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N, N^1 –Hexamethylene Bis - [(2-Methyl-Butanolylo-2) -Carbamate]: Synthesis, Properties and its Biological Activity

MakhsumovAbdulhamid, Haydarov Karim, ValeevaNailya,NabievUtkir

Doctor of Chemical Sciences, professor of the department of chemical technology of oil and gas refining, Honored Inventor of the Republic of Uzbekistan,valid member of the healing academy of Uzbekistan, Academician of the Ac.Sc. "Turon", Tashkent chemical-technological Institute, Uzbekistan
Doctor of Medical Sciences, Academician of the Academy of Sciences of the Republic of Tajikistan, Chief Scientific Officer of the Acting Department pharmacology, Institute of Chemistry named after V.I. Nikitin, Tajikistan.
Ph.D., associate professor, Department of General Chemistry, Tashkent State Technical University named after IslamKarimov, Uzbekistan
C.b.s, associate professor,Department of biology, National University of Uzbekistan named after MirzoUlugbek

ABSTRACT: The proposed article relates to organic chemical synthesis and the study of derivatives of N,N^1-hexamethylene bis [(2-methyl-butanolylo-2) carbamates], its chemical properties and reactions of N, N^1dinitrozozation, metallation, alkylation, and halogenation. The structures of the synthesized compounds were establishedas, well as a growth promoter of industrial plants. The results of the biological activity of derivatives of N,N^1-hexamethylene bis [(2-methyl-butanolylo-2) carbamates], are presented. Trials of the preparation N, N^1-hexamethylene bis [(2-methyl-butanolylo-2) carbamate] are the most effective growth-regulating preparation of vegetables and cotton in the laboratory and further more in-depth study in the field is recommended

KEY WORDS: synthetic organic compounds, Derivatives, Carbamate, Hexamethylene, N,N^1- dichlorination, dinitrosoziration, alkylation, halogenation, Field test.

I.INTRODUCTION

In chemistry and technology of synthetic organic compounds, the direction of fine organic synthesis of substances has gained particular development, among which a significant role is given to derivatives of carbamate and bis-carbamate, obtained on the basis of isocyanates, as well as hydroxyl-containing radicals.

II. SIGNIFICANCE OF THE SYSTEM

The paper mainly focuses on how the chemistry derivative of N,N^1-hexamethylene bis [(2-methyl-butanolylo-2) carbamates] compounds. 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

Numerous studies in the field of carbamates and bis-carbamates derivatives, carried out at the present time, are prompted not only by theoretical, but also by practical needs. From this point of view, derivatives of carbamates and bis-carbamates are of undoubted interest as substances with different technical, biological and pharmacological activity. They are successfully used in almost all sectors of the national economy, in particular in technology as accelerators of vulcanization of rubbers, as thermal stabilizers of polymers, additives to lubricating oils, and are also used as starting materials for the production of polymers, as corrosion inhibitors [1-10].

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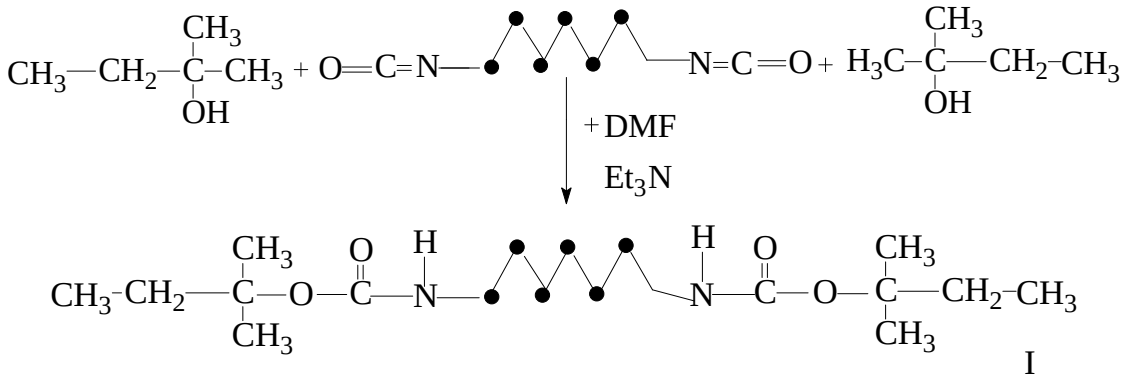
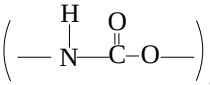
In agriculture, they have found application as pesticides, herbicides, fungicides, defoliants, insecticides, nematicides, bactericides, biostimulants and many others. Of particular interest is the use of these class of compounds in medicine as antitumor, antiviral, antidiabetic, low cholesterol, antiarrhythmic, anti-inflammatory and other drugs [11-22].

IV. PROPOSED METHODOLOGY AND DISCUSSION

In this regard, we presented previously conducted studies in the field of synthesis of new derivatives of N,N^1-hexamethylene bis [(2-methyl-butanolylo-2) carbamates] and studied their chemical properties by reaction centers.
Thus, by the interaction of 2-methylbutanol-2 with diisocyanates, derivatives of N, N^1-hexamethylene bis [(2-methyl-butanolylo-2) carbamate] were obtained according to the following reaction scheme:

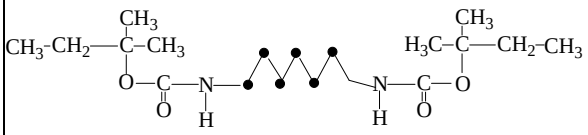


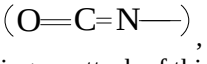
I

The reaction of hexamethylenediisocyanate with 2-methyl-butanol-2 was carried out at a molar ratio of reagents 1: 2 at room temperature 28-36 ^oC for 3,5-4,0 hours. As a result of the reaction, N, N^1-hexamethylene bis [(2-methylbutanolylo-2) carbamate] (I) is formed, which is a snow-white, high melting powder, hardly soluble in water and other non-polar light solvents available, which confirms the presence of two-carbamate  , and also hexamethylene hydrocarbons.

Physico-chemical characteristics N, N^1-hexamethylene bis [(2-methylbutanolylo-2) carbamate] are shown in table 1.

Table 1.Physico-chemical characteristics of the drug (I)

№ п/п	Structural formula	Yield, %	MT, ^oC	R_f	Bruttoformula	Elemental analysis, %		Мм
						Calculated	Found	
						N	N	
I		86,3	207-208	0,74	$C_{18}H_{36}N_2O_4$	8,14	6,19	344

As can be seen from table 1, the yield of N, N^1-hexamethylene bis [(2-methylbutanolylo -2) carbamate] is quite high. The high yield of the obtained derivative of bis [(2-methylbutanolylo -2) carbamate] is apparently due to the high density and light mobility of the electron cloud of the conjugated group  , which leads to an increase in the positive charge on the carbon atom of the isocyanate group, possessing an attack of this nucleophilic agent, and also the absence of steric barriers.

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The structure of the synthesized compound (I) was established by IR and PMR spectroscopy and elemental analysis data (table 2).

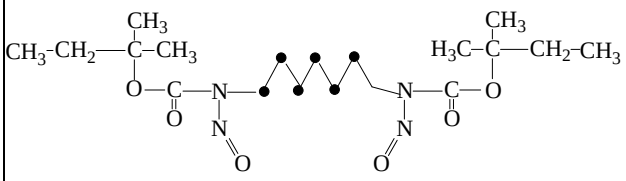
To identify the reactivity of N, N^1-hexamethylene bis [(2-methylbutanolylo -2) carbamate] N-H reactive centers, we carried out the reactions of N, N^1dinitrozozation,metallation, alkylation and halogenation.

Table 2. IR and PMR spectral data of compounds (I)

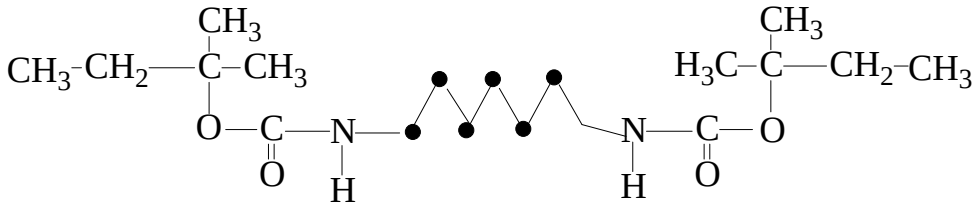
Compound I	IR-spectra, ν, sm^{-1}					PMR spectrum, δ, m.g.		
		NH - CH_2	C=O			CH_3	CH_2–N	CH_2
	1594	1430-1376	1691	3290	753-718	2,19	3,05	1,42-1,40

Obtaining N, N^1-dinitrozo-substituted - N, N^1-hexamethylene bis [(2-methylbutanolylo-2) carbamate].
As a result of the N, N^1 dinitrozozation reaction of the bis [(2 - methylbutanolylo-2) carbamate] derivative with sodium nitrite (in excess) in formic acid, the corresponding N, N^1 dinitrozo of substituted bis [(2-methylbutanolylo-2) carbamate was obtained] with a yield of 82,4% (table 3).

Table 3.Physicochemical parameters of compound (II).

№ p/p	Structural formula	Yield, %	MT, °C	Bruttoformula	Elemental analysis, %		Mм
					Calculated N	Found N	
II		82,4	340(dec).	$C_{18}H_{34}N_4O_6$	13,93	13,79	402

N, N^1-dinitrozozironium proceeds according to the electrophilic substitution mechanism (S_E).



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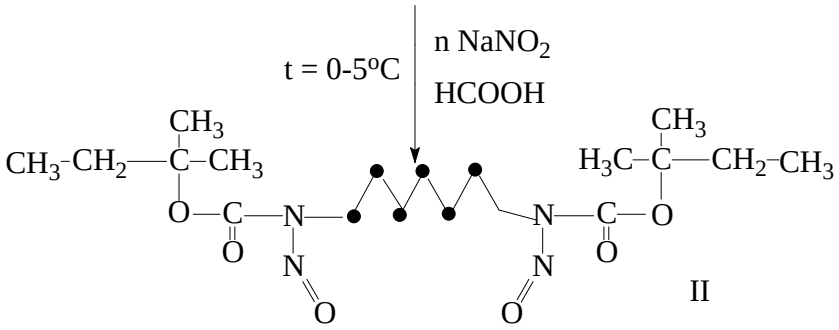


The attacking agent is nitrozonium ion $-\overset{\oplus}{N}O$. Since nitrous acid, which is the most common agent, does not exist in its free form, sodium nitrite and strong acid are used to carry out the process, adding a proton, generating an ion $-\overset{\oplus}{N}O$:

$$NaNO_2 + \underset{(conc)}{HCOOH} \longleftrightarrow H_2\overset{\oplus}{N}O_2 \longleftrightarrow \overset{\oplus}{N}O + HCOONa + H_2O$$

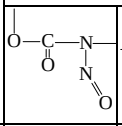
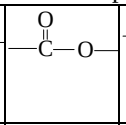
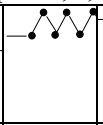
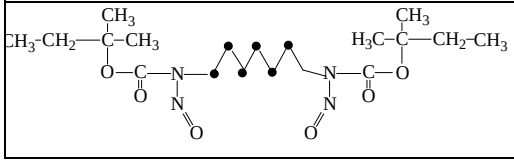
N, N^1-dinitrozozation is carried out by cooling (0-5 °C) of the reaction mixture. An increase in temperature is undesirable, as this reduces the yield of the target product, and sometimes affects the direction of the reaction.

Identification of N, N^1-dinitroso compounds is carried out by the absorption bands of N-nitrozo groups.

A very strong absorption band in the 1528-1442 cm^{-1} region for groups $\diagdown N-N=O$ is characteristic.

The structure of the synthesized compound (II) was established by IR spectroscopy and elemental analysis data (table 4).

Table 4. IR and PMR spectral data of compound (II).

Compound II	IR-spectra, ν, sm^{-1}				PMR spectrum, δ, m.g.		
			 NH - CH_2–	CH_3—	—CH_2—N	—CH_2—
	1528-1442	1692	754-719	1430-1375	2,19	3,05	1,42-1,40

Obtaining N, N^1-disodium substituted N, N^1-hexamethylene bis [(2-methylbutanolylo-2) -carbamate].

One of the metallization methods that can be carried out using N-metalization is the substitution of hydrogen atoms by sodium in the N-H group. N, N^1-hexamethylene bis [2- (methylbutanolylo-2) carbamate] is subjected to directional metallation at the N-H groups with CH_3ONa. The reaction of N, N^1-dimetallation proceeds as follows:

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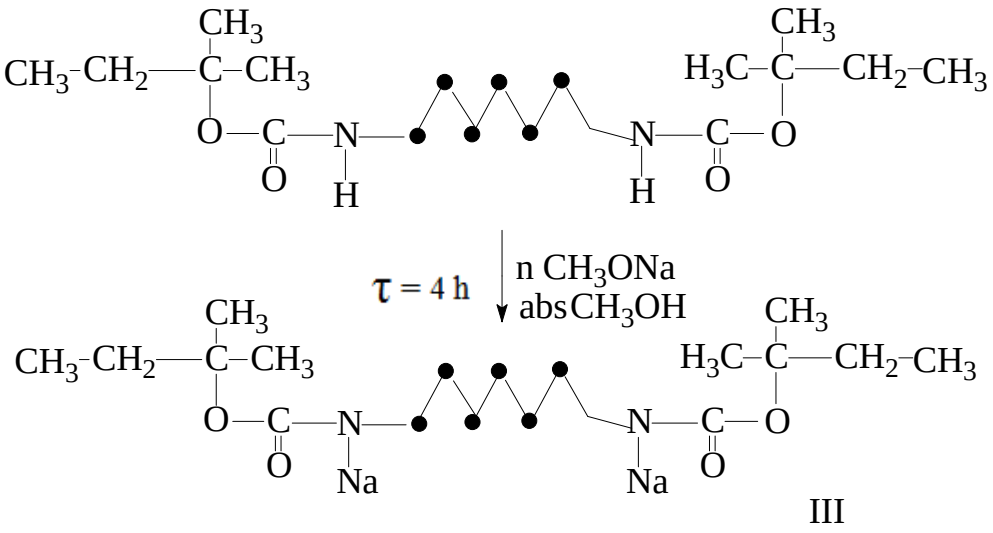
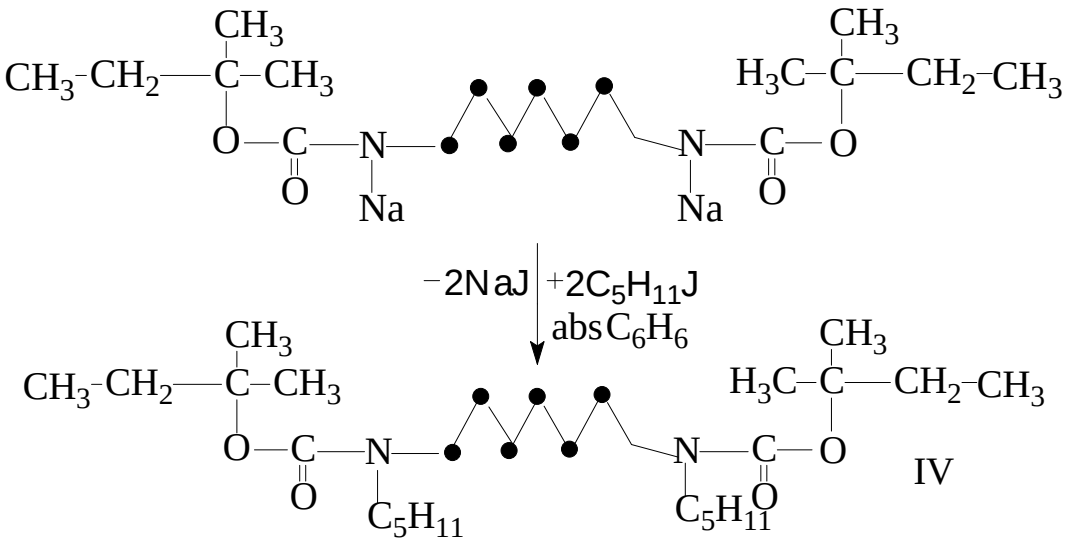


Obtaining N, N^1-diamyloyl substituted N, N^1-hexamethylene bis [(2-methylbutanolylo-2) carbamate] (IV**).**
Anylation in carbamates with alkyl halides is of undoubted interest in elucidating the reactivity of N-H containing compounds. We carried out alkylation reactions by the interaction of N, N^1-disodium derivatives of N, N^1-hexamethylene bis [(2-methylbutanolylo-2) carbamate] with pentane iodide in absolute dry benzene at room temperature 30-38 ^{o}C with stirring for 3,5-4,0 hours according to the scheme:



The course of the alkylation reaction exclusively at the nitrogen atom N, N^1 - is apparently due to the relatively easy dissociation of sodium at this atom due to the presence of neighboring carbonyl groups. The yield of product (IV) is 86,7 %, mp = 164-165 ^{O}C.

Physico-chemical parameters of the obtained product (IV) are shown in table 5.

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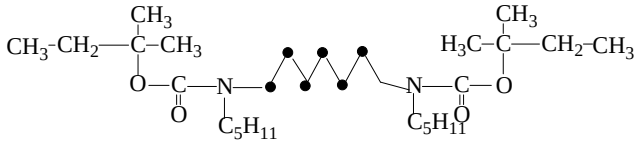
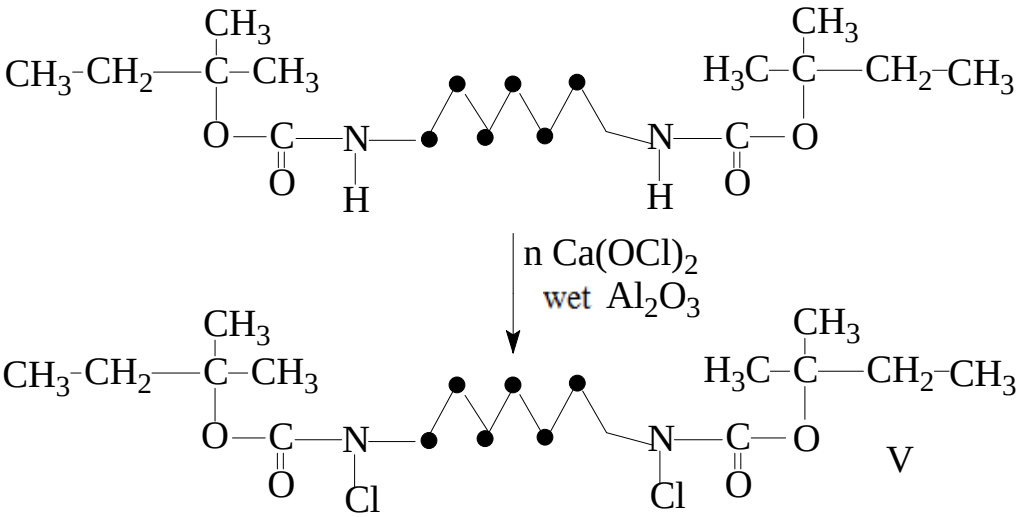
Table 5. Physicochemical Parameters of Compound (IV)

№ p/p	Structural formula	Yield, %	MT, °C	R_f	Bruttoformula	Elemental analysis, %		Мм
						Calculated	Found	
						N	N	
IV		86,7	164-165	0,71	$C_{27}H_{56}N_2O_4$	5,93	5,79	472

Obtaining N, N^1-dichlorosubstituted N, N^1-hexamethylene bis [(2-methylbutanolylo-2) -carbamate] (V)

An effective, affordable, cheap, environmentally friendly method has been developed for the implementation of N, N^1-dichlorination of a bis-carbamate derivative with calcium hypochlorite on wet Al_2O_3. The chemical reaction scheme is as follows:



These reactions are of interest to many specialists in chemists, pharmacologists, biologists, biochemists, bioorganics, microbiologists, and many others, due to the presence of a vital, highly reactive center (-NH group) in the bis-carbamate derivative necessary for nucleophilic and electrophilic substitution.

The product yield and physico-chemical parameters are shown in table 6.

To prove the structure of the newly synthesized N, N^1-dichloro substituted N, N^1-hexamethylene bis [(2-methylbutanolylo-2) -carbamate], IR spectra were taken, elemental analysis and qualitative reactions with $AgNO_3$ were performed.

Thus, methods have been developed for the preparation of N, N_1-hexamethylene bis [(2-methylbutanolylo-2) carbamate] and its chemical properties are studied by N-H reaction centers: nitrozozironium, metallation, alkylation and chlorination reactions.

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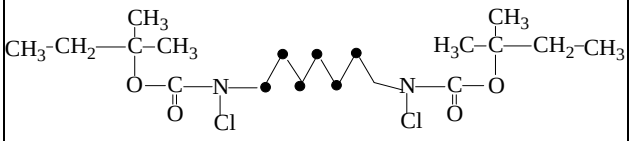
Table 6. Physicochemical parameters of compounds (V).

№ p/p	Structural formula	Yield, %	MT, °C	R_f	Bruttoformula	Elemental analysis, %				$M_м$
						Calcu-lated		Found		
						N	Cl	N	Cl	
IV		92,4	119-120	0,69	$C_{18}H_{34}Cl_2N_2O_4$	6,78	17,19	6,61	17,06	413

V. EXPERIMENTAL RESULTS

The progress of the reaction and the individuality of the compounds are monitored by TLC on alumina of (II) degree of activity with the appearance of spots by iodine vapor. IR spectra were recorded on a VR-20 spectrometer.

A. Synthesis of N, N^1-hexamethylene bis [(2-methylbutanolylo-2) -carbamate] (I).

7 ml of triethylamine are added to 17,6 ml (0,2 mol) of 2-methylbutanol-2, 16,8 ml (0,1 mol) of hexamethylenediisocyanate dissolved in 40 ml of DMF are added dropwise with stirring at room temperature. The reaction mixture is stirred for 3 hours at a temperature of 30-44 °C. After a time, the contents of the flask are transferred to a glass, water is added. The precipitate was washed with TLC. After drying, a colorless powder is obtained; the yield of product (1) is 29,6 g (86,3 % of theory); Mp = 207-208 °C. Rf = 0,74;

Found, %: C 62,63; H 10,37; N 7,96

Calculated for $C_{18}H_{36}N_2O_4$, %: C 62,79; H 10,46; N 8,14

B. Synthesis of N, N^1-dinitrozo-N, N^1-hexamethylene bis [(2-methylbutanolylo) carbamate] (II).

0,8 g of sodium nitrite in excess is added in portions constantly stirring at a temperature of 0-5 °C for 3,5-4,0 hours to 3,44 g (0,01 mol) (I) dissolved in 80 ml of formic acid. After the end, it is poured into a glass, water is added, the precipitate formed is filtered off, washed with benzene and dried, TLC on Silifol plates, yield 82,4 %; Mp = 340 °C (dec).

Found, %: C 53,58; H 8,39; N 13,79

Calculated for $C_{18}H_{36}N_4O_6$, %: C 53,73; H 8,45; N 13,93

C. Synthesis of N, N^1 - disodium - N, N^1 - hexamethylenebis [(2-methylbutanolylo) carbamate] (III).

3,44 g (0,01 mol) (I) are added to CH_3ONa (from 0,031 g / mol and 80 ml of abs. CH_3OH). The mixture is stirred for 2 hours at a temperature of 20 °C and 2 hours at 40 °C. The precipitate is filtered off, washed with absolute. CH_3OH and get (III), yield 3,38 g –87,3 % (of theoretical); Mp = 330 °C (dec).

D. Synthesis of N, N^1 -diamylo - N, N^1 - hexamethylenebis [(2-methylbutanolylo) carbamate] (IV).

3,38 g (0,087 mol) (III) is placed in 15 ml of DMF, 3,9 ml (0,02 mol) of pentane iodide is added dropwise with stirring, the mixture is stirred for 10.5 hours while heating in a boiling water bath, cooled and washed with 20 ml of water, the precipitate was separated, recrystallized from 50% alcohol, dried and obtained (IV) with the release of 3,18 g – 86,7 % (of theoretical); Mp = 164-165 °C (dec). Rf = 0,71;

Found, %: C 68,56; H 11,73; N 5,79

Calculated for $C_{27}H_{56}N_2O_4$, %: C 68,64; H 11,86; N 5,93

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E. Synthesis of N, N^1-dichloro-N, N^1-hexamethylene bis [(2-methylbutanolylo-2) carbamate] (V).

4,0 g of calcium hypochlorite are added dropwise at 40 °C over 1 hour to 3,44 g (0,01 mol) (1), 60 ml of CCl_4, 20 g of wet alumina. The reaction mass is 21 hours. It is filtered off, washed with ether, alcohol, dried and obtained (5). With a yield of –3,81 g (92,4% of theory); Mp = 119-120 °C. Rf = 0,69;

Found, %: C 52,21; H 8,11; N 6,61; Cl 17,06

Calculated for $C_{18}H_{34}Cl_2N_2O_4$, %: C 52,30; H 8,23; N 6,78; Cl 17,19

VI. CONCLUSION AND FUTURE WORK

The biological activity of the drug (I)

In the laboratory of phytotoxicology of the Institute of Plant Chemistry of the Academy of Sciences of the Republic of Uzbekistan, tests were conducted for an initial assessment of the growth-promoting effect of newly synthesized chemical preparations.

We studied the preparation of N, N^1-hexamethylene bis - [(2-methylbutanolylo-2) -carbamate] for growth-promoting activity. Biotests were seeds of vegetable crops and cotton. A simple, fairly accurate assessment of the primary biological activity of the preparations on the seeds of the above biotests is presented.

Primary screening was performed according to the method of Yu.V. Rakitina. This method allows you to quickly determine the degree of physiological activity of chemical compounds, which is detected by stimulation or inhibition of germination of plant seeds, as well as by changing the length of the roots and the length of the stem part.

The preparations were tested by the method of seed locking in solutions of various concentrations, followed by germination in Petri dishes. Control seeds were soaked in distilled water.

The experiments used cucumbers of the Uzbekistan-740 variety, tomatoes of the Temp variety and medium-fiber cotton of the S-6524 variety.

The drug was dissolved in DMSO and the method of pre-locking the seeds for 18-20 hours was used. 3 concentrations were used: 0,1%; 0,01%; 0,001%. The repetition of experiments is 4-fold. The counts were carried out by measuring the length of the stem and root of 10-day-old seedlings of vegetable crops (cucumbers and tomatoes) and 7-day-old seedlings of cotton.

It was found that when the tomato seeds are clasped, the preparation (I) in a concentration of: N, N^1-hexamethylene bis - [(isoamyloyl) carbamate] – 0,1; 0,01 %% contributed to an increase in their germination. The seeds showed the most significant increased germination when the concentration of the drug (I) was 0,001%, ahead of the control by 48,8 %. The best effect of stimulating the roots and stems of tomato seedlings was obtained by us when the seeds were locked in a solution of the preparation (I) – 0,01; and 0,001; (24,9 and 32,2 %), and the stem part of 27,6 % compared with the control (table 7).

Table 7. The effect of the drug (I) on seed germination and growth of tomato seedlings

Experiences A drug	Concentration, %	Germination, %	Tomato	
			Root growth	Stem growth
Control - water	w/t	70,0	100,0	100,0
N, N^1-hexamethylene bis - [(2-methylbutanolylo-2) -carbamate]	0,1	105,4	115,2	113,4
	0,01	68,0	125,9	127,6
	0,001	90,0	134,7	114,8
«Roslyn» (famous)	0,75-1,0	79,0	110,9	111,4

Thus, among the tested preparations, (I) is the most effective growth-regulating preparation of vegetable crops under lobor conditions, and further more in-depth study in the field is recommended.

The study of the studied preparation (I) on the growth-promoting activity of cotton showed that the preparation contributed to the germination of seeds and the development of the root system of seedlings. So, the drug (I) – 0,01 % concentration accelerated seed germination on the 3rd day from 16,7 % to 30,2 % above the control (table 8).

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Table 8. The effect of the drug (I) on seed germination and seedling growth
Cotton of "S-6524" varieties.

Experiences A drug	Concentration, %	Germination, %	Cotton	
			Root growth	Stem growth
Control - water	w/t	85,0	100,0	100,0
N, N^1-hexamethylene bis - [(2-methylbutanolylo-2) -carbamate]	0,1	93,0	110,7	124,5
	0,01	96,2	120,2	118,3
	0,001	98,3	122,3	120,7
«Roslyn» (famous)	0,75-1,0	85,0	104,8	106,4

The best effect on root formation of drug (I) at a concentration of 0,001 % increased root formation by 22,3 % more than in the control. On the cucumber culture, the preparation (I) showed activity on the growth of the root part at a concentration of 0,1 % (148,4 %); = 0,01 % (124,8 %); at a concentration of 0,001 % (148,87 %); and stem growth is much lower - (110,5 %) higher than control (table 9).

Table 9. The effect of the drug AGM-96 on seed germination and seedling growth
cucumbersof "Uzbekistan-740" varieties

Experiences A drug	Concentration, %	Germination, %	Cucumbers	
			Root growth	Stem growth
Control - water	w/t	90,0	100,0	100,0
N, N^1-hexamethylene bis - [(2-methylbutanolylo-2) -carbamate]	0,1	83,0	148,4	103,3
	0,01	100,0	124,8	98,6
	0,001	96,6	148,87	110,5
«Roslyn» (famous)	0,75-1,0	90,2	105,7	104,4

Thus, this preparation (I) can be used on cotton culture both by spraying vegetative plants (in the budding phase) and by treating cotton seeds. On a cucumber culture, the preparation (I) at a concentration of 0,1 %; 0,01 %; and 0,001 % can be used when spraying. And on tomatoes, it can be used at a concentration of 0,01-0,001 % both when locking the seeds, and when spraying plants.
Thus, trials of the preparation (I) are the most effective growth-regulating preparation of vegetables and cotton in the laboratory and further more in-depth study in the field is recommended.

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AUTHOR'S BIOGRAPHY

Makhsumov Abdukhamid Gofurovich - Honored Inventor of the Republic of Uzbekistan, Academician, Doctor of Chemical Sciences, Professor, valid member of the healing academy of Uzbekistan, Academician of the Ac.Sc. "Turon", Tashkent chemical-technological Institute, Uzbekistan

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.