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Research in the Field of Derivatives N, N¹– Polialkylene Bis [(4-Dialkylamino- (Butin-2-Il) -Karbamats] and Their Biological Properties

Makhsufov A.G., Valeeva N. G., Absalyamova G.M., Khaitov Zh. K.

D.Sc., Professor, Department of NGCICT, Tashkent chemical-technological institute, Uzbekistan
Ph.D., associate professor, Department of General Chemistry, Tashkent State Technical University named after
Islam Karimov, Uzbekistan

Senior Lecturer Department of NGCICT, Tashkent chemical-technological institute, Uzbekistan
Senior Lecturer, Department of "Technological machines and equipment" Karshi Engineering Economic Institute,
Uzbekistan

ABSTRACT: The proposed article relates to organic chemical synthesis and the study of N, N¹ - polyalkylenebis - [(4-dialkylamino- (butin-2-il) -carbamates and its chemical properties. The specific features of the synthesis were revealed, the structure, charge distribution, electron density and energy characteristics of molecules were studied by a semiempirical quantum-chemical method, the reaction centers of the used molecules, the reactivity were revealed. The structures of the synthesized compounds were established, the biological activity of N, N¹ - polyalkylenebis - [(4-dialkylamino- (butin-2-il) -carbamates "as a biostimulant of vegetable growth. A stimulating effect on seed germination was noted depending on the concentration, paths of their practical application were outlined.

KEY WORDS: carbamate, polyalkylene, hexamethylene, diisocyanate, ortho-aminoacetylphenol, biological activity, charges, electron density, field test.

I. INTRODUCTION

The rapidly developing chemistry of acetylene derivatives of carbamate compounds currently attracts the attention of many researchers, both in Uzbekistan and abroad [1-6]. It is connected, on the one hand with those rich possibilities of various chemical transformations which represent acetylene, carbamate groups in molecules of organic compounds, and on the other hand, with valuable for practical use properties of organic compounds with triple carbon-carbon bonds, and also carbamate groups. There are many examples where the introduction of triple bond led to the emergence of a range of different series of biological, physiological and pharmacological activity, as well as the ability to inhibit the corrosion of metals, to form oligomers, polymers and more.

This is due to the high reactivity and tendency to complex formation of a highly saturated terminal triple bond.

II. SIGNIFICANCE OF THE SYSTEM

The paper mainly focuses on how the chemistry derivatives of polyalkylenebis - [(4-dialkylamino- (butin-2-ol) -carbamates. The study of literature survey is presented in section III, Proposed methodology and discussion is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and Conclusion.

III. LITERATURE SURVEY

Derivatives of propargyl alcohol, butyn-2-diol 1,4-bis-carbamates, esters and ethers, acetals, and many other compounds containing mono- and bis-acetylene groups [7–10] occupy a special place among acetylene compounds. $\text{HC} \equiv \text{C} - \text{CH}_2\text{OH}$, $\text{HOCH}_2\text{C} \equiv \text{C} - \text{CH}_2\text{OH}$ are kind of universal, unique chemical compounds containing three super-highly reactive centers in a small and stable molecule: hydroxyl group, triple carbon - carbon bond and super mobile

terminal acetylene hydrogen atom. The presence of all three reaction centers is fully characterized by bis-butin-2-yl carbamates.

In the patents [21] prior to our studies, mainly the simplest representatives of acetylene mono-carbamates and others were described, however, drugs with valuable properties for practical use were also found among them. So, for example, diphenylpropargylcarbamates had pesticidal, bactericidal properties; propargylbenzoate was used as an inhibitor of steel corrosion, etc. [11-22].

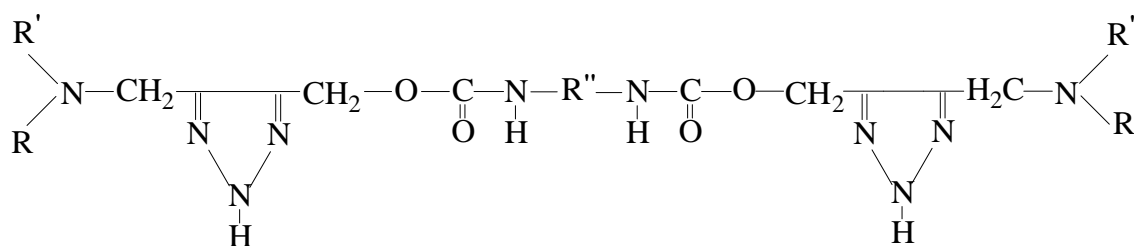
Thus, the further development of the chemistry of mono-acetylene carbamates and, moreover, bis-amino-acetylene carbamates is an extremely urgent task of the 21st century.

IV. PROPOSED METHODOLOGY AND DISCUSSION

The object of the study was the derivatives of N, N^1 - polyalkylenebis - [(4-dialkylamino- (butin-2-yl) -carbamates). The course of the reaction and the individuality of the compounds are monitored by TLC on aluminum oxide of (II) degree of activity with the appearance of spots by iodine vapor. IR spectra recorded on a spectrometer VR-20. Using the widely used HYPER CHEM program, the semi-empirical quantum-chemical method of RMZ, 3D structures, the distribution of charges and electron density of compounds were investigated. Laboratory tests have been conducted to identify the growth-promoting activity of N^1, N^1 - polyalkylenebis - [(4-dialkylamino- (butin-2-yl) -carbamates. Their physical and chemical properties were studied.

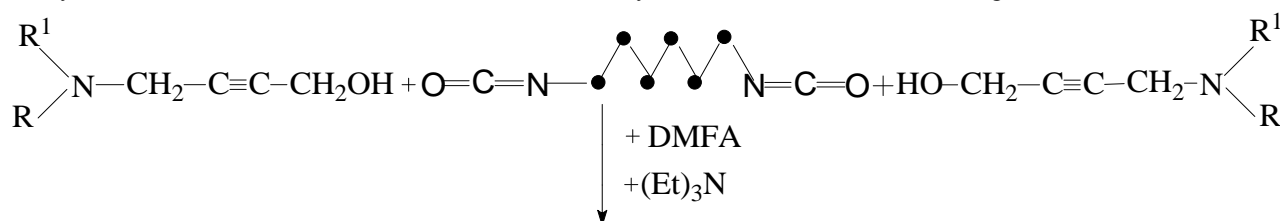
One of the main objectives of this study is a broad and systematic study of the synthesis of bis- (dialkylamine-butin-2-carbamates) based on various types of reagents and diisocyanates. It is necessary to determine the high-precision optimal methods for introducing acetylene carbamate groups into the indicated types of compounds and to study the dependence of the used carbonylation, carboxylation reactions on the presence of certain substituents in the compounds.

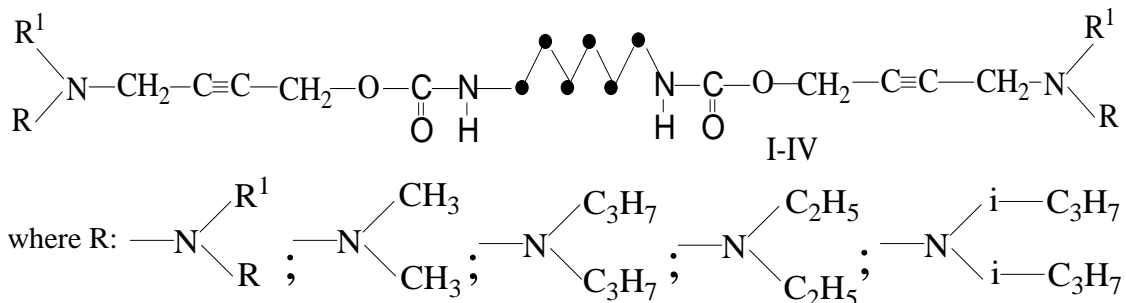
Another objective of the study was to study the chemical transformations of bis- (dialkylamino- (butin-2-yl) -carbamate) at the expense of a moving proton with N – H replacement functional groups and a triple bond. These transformations included cyclization reactions with the formation of various heterocyclic derivatives of the following type:



Features of the synthesis and reactivity were identified in chemical and physico-chemical properties. As a result, a new field of chemistry of aminoacetylene and carbamate compounds, which is the chemistry of butinyl carbamates, appeared, which has been little studied and presented only by simplest examples.

By the interaction of aliphatic {4-[N-dialkylamino]-butin-2-yl-1} with hexamethylenediisocyanate, new derivatives of dialkylamino-(butin-2) bis-carbamate were obtained. The synthesis was carried out according to the scheme:





The reaction was carried out in dimethylformamide at room temperature (without heating) for 3,5-4,0 hours. It should be noted that the derivatives of bis-aminobutiril-carbamates were obtained in rather high yields - 93-97 %.

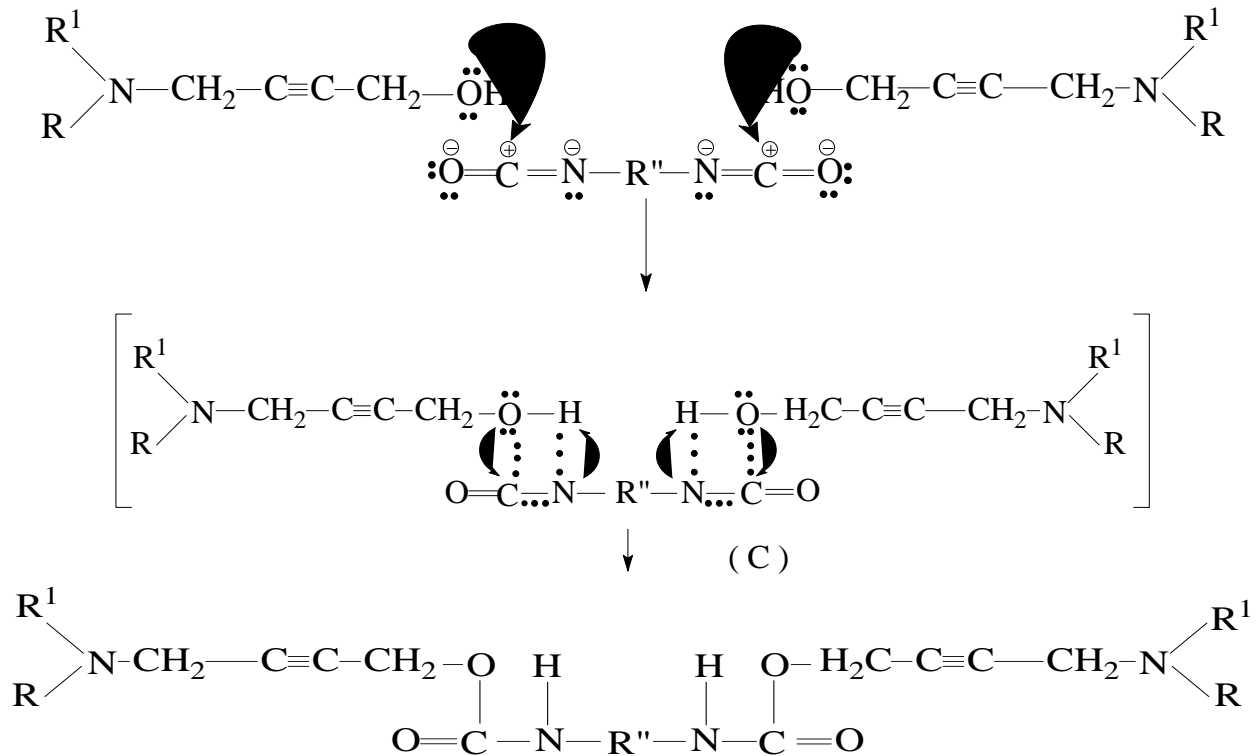
Physico-chemical characteristics of bis-carbamates I and II are shown in table 1.

Table 1. Physico-chemical characteristics of bis-carbamates I – IV

	$\begin{matrix} R^1 \\ \\ R-N \\ \\ R \end{matrix}$	Yield, %	MT, °C	R_f	Bruttoformula	Elemental analysis, %	
						Calculated	Camputed
						N	N
I	$\begin{matrix} CH_3 \\ \\ -N \\ \\ CH_3 \end{matrix}$	93,7	164-165	0,71	$C_{20}H_{34}N_4O_4$	14,21	14,02
II	$\begin{matrix} C_2H_5 \\ \\ -N \\ \\ C_2H_5 \end{matrix}$	96,2	168-169	0,70	$C_{24}H_{42}N_4O_4$	12,44	13,88
III	$\begin{matrix} C_3H_7 \\ \\ -N \\ \\ C_3H_7 \end{matrix}$	96,4	166-167	0,76	$C_{28}H_{50}N_4O_4$	11,06	11,02
IV	$\begin{matrix} i-C_3H_7 \\ \\ -N \\ \\ i-C_3H_7 \end{matrix}$	92,7	154-156	0,74	$C_{28}H_{50}N_4O_4$	11,06	10,88

The compounds were purified by recrystallization from ethyl alcohol; the individuality of the substances was established by thin-layer chromatography on Silifol plates. The following mixtures were used as eluents: $CHCl_3 : CH_3OH : HCOOH = (10 : 0,8 : 0,1)$.

Obviously, in the process of the proposed mechanism of nucleophilic addition of (A_N) 1-dialkylamino-butin-2-ol-1 and hexamethylenediisocyanate to the group $-N=C=O$, the transition of hydrogen to the nitrogen atom is easier, with the formation of an intermediate product that is an onium complex that carries partial charges on oxygen and nitrogen atoms. Subsequently, the four-membered cyclic transition state leads to the formation of bis-amino-butinil-2-carbamate.



Based on the available data, it is too early to specify the reaction mechanisms between the aminobutyn-2-ol-1 complex, the catalyst (base) and diisocyanate. The reaction proceeds in one act with the simultaneous transfer of a lone pair of electrons from isocyanate nitrogen to the 4-N-dialkylamino-butyn-2-ol-1 proton, on the one hand from alcohol oxygen to isocyanate carbon, on the other hand from isocyanate nitrogen to alcohol hydrogen.

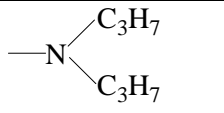
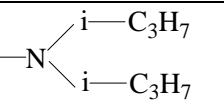
With this mechanism, the reaction of the extension of the OH bond and the approximation of its size to the length of the C = N bond in the isocyanate group (1,28 Å) should significantly facilitate the formation of a four-membered activated complex and reduce the absolute value of the negative activation energy.

Thus, in our cases, the —OH group of 1-dialkylamino-butyn-2-ol-1, having a free electron pair, attacks the electrophilic center in the diisocyanate molecule with the formation of intermediate product (C), which regroups into the final reaction product.

The formation of bis-carbamates derivatives was confirmed using the IR spectrum. The results are shown in table 2.

Table 2. Spectral data of N, N¹ -hexamethylenebis [(N-dialkylamino-butyn-2) -carbamates].

$\begin{matrix} R^1 \\ \diagup \\ R-N \\ \diagdown \\ R \end{matrix}$	IR-spectra, ν , cm^{-1}							
	NH - CH ₂ -	C=O	C≡C-	$\begin{matrix} N-C-O- \\ \quad \\ H \quad O \end{matrix}$	(-CH ₂) ₆	$\begin{matrix} N- \\ \\ H \end{matrix}$	-CH ₂ -	CH ₂ -C≡C-
$\begin{matrix} CH_3 \\ \diagup \\ -N \\ \diagdown \\ CH_3 \end{matrix}$	1418	1674	2218	1284	754-717	3285	2941	1460
$\begin{matrix} C_2H_5 \\ \diagup \\ -N \\ \diagdown \\ C_2H_5 \end{matrix}$	1420	1672	2220	1282	748-720	3290	2940	1460

	1421	1676	2222	1284	746-721	3298	2942	1458
	1419	1672	2220	1284	754-720	3319	2942	1461

In the IR spectrum of bis-carbamates I-IV, all absorption bands characteristic for N-dialkylamin-butin-2-yl-carbamates are present.

The result of the application of quantum chemistry methods is information about the density of electronic states, the distribution of electron density and potential surfaces.

Studies of the distribution of charges and electron density by atoms in the N-dimethylamino - (butin-2) -bis-carbamate molecule (Fig. 1) showed that this molecule has symmetry, the largest amount of negative charge and electron density is concentrated in oxygen atoms. Moreover, both oxygen atoms have the same activity in the reaction for the production of dimethylamino (butin-2) bis-carbamates.

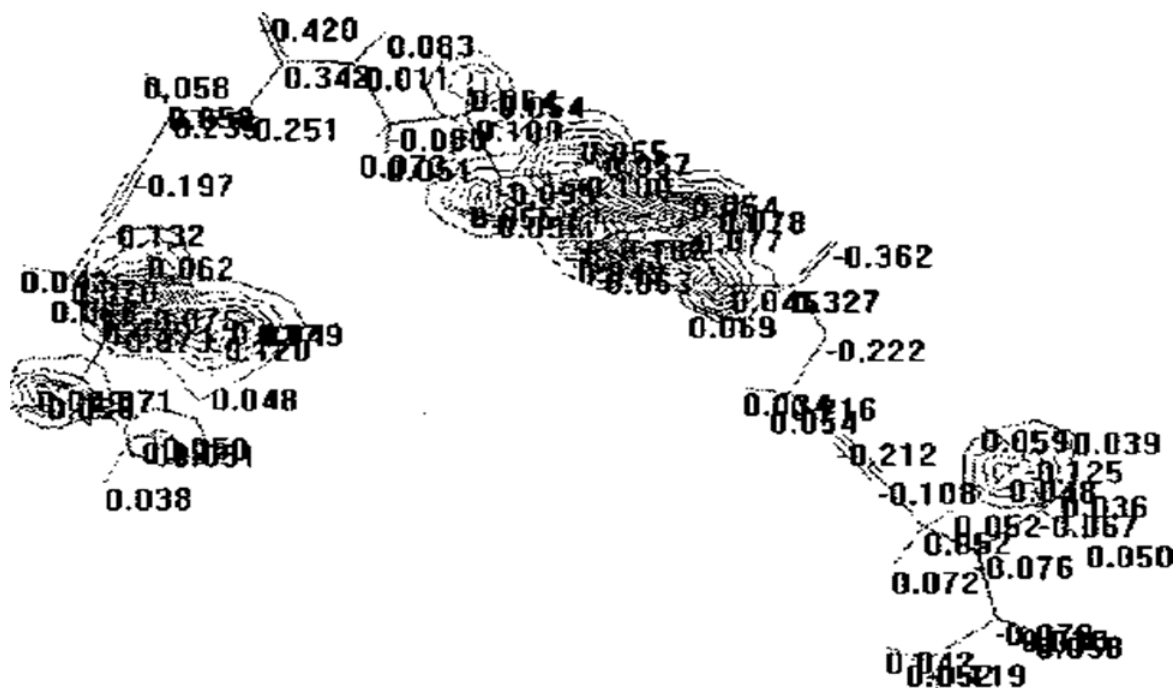


Fig. 1. The distribution of charges in the molecule and the distribution of electron density by atoms in the molecule of N-diethylamino - (butin-2) -bis-carbamates.

We also carried out quantum chemical calculations (total energy, formation energy, heat of formation, electron energy, nuclear energy, dipole moment, oxygen atom charge) (Table 3).

Thus, based on the results of studies of the structure, charge distribution, electron density, and energy characteristics of selected molecules by a semi-empirical quantum-chemical method, the reaction centers of the used molecules are identified, which in turn are used to specifically coordinate the studied reaction components molecules.

REACTIONS AND CALCULATIONS OF VARIOUS SPECTROSCOPIC QUANTITIES

At present, quantum chemistry methods are the cheapest, accessible, and universal methods for studying the electronic structure of molecules. True, it must be understood that, nevertheless, it is impossible to completely abandon the expensive experimental methods for studying substances.

Using the widely used HYPER CHEM program, the semi-empirical quantum-chemical method of RMZ, 3D structures, the distribution of charges and electron density of {4 [(N-dimethylamino) -butin-2] -ola-1}; {4 [(N-diethylamino) -butin-2]-ola-1}; {4 [(N-dipropylamino) -butin-2] -ol-1} and {4 [(N-diisopropylamino) -butin-2] -ol-1}; hexamethylenediisocyanate, N-dimethylamino- (butin-2) bis-carbamate, N-diethylamino (butin-2) -bis-carbamate; N-dipropylamino (butin-2) bis-carbamate were investigated.

As an example, we present the results of studying the geometry and electronic structure of the molecules 4 - [(N-dimethylamino) -butin-2] -ola-1, 4 - [(N-diethylamino) -butin-2] -ola-1, hexamethylenediisocyanate, dimethylamino-(butin-2) -bis-carbamates, -diethylamino- (butin-2) -bis-carbamates.

Studies of the distribution of charges and electron density among atoms in the dimethylamino- (butin-2) -bis-carbamates molecule (Fig. 1) showed that this molecule has symmetry, the greatest amount of negative charge and electron density is concentrated in oxygen atoms. Moreover, both oxygen atoms have the same activity in the reaction of obtaining α -dimethylamino- (butin-2) -bis-carbamates, -diethylamino-(butin-2) -bis-carbamates.

Table 3. Quantum-chemical calculations of the studied compounds

The nature of the compounds	The Total energy, kcal/mol	Energy of formation, kcal/mol	Heat of formation, kcal/mol	Electron Energy, eB	Nuclear Energy, kcal/mol	Dipole moment, D	Charge of an oxygen atom
Parent substances							
4 - [(N-dimethylamino) -butin-2] -ola-1	-30787,62	-1773,68	-2,66	-136473,7	105686,06	1,072	-0,303
4 - [(N-diethylamino) -butin-2] -ola-1,	-37686,05	-2336,05	-14,84	-197898,32	160212,26	2,226	-0,310
hexamethylene diisocyanate	-47140,61	-2387,79	-50,32	-224668,53	177527,92	4,608	-0,221
The synthesized compounds							
dimethylamino- (butin-2) -bis-carbamates	-108708,30	-5927,59	-48,08	-899093,25	790384,93	4,616	-0,219
diethylamino- (butin-2) -bis-carbamates	-122545	-7092,17	-112,29	-1008761,56	886216,56	6,103	-0,222

Based on the results of studying the structure of the charge distribution and the electron density of the energy characteristics of the selected molecules by the semi-empirical quantum-chemical method, the reaction centers of the used molecules are identified, which, in turn, are used for specific coordination of the studied molecules of the reaction components.

V. EXPERIMENTAL RESULTS
Synthesis of N, N¹ – hexamethylenebis [(4-N-diethylamino) -butin-2-il) -carbamate (II).

28,2 g (0,2 mol) of 4- (N-diethylamino-butin-2-ol-1, 30 ml of DMF are placed in a dry three-necked flask, equipped with a reflux condenser with a calcium chloride tube, an automixer, and a thermometer, to which 2 ml of freshly distilled dry triethylamine are added and 16,8 g (0,1 mol) of freshly distilled hexamethylenediisocyanate dissolved in

15 ml of DMF are added dropwise while stirring. The reaction mixture is stirred for 4,0 hours. After the end of the reaction, the mass is transferred into a 0,5 l glass, where 100 ml of water is added. White precipitates are dissolved in ether and chromatographed on an Al₂O₃ column. after distillation of the solvent and drying, a white powder is obtained. Yield II of the product 43,2 g (96,2% of theory). Mp = 168-169 °C; Rf = 0,70.

Found, % : C 64,07; H 9,22; N 12,36.

Calculated for C₂₄H₄₂N₄O₄, % : C 64,0; H 9,33; N 12,44.

Bis-carbamate (II) is dissolved in DMF, pyridine DMAC, DMSO, nitrobenzene and other organic solvents.

A N,N¹-hexamethylene bis [(4-N-dimethylamino-butin-2-il) carbamate]; N,N¹-hexamethylene bis [(4-N-dipropylamino-butin-2-il) carbamate]; and N,N¹-hexamethylene bis [(4-N-diisopropylamino-butin-2-il) carbamate] were obtained in a similar way.

VI. CONCLUSION AND FUTURE WORK

To identify the biostimulating activity of N, N¹-hexamethylene bis [(4-dialkylamino-butin-2-il) -carbamate] derivatives, the preparations were tested in the laboratory of phytotoxicology Institute of Chemistry of Plant Substances of the Academy of Sciences of the Republic of Uzbekistan, vegetable seeds and cotton were served as biotest, the primary cracking was carried out according to the method Yu.V. Rakitin. This method allows you to quickly determine the degree of physiological chemical activity of the compounds, which is detected by stimulating or inhibiting the germination of plant seeds, as well as by changing the length of the roots and the length of the stem part.

It has been established that when cottonseed is locked, the studied preparations of N, N¹ – hexamethylenebis [(4-N-dialkylamino-butin-2-il) -carbamates] at a concentration of 0,1; 0,001; 0,001% contributed to an increase in seed germination ahead of the control samples for biostimulating activity. It was shown that preparations contributing to the development of the root system of the seedling at a concentration of 0,001% accelerated the possibility of seeds on the fifth day several times higher than the control ones (Table 4).

Preparations I-IV at a concentration of 0,001% (i.e., at a dilution of 7500 times) biostimulated the germination of cotton seedlings by 145-152 %, and root growth by 125,6 % and stem growth by 106,7 % above the control (Table 4).

Table 4. Cotton biotest results on drug concentration

Structural formula and name	Concentration, %	Seed germination after 5 days, %	The growth of seedlings on the 10th day, in %	
			root	stem
N, N ¹ -hexamethylene bis [(4-N-dimethylamino-(butin-2-il) carbamate]	0,1	80	114,4	105,6
N, N ¹ -hexamethylene bis [(4-N-diethylamino-(butin-2-il) carbamate]	0,01	80	134,6	115,7
N, N ¹ -hexamethylene bis [(4-N-dipropylamino-(butin-2-il) carbamate]	0,001	80	128,4	109,5
Control –H ₂ O	w/t	80	100	100
"Roslyn" - (famous)	0,75	80	101,6	97,0

Preparations I-IV at a concentration of 0,001 % (ie, at a dilution of 7500 times) stimulated the germination of tomato seedlings by 146 %, and root growth by 134 % and stem growth by 122,2 % higher than the control (Table 5).

Table 5. Tomato biotest results on drug concentration

Structural formula and name	Concentration, %	Seed germination after 5 days, %	The growth of seedlings on the 10th day, in %	
			root	stem
N, N ¹ -hexamethylene bis [(4-N-dimethylamino-(butin-2-il) carbamate]	0,1	40	116,6	108,6
	0,01	48	127,3	118,4
	0,001	53	130,2	120,6

N, N ¹ -hexamethylene bis [(4-N-diethylamino-(butin-2-il) carbamate]	0,1	45	114,8	109,6
	0,01	47	119,6	114,5
	0,001	50	127,9	123,2
N, N ¹ -hexamethylene bis [(4-N-dipropylamino-(butin-2-il) carbamate]	0,1	42	114,5	111,7
	0,01	45	121,2	116,5
	0,001	53	131,3	120,6
N, N ¹ -hexamethylene bis [(4-N-diisopropylamino-(butin-2-il) carbamate]	0,1	43	119,2	110,1
	0,01	49	126,6	117,1
	0,001	52	133,5	121,2
Control –H ₂ O	w/t	30	100	100
"Roslyn" - (famous)	0,75	40	100	100

Experiments on cucumbers of the variety "Uzbekistan-740" showed that preparations I-IV actively influenced the growth of the root system of seedlings (Table 6).

Thus, new preparations I-IV at a concentration of 0,001 are the most highly effective biostimulants for vegetables and cotton in the laboratory and are recommended for further more in-depth study in the field of Navoi, Kashkadarya and Andijan regions.

Table 6. The results of the cucumber biotest on the concentration of drugs

Structural formula and name	Concentration, %	Seed germination after 5 days, %	The growth of seedlings on the 10th day, in %	
			root	stem
N, N ¹ -hexamethylene bis [(4-N-dimethylamino-(butyn-2-il) carbamate]	0,1	100	119,2	112,1
	0,01	100	126,7	117,8
	0,001	100	139,6	122,5
N, N ¹ -hexamethylene bis [(4-N-diethylamino-(butin-2-il) carbamate]	0,1	100	116,5	111,6
	0,01	100	128,2	117,5
	0,001	100	133,6	122,4
N, N ¹ -hexamethylene bis [(4-N-dipropylamino-(butin-2-il) carbamate]	0,1	100	120,2	116,5
	0,01	100	124,4	118,5
	0,001	100	136,7	119,6
N, N ¹ -hexamethylene bis [(4-N-diisopropylamino-(butin-2-il) carbamate]	0,1	100	122,1	110,6
	0,01	100	126,3	117,3
	0,001	100	132,8	119,2
Control –H ₂ O	w/t	100	100	100
"Roslyn" - (famous)	0,75	100	101,3	98,7

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AUTHOR'S BIOGRAPHY

Makhsumov Abdukhamid Gofurovich - Honored Inventor of the Republic of Uzbekistan, Academician, Doctor of Chemical Sciences, Professor.

Abdukhamid Gofurovich Makhsumov (born in 1936) created a scientific school in the direction of "Synthesis, properties and use of biologically active compounds." He published more than 1,800 scientific works, including 310 patents, more than 10 textbooks and teaching aids. He introduced more than 10 drugs in medicine, 115 biostimulators, herbicides in agriculture, animal husbandry, over 50 dyes in the textile, chemical industries, national economy.