



ISSN: 2350-0328

**International Journal of Advanced Research in Science,  
Engineering and Technology**

**Vol. 6, Issue 11, November 2019**

# **N,N<sup>1</sup> –hexamethylenebis - [(isobutanoyilo) - carbamate]: synthesis, properties and its biostimulating activity**

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**ABSTRACT:** The proposed article relates to organic chemical synthesis and the study of derivatives of N,N<sup>1</sup>-hexamethylenebis - [(isobutanoyilo) - carbamate], its chemical properties and reactions of N, N<sup>1</sup>- dinitrosation, metallation, alkylation, and halogenation. The structures of the synthesized compounds were established as well as a growth promoter of industrial plants. The results of the biological activity of derivatives of N,N<sup>1</sup>-hexamethylenebis - [(isobutanoyilo) - carbamate] are presented. Trials of the preparation N,N<sup>1</sup>-hexamethylenebis - [(isobutanoyilo) - 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, Isobutanoyilo, Hexamethylene, N,N<sup>1</sup>-dichlorination, Dinitrosation, Alkylation, Halogenation, Field test, Cotton, Cucumber, The drug "Roslin".

## **I. INTRODUCTION**

In the 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<sup>1</sup>-hexamethylenebis - [(isobutanoyilo) - carbamate]. 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**


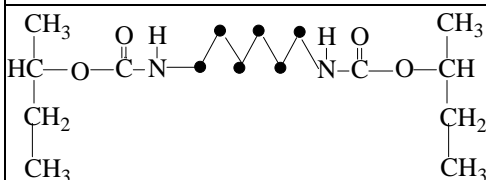
Numerous studies in the field of derivatives of carbamates and bis-carbamates, carried out at present, are prompted not only by theoretical, but also by practical needs. From this point of view, derivatives of carbamates and bis-carbamates are of undoubted interest as substances with different technical, 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 used as starting materials for the production of polymers, as corrosion inhibitors [1-8].



mobility of the electron cloud of the conjugated ( $\text{—N=C=O}$ ) group, which leads to an increase in the positive charge on the carbon atom of the isocyanate group, having an attack of this nucleophilic agent and the absence of steric hindrances.

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

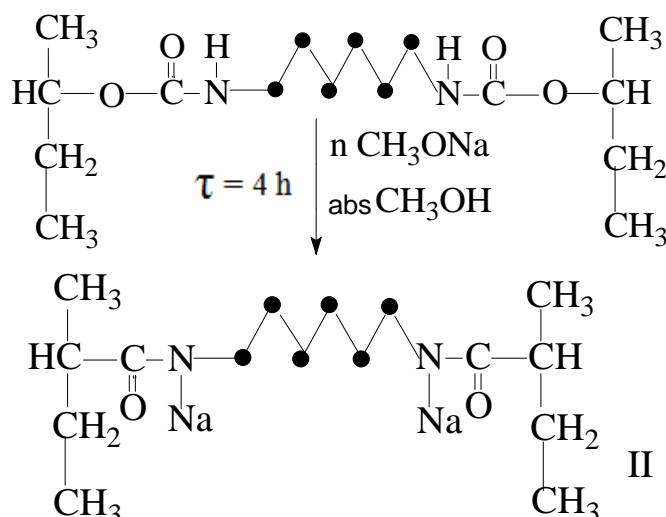
Table 2. IR and PMR spectral data of compounds (I)

Compound I	IR-spectra, $\nu$ , $\text{cm}^{-1}$					PMR-spectra, $\delta$ , m.g.		
	$\text{—O—C(=O)—N—H—}$	$\text{H—N—CH}_2\text{—}$	$\text{O=C—}$	$\text{—N—}$		$\text{—CH}_3$	$\text{—N—CH}_2\text{—}$	$\text{—CH}_2\text{—}$
	1593	1430-1374	1693	3292	754-718	2,19	3,05	1,42-1,40

We carried out the reactions of N, N<sup>1</sup>-dimetallation, alkylation, halogenation and dinitrosation to reveal the reactivity of N, N<sup>1</sup>-hexamethylene bis-[(isobutanoylo) carbamate] at the N-H reaction centers.

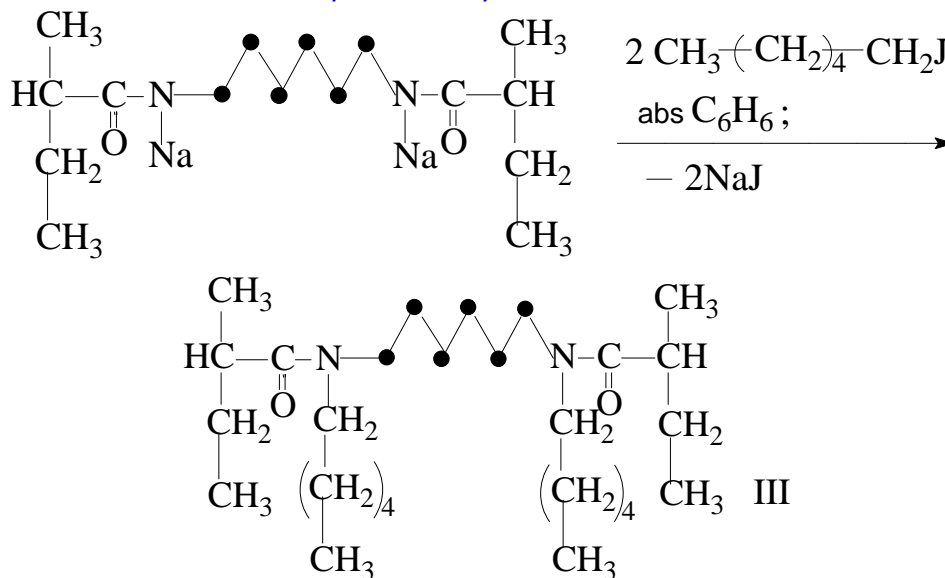
### Obtaining N, N<sup>1</sup>-disodium substituted - N, N<sup>1</sup>-hexamethylene bis-[(isobutanoylo) carbamate] (II).

One of the metallization methods that can be carried out using N-metalization is the replacement of hydrogen atoms with sodium in the N-H group. N, N<sup>1</sup>-hexamethylenebis - [(isobutanoylo) carbamate] is subjected to directional metallation at the N-H groups with  $\text{CH}_3\text{ONa}$ . The reaction of N, N<sup>1</sup>-dimetallation proceeds according to the following scheme:



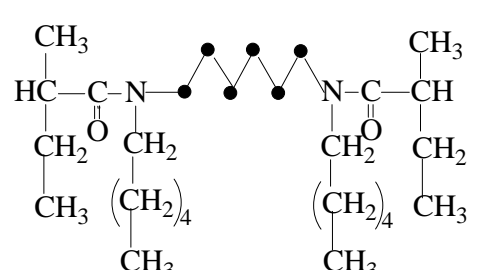
### Obtaining N, N<sup>1</sup>-dihexyllyl substituted - N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] (III).

n-Hexylation 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<sup>1</sup>-disodium derivatives of N, N<sup>1</sup>-hexamethylene bis[(isobutanoylo) -carbamate] with n-hexane iodide in absolute dry benzene at room temperature 28-38 °C with stirring for 3,5-4,0 hours according to the scheme:



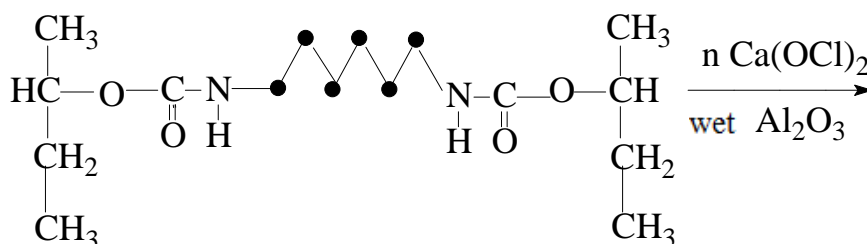
The occurrence of the alkylation reaction exclusively at the nitrogen atom N, N<sup>1</sup> - is apparently due to the relatively easy dissociation of sodium at this atom due to the presence of neighboring carbonyl groups. The yield of product (III) is 82,6%; Mp = 139-140 °C. Physico-chemical parameters of the obtained product (III) are shown in table 3.

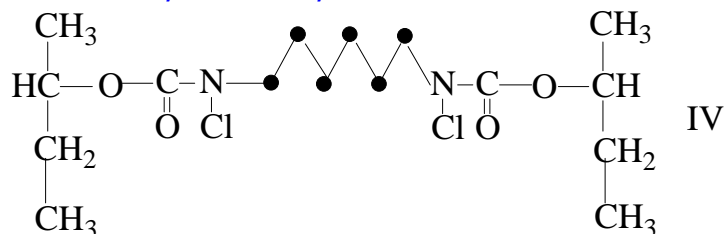
Table 3. Physico-chemical parameters of compound (III).

Compound III	Yield, %	MT, °C	R <sub>f</sub>	Brutto formula	Elemental analysis, %		M <sub>M</sub>
					Calculated	Found	
					N	N	
	82,6	139-140	0,69	C <sub>28</sub> H <sub>44</sub> N <sub>2</sub> O <sub>4</sub>	5,93	5,81	472

**Obtaining N, N<sup>1</sup>-dichloro substituted N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] (IV)**

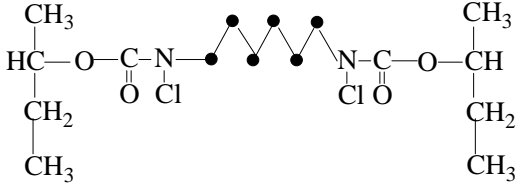
An effective, affordable, cheap, environmentally friendly method has been developed for the implementation of N, N<sup>1</sup> - dichlorination of a bis-carbamate derivative with calcium hypochlorite on wet Al<sub>2</sub>O<sub>3</sub>. The chemical reaction scheme is as follows:





These reactions are of interest to many specialists in the field of chemistry, pharmacology, biology, biochemistry, bioorganics, microbiology and many others, due to the presence of a vital, highly reactive center (N-H group) in the bis-carbamate derivative, which is necessary for nucleophilic and electrophilic substitution reactions. The product yield (IV) and physicochemical parameters are shown in table 4.

Table 4. Physico-chemical parameters of compound (IV).

Structural formula	Yield, %	MT, °C	R <sub>f</sub>	Brutto formula	Elemental analysis %				M <sub>M</sub>
					Calculated		Found		
					N	Cl	N	Cl	
	92,3	113-114	0,72	C <sub>16</sub> H <sub>30</sub> Cl <sub>2</sub> N <sub>2</sub> O <sub>4</sub>	7,27	18,44	7,17	18,36	385

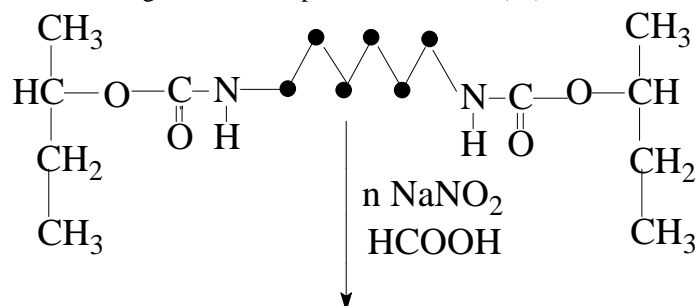
To prove the structure of the newly synthesized N, N<sup>1</sup>-dichloro-substituted N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] IR spectra were taken, elemental analysis and qualitative reactions with AgNO<sub>3</sub> were performed.

#### Obtaining N, N<sup>1</sup>-dinitroso substituted N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] (V)

As a result of the N, N<sup>1</sup>-dinitrosation reaction of the derivative of bis - [(isobutanoylo) carbamate] with sodium nitrite (in excess) in formic acid, the corresponding N, N<sup>1</sup>-dinitrososubstituted bis - [(isobutanoylo) carbamate] was obtained in 82,4 yield % (table 5).

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

N, N<sup>1</sup>-dinitrosation proceeds according to the electrophilic substitution (S<sub>E</sub>) mechanism.



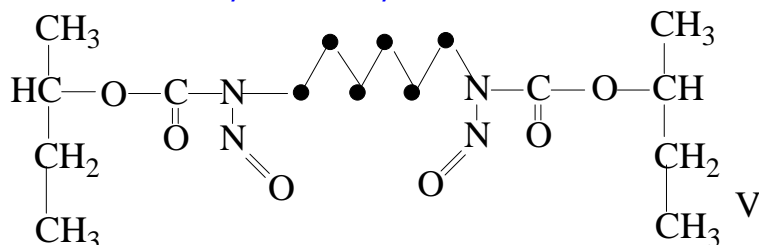
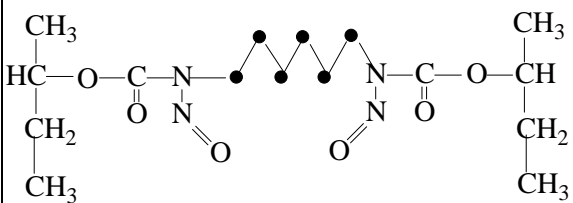
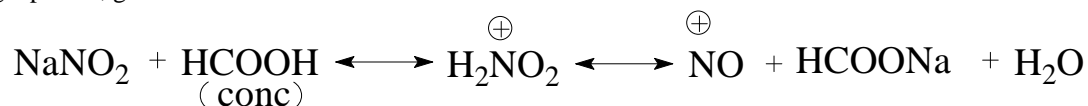


Table 5. Physico-chemical parameters of the compound (V)

Compound III	Yield, %	MT, °C	Brutto formula	Elemental analysis, %		M <sub>M</sub>
				Calculated	Found	
				N	N	
	82,4	340 °C (decomp)	C <sub>16</sub> H <sub>30</sub> N <sub>4</sub> O <sub>6</sub>	14,97	14,88	374

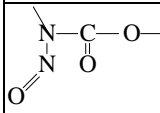
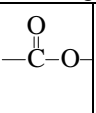
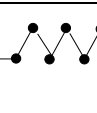
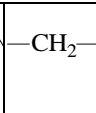
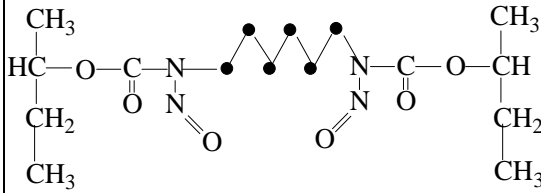
The attacking agent is nitrosonium ion  $\text{NO}^{\oplus}$ . Since nitrous acid, which is the most common agent, does not exist in its free form, sodium nitrite and strong acid are used to carry out the process, the nitrous acid formed in this process, attaching a proton, generates an ion  $\text{NO}^{\oplus}$ .



N, N<sup>1</sup>-dinitrosation is carried out by cooling (0-5 °C) of the reaction mixture. An increase in temperature is not desirable, since this reduces the yield of the target product, and sometimes affects the direction of the reaction.

Identification of N, N<sup>1</sup>-dinitroso compounds is carried out by absorption bands of N-nitroso groups. A very strong absorption band in the 1528-1440 cm<sup>-1</sup> region for groups  $\text{>N-N=O}$  is characteristic. The structure of the synthesized compound (V) was determined by IR spectroscopy and elemental analysis data. (Table 6).

Table 6. IR spectral data of compound (V).

Structural formula	ИК-spectra, v, cm <sup>-1</sup>			
				
	1558-1432	1717	766-724	2940

The progress 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 were recorded on a VR-20 spectrometer.

**A. Synthesis of N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] (I).**

8 ml of triethylamine, 40 ml of DMF are added to 14,8 ml (0,1 mol) of isobutanol. 16,8 ml (0,1 mol) of hexamethylenediisocyanate dissolved in 40 ml of DMF was added dropwise with stirring at room temperature. The reaction mixture is stirred for 3 hours at a temperature of 39-46 °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 (I) is 2,9 g (91,7 % of theoretical); Mp = 268-269 °C.

Found, %: C 60,27; H 10,01; N 8,78  
Calculated for C<sub>16</sub>H<sub>32</sub>N<sub>2</sub>O<sub>4</sub>, % : C 60,76; H 10,12; N 8,86

**B. Synthesis of N, N<sup>1</sup>-disodium, -N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] (II).**

3,16 g (0,01 mol) (I) was added to CH<sub>3</sub>ONa (from 0,031 g / mol and 80 ml abs. CH<sub>3</sub>OH). 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 abs. CH<sub>3</sub>OH and get (II), yield 86,3 %; Mp = 330 °C (decomp.).

**C. Synthesis of N, N<sup>1</sup>-dihexanoyl - N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] (III).**

3,11 g (II) was placed in 15 ml of DMF, 4,3 g of hexane iodide was added dropwise with stirring. The mixture is stirred for 11 hours while heating in a boiling water bath, cooled and washed with 20 ml of water, the precipitate is separated, recrystallized from 50 % alcohol, dried and obtained (III) with a yield of 3,9 g (82,6% of theoretical); Mp = 139-140 °C.

Found, %: C 71,07; H 9,19; N 5,81  
Calculated for C<sub>28</sub>H<sub>44</sub>N<sub>2</sub>O<sub>4</sub>, % : C 71,18; H 9,32; N 5,93

**D. Synthesis of N, N<sup>1</sup>-hexamethylene-N, N<sup>1</sup>-dichloro-bis - [(isobutanoylo) carbamate] (IV).**

3,16 g (0,01 mol) (I), 50 ml of CCl<sub>4</sub>, 18 g of wet alumina are placed, and 4,0 g of calcium hypochlorite are added dropwise at 40 °C over 1 hour.

The reaction mass is left for 23,0 hours. It is filtered off, washed with ether, alcohol, dried, and (IV) is obtained in a yield of 3,57 g (92,3% of theory); Mp = 113-114 °C. Rf = 0,72.

Found, %: C 49,73; H 7,64; N 7,17; Cl 18,36  
Calculated for C<sub>16</sub>H<sub>30</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub>, % : C 49,87; H 7,79; N 7,27; Cl 18,44

**E. Synthesis of N, N<sup>1</sup>-hexamethylene-N, N<sup>1</sup>-dinitrosobis - [(isobutanoylo) carbamate] (V).**

0,7 g of sodium nitrate is added in portions in excess over 4 hours to 3,16 (0,01 mol) (I) dissolved in 80 ml of formic acid (conc.), constantly stirring at a temperature of 0-5 °C. After completion, it is poured into a glass, water is added, the precipitate formed is filtered off, washed with benzene and dried by TLC on Silifol plates, the yield of product (V) is 3,1 g (82,4% of theory); Mp = 340 °C (dec);

Found, %: C 51,21; H 7,89; N 14,83;  
Calculated for C<sub>16</sub>H<sub>30</sub>N<sub>4</sub>O<sub>6</sub>, % : C 51,33; H 8,02; N 14,97.

**VI. CONCLUSION AND FUTURE WORK**

**Growth-stimulating activity of the drug (I)**

To identify the growth-promoting activity of compounds (I) N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate], tests were carried out in the laboratory of the Institute of Plant Chemistry of the Academy of Sciences of the Republic of Uzbekistan under laboratory conditions, seeds of vegetable crops and cotton were used as biotests [17-21].

The experiments used cucumbers of the Uzbekistan-740 variety, tomatoes of the Temp variety and medium-fiber cotton of the S-6524 variety. The preparations were dissolved in DMF and used by the presowing seed lock method for 18-20 hours. The concentrations used were 0,1; 0,01; 0,001; 0,0001; and 0,00001 %. The repetition of experiments was 4-fold. The counts were carried out by measuring the length of the stem and root of 10-day-old seedlings of cotton. It was noted that all preparations tend to stimulate the growth of the root system of young seedlings, both vegetable crops and cotton.

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 new chemical compounds, which is detected by stimulation or 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.

Each series of experiments is accompanied by control. In control variants, only a pure solvent is added to the nutrient medium.

The result of the experiments is recorded 3, 5, 7 and 10 days after inoculation (tables 7-9). Comparative tests show that the test drug (I), that is, a derivative of N, N<sup>1</sup>-hexamethylene bis - [(isobutanoylo) carbamate] of 7,5 to 75,000 times lower concentration than our drug, showed a higher growth-promoting activity than now ROSLYN, a drug used in many sectors of agriculture in Uzbekistan.

Table 7. The effect of the preparation (I) on the germination of seeds and the growth of seedlings of cotton cultivar "S-6524"

Experiences Preparation	Concentration, %	Germination, %	Cottonplant	
			Growth of roots, %	Stem growth , %
Control-water	without	80,0	100,0	100,0
N,N <sup>1</sup> -hexamethylene-bis - [(isobutanoyl) - carbamate]	0,1	84,6	111,6	106,7
	0,01	83,8	124,6	110,5
	0,001	89,5	144,4	119,5
	0,0001	85,7	116,6	117,8
	0,00001	83,4	123,6	118,4
«Roslyn» (famous)	0,75-1,0	80,0	102,6	102,3

The preparation (I) on cotton culture showed biological activity at a concentration of 0,00001% (when diluted 75,000 times). The root growth was stimulated by 129,6%, and the stem growth was 118,4% higher than the ROSLIN preparation (concentration 0,75-1,0%).

The drug (I) on tomatoes, similar to previous cultures, showed a very high biological activity, 125,4% at a concentration of 0,01% (even when diluted 750 times).

The drug AGM-XM-16 on tomatoes, similar to previous cultures, showed a very high biological activity, 151,6% at a concentration of 0,001 (even when diluted 750 times).



Table 8. The effect of the drug (I) on the germination of seeds and the growth of seedlings of tomato varieties "Temp".

Experiences Preparation	Concentration, %	Germination, %	Tomatoes	
			Growth of roots, %	Stem growth, %
Control-water	without	70,0	100,0	100,0
N,N <sup>1</sup> -hexamethylene-bis - [(isobutanoyl) - carbamate]	0,1	90,0	105,1	114,8
	0,01	60,0	129,4	118,4
	0,001	90,0	134,2	127,6
	0,0001	90,0	100,4	114,8
	0,00001	81,0	119,4	106,8
«Roslyn» (famous)	0,75-1,0	89,0	120,9	113,0

The drug (I) on the culture of cucumber, also showed biological activity, at a concentration of 0,001% (that is, when diluted 7500 times). It contributed to root growth of 139,7%, slightly lower than stem growth by 131,0% higher than the control and the well-known drug "ROSLYN" (concentration 0,75-1,0%).

Table 9. The effect of the drug (I) on seed germination and seedling growth of cucumbers of the "Uzbekistan-740" variety.

Experiences Preparation	Concentration, %	Germination, %	Cucumbers	
			Growth of roots, %	Stem growth , %
Control-water	without	100,0	100,0	100,0
N,N <sup>1</sup> -hexamethylene-bis - [(isobutanoyl) - carbamate]	0,1	100,0	109,3	108,6
	0,01	100,0	113,6	112,3
	0,001	100,0	124,5	118,7
	0,0001	100,0	139,7	131,0
	0,00001	100,0	120,6	109,4
«Roslyn» (famous)	0,75-1,0	100,0	103,4	101,7

Thus, the low-toxic (LD≈6130 mg / kg) preparation (I) showed high stimulating properties on the seeds of tomato, cucumbers and cotton at 0,001; 0,0001 and 0,00001% concentration.

### Field tests for growth-promoting activity of the drug (I).

After the initial tests for the growth-promoting activity of the drug (I), field trials were recommended on the farm of S. Agzamov in the Kasbinsky fog of the Kashkadarya and Andijan regions of Uzbekistan from April 2017 to November 2018.

The growth stimulator (I) was tested at various concentrations (from 0,001 to 0,00001 %). The farm used "Temp"tomatoes, "Uzbekistan-740" cucumbers, "C-6524" medium fiber cotton, corn and sunflowers on an area of 110 ha. 230 tons of cotton received additionally. Similarly, good results were obtained on tomatoes, cucumbers, sunflowers and corn.

Thus, the drug (I), recommended by us in a solution of a concentration of 0,001-0,00001 %, surpasses many known drugs in biostimulating activity.

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ISSN: 2350-0328

## International Journal of Advanced Research in Science, Engineering and Technology

Vol. 6, Issue 11, November 2019

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