using IR and PMR spectra [8,9]. Synthesized carbamates are white needle-like crystals that dissolve in polar organic solvents.

Results and discussions. The main lines and physicochemical characteristics of the IR and PMR spectra of new carbamates are given in Tables 1 and 2.

As can be seen from Table 1, acetylenic amino acids are not absorbed by the hydroxyl group in the IR spectra (in the range of $\sim 3600 \text{ cm}^{-1}$), but in the carbonyl group (in the range of 1730 cm^{-1}) and absorption bands on the aromatic rings.

Also, acetylenic amino acids are clearly visible in the absorption bands (IR spectra) and chemical shift lines (PMR spectra) characteristic of functional groups and compounds that determine the composition of carbamates.

White or light yellow crystals, dissolved in many organic solvents, have a weak characteristic of these compounds (Tables 2,3). It is known that the presence of several functional groups in a molecule can give the molecule exceptional properties and increase its biological activity. From this point of view, it was particularly interesting to determine the microbiological activity of carbamates from acetylenic amino alcohols, taking into account the biological, in particular, testing ease and low cost.

To detect microbiological activity, we investigated a number of new carbamates and thiocarbamates using certain methods [10-12]. From the results it can be seen that almost all investigated compounds showed a certain degree of microbiological activity.

For example, were obtained the result of the antimicrobial activity of 1-cyclohexyl-3-diethylaminoprop-3-yl-1-yl ester is given under the name AA-008 - N-phenylcarbamic acid and -2-methyl-5-dibenzylaminopentin-3-yl-2-phenylcarbamate, conditionally called AA-007 - N-phenylcarbamic acid.

For conducting microbiological tests, the obtained 1% solution of AA-007 and AA-008 preparations in ethyl alcohol (1:1 ratio of ethyl alcohol and water) were used, using serial dilution of solutions of 0.05% to 0.0001%.

Table 1 - IR and PMR spectra of carbamates from acetylenic amino alcohols

Number of compounds	IR spectra (KBr), ν , cm^{-1}	PMR spectra, (CDCl_3), δ , ppm
(IX)	2190 (C≡C); 3300 (N-H); 1180 (C—O—C); 1755 (C=O); 2920 (-CH ₂ -); 1580 (-C ₆ H ₅)	0,91 t 3H(CH ₃); 1,21 t 3H(CH ₃); 1,37 m 2H(CH ₂); 2,57 t (C-N); 2,60-2,71 m (cycle H); 3,52 t (C-O)
(X)	2195 (C≡C); 3310 (N-H); 1170 (C—O—C); 1745 (C=O); 2930 (-CH ₂ -); 1585 (-C ₆ H ₅)	0,93 t 3H(CH ₃); 1,20 t 3H(CH ₃); 1,37 m 2H(CH ₂); 2,58 t (C-N); 2,60-2,71 m (cycle H); 3,53 t (C-O)
(XI)	2195 (C≡C); 3320 (N-H); 1190 (C—O—C); 1750 (C=O); 2940 (-CH ₂ -); 1583 (-C ₆ H ₅)	0,90 t 3H(CH ₃); 1,19 t 3H(CH ₃); 1,35 m 2H(CH ₂); 2,59 t (C-N); 2,60-2,70 m (cycle H); 3,55 t (C-O)
(XII)	2198 (C≡C); 3315 (N-H); 1180 (C—O—C); 1753 (C=O); 2880 (-CH ₂ -); 1575 (-C ₆ H ₅)	0,92 t 3H(CH ₃); 1,22 t 3H(CH ₃); 1,38 m 2H(CH ₂); 2,58 t (C-N); 2,61-2,71 t (cycle H); 3,55 t (C-O)
(XIII)	2200 (C≡C); 3310 (N-H); 1185 (C—O—C); 1745 (C=O); 2890 (-CH ₂ -); 1580 (-C ₆ H ₅)	0,93 t 3H(CH ₃); 1,19 t 3H(CH ₃); 1,37 m 2H(CH ₂); 2,60 t (C-N); 2,61-2,72 m (cycle H); 3,49 t (C-O)
(XIV)	2195 (C≡C); 3313 (N-H); 1190 (C—O—C); 1740 (C=O); 2890 (-CH ₂ -); 1575 (-C ₆ H ₅)	0,91 t 3H(CH ₃); 1,21 t 3H(CH ₃); 1,38 m 2H(CH ₂); 2,59 t (C-N); 2,60-2,71 m (cycle H); 3,54 t (C-O)
(XV)	2205 (C≡C); 3315 (N-H); 1195 (C—O—C); 1742 (C=O); 2910 (-CH ₂ -); 1570 (-C ₆ H ₅)	0,92 t 3H(CH ₃); 1,21 t 3H(CH ₃); 1,36 m 2H(CH ₂); 2,56 t (C-N); 2,60-2,70 t (cycle H); 3,55 t (C-O)
(XVI)	2201 (C≡C); 3320 (N-H); 1185 (C—O—C); 1740 (C=O); 2905 (-CH ₂ -); 1570 (-C ₆ H ₅)	0,91 t 3H(CH ₃); 1,20 t 3H(CH ₃); 1,38 m 2H(CH ₂); 2,57 t (C-N); 2,60-2,71 m (cycle H); 3,53 t (C-O)

Table 2 - Some physicochemical characteristics of carbamates from acetylenic amino alcohols

Number of compounds	Formula	Yield, %	t^o melting, °C	R_f
IX	$\begin{array}{c} \text{OCONHC}_6\text{H}_5 \\ \\ (\text{CH}_3)_2\text{C}-\text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	61	101-102	0,63
X	$\begin{array}{c} \text{OCONHC}_6\text{H}_5 \\ \\ \text{C}_2\text{H}_5(\text{CH}_3)\text{C}-\text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	64	107-108	0,54
XI	$\begin{array}{c} \text{OCONHC}_6\text{H}_5 \\ \\ \tilde{\text{N}}_3\text{H}_7(\text{CH}_3)\text{C}-\text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	58	123-124	0,51
XII	$\begin{array}{c} \text{H}_5\text{C}_6\text{NHOCO} \diagup \quad \diagdown \\ \text{C}_6\text{H}_11 \quad \text{C}_6\text{H}_11 \\ \text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	52	135-136	0,39
XIII	$\begin{array}{c} \text{OCONHC}_6\text{H}_4\text{Cl}-n \\ \\ (\text{CH}_3)_2\text{C}-\text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	53	156-157	0,43
XIV	$\begin{array}{c} \text{OCONHC}_6\text{H}_4\text{Cl}-n \\ \\ \text{C}_2\text{H}_5(\text{CH}_3)\text{C}-\text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	49	163-164	0,38
XV	$\begin{array}{c} \text{OCONHC}_6\text{H}_4\text{Cl}-n \\ \\ \tilde{\text{N}}_3\text{H}_7(\text{CH}_3)\text{C}-\text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	47	175-176	0,33
XVI	$\begin{array}{c} n\text{-ClH}_4\text{C}_6\text{NHOCO} \diagup \quad \diagdown \\ \text{C}_6\text{H}_11 \quad \text{C}_6\text{H}_11 \\ \text{C}\equiv\text{C}-\text{CH}_2-\text{N}(\text{CH}_2\text{C}_6\text{H}_5)_2 \end{array}$	43	201-202	0,26

Table 3 - Elemental analysis of carbamates from acetylenic amino alcohols

Number of compounds	discovered, %			Gross formulas	calculated, %		
	C	H	N		C	H	N
IX	78,60	7,03	6,81	$C_{27}H_{28}N_2O_2$	78,64	6,79	6,79
X	78,94	7,12	6,68	$C_{28}H_{30}N_2O_2$	78,87	7,04	6,57
XI	79,31	7,37	6,49	$C_{29}H_{32}N_2O_2$	79,09	7,27	6,36
XII	79,76	7,21	6,30	$C_{30}H_{32}N_2O_2$	79,65	7,08	6,19
XIII	76,08	6,46	6,71	$C_{27}H_{27}N_2O_2Cl$	75,97	6,33	6,56
XIV	72,98	6,37	6,26	$C_{28}H_{29}N_2O_2Cl$	72,96	6,29	6,08
XV	73,40	6,58	6,08	$C_{29}H_{31}N_2O_2Cl$	73,34	6,53	5,90
XVI	74,09	6,42	5,86	$C_{30}H_{31}N_2O_2Cl$	73,99	6,37	5,75

The objects of the microbiological test are microorganisms *Bacillus subtilis*; *Botrytis cinerea*, *Echerchia coli*, *Ervinia caratovorum*, *Candida albicans*, *Fusarum solani* и *Helminthosporium*.

The test results showed that all studied compounds have a certain antimicrobial activity, which inhibits or even aggravates their growth. The results of microbiological testing of drugs AA-007, AA-008 are presented in Table 4.

Table 4 - Results of microbiological testing of synthesized carbamates

A drug	Test microorganisms	Drug concentration % (mass.)*				
		0,0001	0,001	0,005	0,01	0,05
AA-007	<i>Bacillus subtilis</i>	-	-	±	+	++
	<i>Botrytis cinerea</i>	-	-	+	+	++
	<i>Candida albicans</i>	-	±	+	+	++
	<i>Echerchia coli</i>	-	-	+	+	++
	<i>Ervinia caratovorum</i>	-	±	+	+	++
	<i>Fusarum solani</i>	±	±	+	+	++
	<i>Helminthosporium</i>	±	+	+	++	++
AA-008	<i>Bacillus subtilis</i>	-	-	±	+	++
	<i>Botrytis cinerea</i>	±	+	+	++	++
	<i>Candida albicans</i>	±	+	+	+	++
	<i>Echerchia coli</i>	±	+	+	+	++
	<i>Ervinia caratovorum</i>	±	±	+	+	++
	<i>Fusarum solani</i>	±	+	+	++	++
	<i>Helminthosporium</i>	±	+	+	++	++

* - (-) – in this case, do not show biological activity; (±) - the zone for the removal of microorganisms does not exceed 5 mm; (+) - zone for the removal of microorganisms above 5 mm; (++) - zone for the removal of microorganisms above 10 mm

As can be seen from table 4, any of the phenylcarbamates exhibits significant bactericidal activity against microorganisms at significantly lower concentrations.

At the same time, it is clear that the antimicrobial properties of these compounds definitely depend on their composition. For example, the bactericidal activity of the drug AA-008 is significantly higher than AA-007.

In our opinion, this difference is due to the fact that the drug molecule AA-008 contains an alcohol fragment containing a ring group as a hexyl radical, since the other components of both drugs are the same.

Conclusion. The relatively high microbiological activity of the phenylcarbamate molecules under study may be due to the presence of a set of active functional groups, for example, if the interrelated chemical bonds with using unused electron pairs in outer electron shells ($-C\equiv C-$) π -electrons and several

heteroatoms (E, N, Cl) tend to donor-acceptor and chemical-coordinate bonds, methyl groups and carbocycles can enhance these properties and improve their interaction with receptors of microorganisms.

Compared with the current bactericidal activity, in most cases the priority of bactericidal activity of new compounds is observed.

The advantage of new bactericides is that they are obtained without any difficulties on the basis of the compounds obtained in the volume of production, the high bactericidal activity of their highly diluted solutions and the simplicity of their use.

In the case of using new compounds, the socio-economic benefits are as follows:

- well - completed seed stock in agriculture;
- protection against root diseases during the cultivation and development of crops;
- cost reduction during storage of agricultural products;
- reduce the cost of sanitizing the building, warehouses, various premises, securities, and so on, because solutions with a very low concentration are used due to the high bactericidal activity of AA-007 and AA-008;
- possibility of use as a solvent in technical waters of different hardness.

Thus, thanks to the aforementioned benefits, AA-007 and AA-008 and other drugs of the same type can be used in agriculture, medicine and sanitation.

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АЦЕТИЛЕН АМИНСПИРТТЕРІ Н-ФЕНИЛКАРБАМАТТАР ТУЫНДЫЛАРЫНЫҢ СИНТЕЗІ ЖӘНЕ МИКРОБИОЛОГИЯЛЫҚ БАҒАЛАУ

Аннотация. Жұмыста бірката ацетилен аминспирттері синтезделіп, олардың N-фенилкарбаматтары алынды және соңғыларының микробтарға қарсы әрекетті зерттелді. Микробиологиялық сынақ объектілері ретінде *Bacillus subtilis*; *Botrytis cinerea*, *Echerchia coli*, *Ervinia caratovorum*, *Candida albicans*, *Fusarum solani* және *Helminthosporium* сынды микроорганизмдер қарастырылды. Ацетилен катары аминспирттерінің N-фенилкарбаматтарының физика-химиялық және бактерицидтік қасиеттері зерттелген. Карбаматтардың шығымы ацетилен аминспирттерінің құрамы мен құрылышына байланысты 68-ден 88% аралығында болды. Алынған қосылыстардың тазалығы, даралығы, құрылышы жұқа қабатты және газсүйік хроматографиялық әдістері бойынша бақыланып, элементтік сараптау және ИК мен ПМР спектрлерін түсіру арқылы анықталды. Атальшы қосылыстардың жануарлар мен осімдіктер ауруларын қозdırратын микроагзаларға қарсы пәрменділігінің жоғары екендігі көрсетілген. Зерттелген фенилкарбаматтардың салыстырмалы турде микробиологиялық белсенділіктерінің жоғары болуы молекулалары құрамында функционалдық белсенді топтардың болуына байланысты екендігі анықталды.

Түйін сөздер: карбаматтар, ацетилен аминспирттері, бактерицидтік қасиеттері, микроорганизмдер, микробиологиялық белсенділік.

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СИНТЕЗ И МИКРОБИОЛОГИЧЕСКАЯ ОЦЕНКА ПРОИЗВОДНЫХ АЦЕТИЛЕНОВЫХ АМИНОСПИРТОВ Н-ФЕНИЛКАРБАМАТОВ

Аннотация. В работе представлены результаты синтеза производных ацетиленовых амино спиртов N-фенилкарбаматов и дана микробиологическая оценка. В качестве объектов микробиологических исследований были рассмотрены микроорганизмы: *Bacillus subtilis*; *Botrytis cinerea*, *Echerchia coli*, *Ervinia caratovorum*, *Candida albicans*, *Fusarum solani* и *Helminthosporium*. Исследованы физико-химические и бактерицидные свойства N-фенилкарбаматов аминоспиртов ацетиленового спирта. Выход карбаматов зависит от содержания и строения ацетиленовых аминоспиртов и составляет 68-88%. Чистота, идентификация и строение полученных соединений определены тонкослойной и газожидкостной хроматографией, методами элементного анализа, ИК- и ПМР- спектроскопии. Установлено, что

указанные соединения проявляют высокую антимикробную активность против возбудителей некоторых болезней животных и растений. Относительно высокая микробиологическая активность исследованных фенилкарбаматов связана с наличием в составе молекул различных функциональных групп.

Ключевые слова: карбаматы, ацетиленовые аминоспирты, бактерицидные свойства, микроорганизмы, микробиологическая активность.

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ISSN 2518-1491 (Online), ISSN 2224-5286 (Print)

Редакторы: *M. С. Ахметова, Т. А. Апендиев, Аленов Д.С.*
Верстка на компьютере *A.M. Кульгинбаевой*

Подписано в печать 05.08.2019.
Формат 60x881/8. Бумага офсетная. Печать – ризограф.
5,25 пл. Тираж 300. Заказ 4.

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050010, Алматы, ул. Шевченко, 28, т. 272-13-18, 272-13-19*