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Synthesis and research of N,N¹ – hexamethylene bis¹- {bis²- [2,2¹– (phenyl -azo) -1,1¹– (naphthol) – glyceryl -2,2¹ – dioxy-β-ola] -carbamate}, its property and application.

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ABSTRACT: The proposed article relates to organic chemical synthesis and the study of N,N¹- hexamethylene bis¹- {bis²- [2,2¹- (phenylazo) -1,1¹- (naphthol-glyceryl-2,2¹-dioxy-β-ola) -carbamate } , its chemical properties and reactions of dichlorination, dinitrosylation, dibenzilation. The structures of the synthesized compounds were established as, well as a growth promoter of industrial plants. The results of the biological activity of N,N¹- hexamethylene bis¹- {bis²- [2,2¹- (phenylazo) -1,1¹- (naphthol-glyceryl-2,2¹-dioxy-β-ola) -carbamate } are presented. The relationship between structure and activity is established. A stimulating effect on the germination of seeds was noted depending on the concentration. The ways of their practical application are outlined.

KEYWORDS: Carbamate, Hexamethylene diisocyanate, Ortho-aminoacetyl-phenol, N,N¹- dichlorination, dinitrosylation, dibenzilation, Field test.

I. INTRODUCTION

The currently intensively developing chemistry of azo-carbamate compounds attracts the attention of many researchers, both in our Uzbekistan and abroad [1-7]. It is connected, on the one hand, with those rich possibilities of various chemical transformations, which represent azo-, phenyl, naphthyl, and carbamate groups in molecules of organic compounds, and on the other hand, with glycerin groups, which are valuable for practical use of the properties of organic compounds themselves, and also carbamate links.

II. SIGNIFICANCE OF THE SYSTEM

The paper mainly focuses on how the chemistry of azo-carbamate 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

There are many instances where introduction of azo-and glycerol bridge led to the appearance of various types of gammas of biological, pharmacological activity and ability to inhibit the corrosion of metals as well as possible use as antidegradant of rubbers vulcanizing, producing of the intensification solvatoion theory of dyeing and printing of fabrics of natural and chemical fibers in liquid ammonia and organic solvents. Furthermore the study of patterns of the

solid-phase setting of textile dyes, producing theoretical basis for the use of biocatalytic systems for the processes of upgrading textile materials, plasmachemical activity of fiber-forming polymers, the use of high-frequency fields, and microwave radiation in the chemical-textile industry[8-32].

This is due to the high reactivity to complexation of a highly diverse saturated functional group.

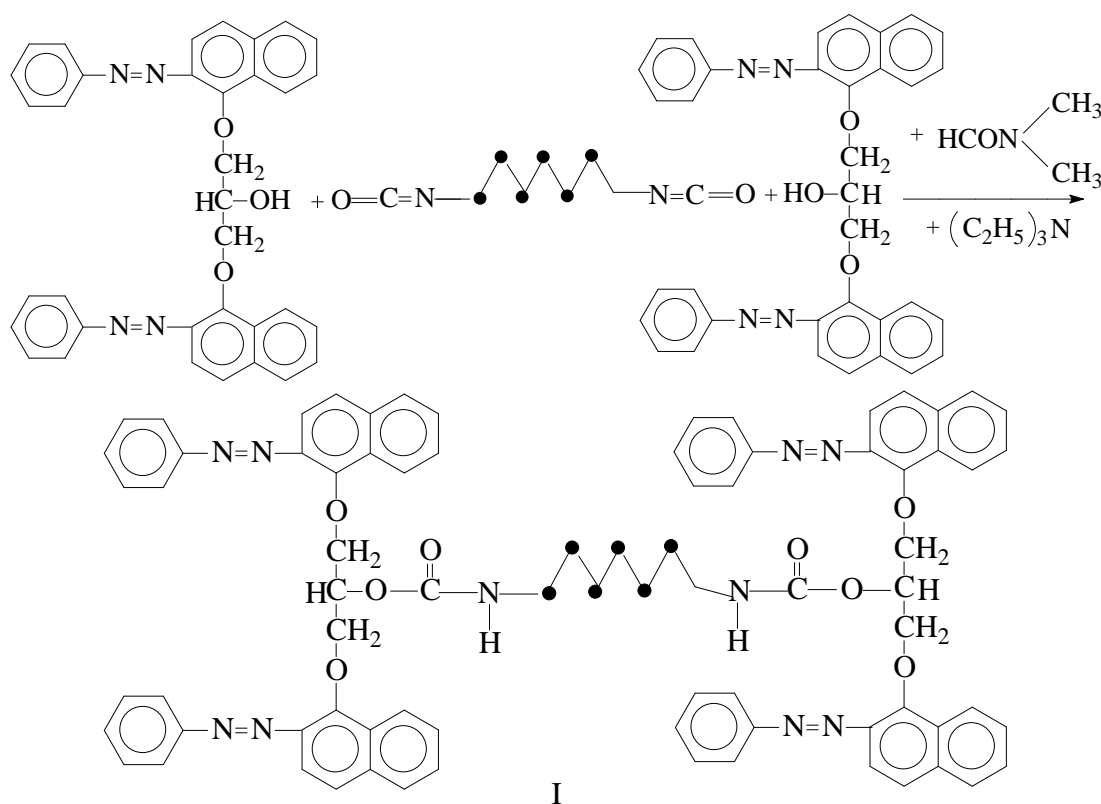
It was necessary to determine high-precision unique optimal methods for the introduction of azo-carbamate groupings into the indicated types of compounds and study the dependence of the used reactions due to the mobile proton at N-H substitute functional groups.

As a result, a new, previously little studied and presented only by simplest examples, the field of chemistry of N-H carbamate compounds, which are derivatives of the tetra-azo-containing bis-carbamates.

IV. PROPOSED METHODOLOGY AND DISCUSSION

The object of the study was the derivatives of N,N^1 - hexamethylene bis¹- {bis²- [2,2¹- (phenylazo) -1,1¹- (naphthol-glyceryl-2,2¹-dioxy- β -ola) -carbamate } . The course of the reaction and the individuality of the compounds are monitored by TLC on aluminum oxide of the II degree of activity with the appearance of spots by iodine vapor. Their physical and chemical properties, biostimulating activity were studied. IR spectra recorded on a VR-20 spectrometer in KBr tablets.

Reacting bis- [2,2¹- (phenyl-azo)-1,1¹- (naphthol)- glyceryl 2,2¹-dihydroxy- β -ola] (ShA-22) with hexamethylene diisocyanate, new derivatives N,N^1 -hexamethylene bis¹-{bis²-[2,2¹-(phenyl-azo)-1,1¹-(naphthyl)]glycerol-2,2¹-dioxy- β -ol}-carbamates were obtained. Cost-effective synthesis was carried out according to the scheme:



The reaction is carried out in the state of dimethylformamide and triethylamine at room temperature (without heating) for 4 hours.

It should be noted that the derivatives of N,N¹-hexamethylene-bis¹-{bis² - [2,2¹-(phenyl-azo)-1,1¹-(naphtholo)-glyceryl-2,2¹-dioxy-β-ol]-carbamates} were obtained with a rather high yield.

Physical-chemical characteristics of the preparation I are shown in table 1.

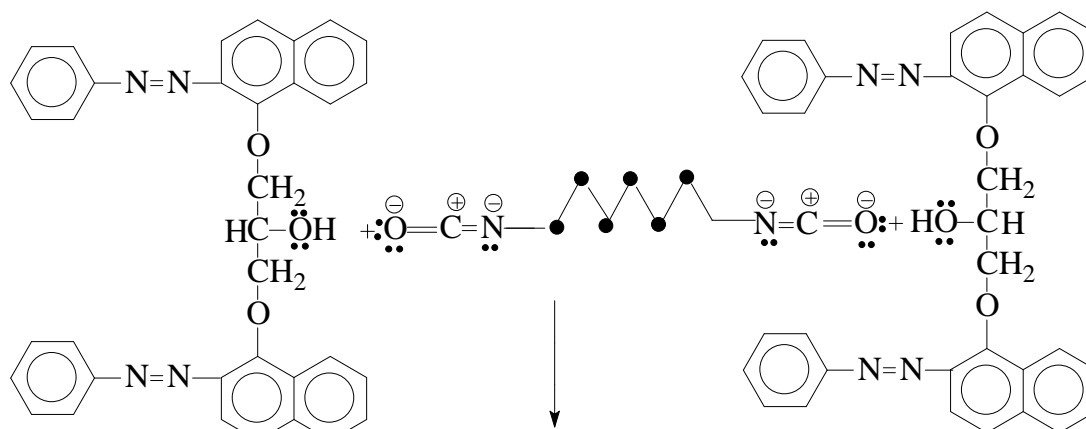
The high density, selectivity and easy mobility of the electron cloud of —N=C=O the group determines its high reactivity. The product yield was 92 %, as expected, products were obtained with good yields by the A_N reaction mechanism.

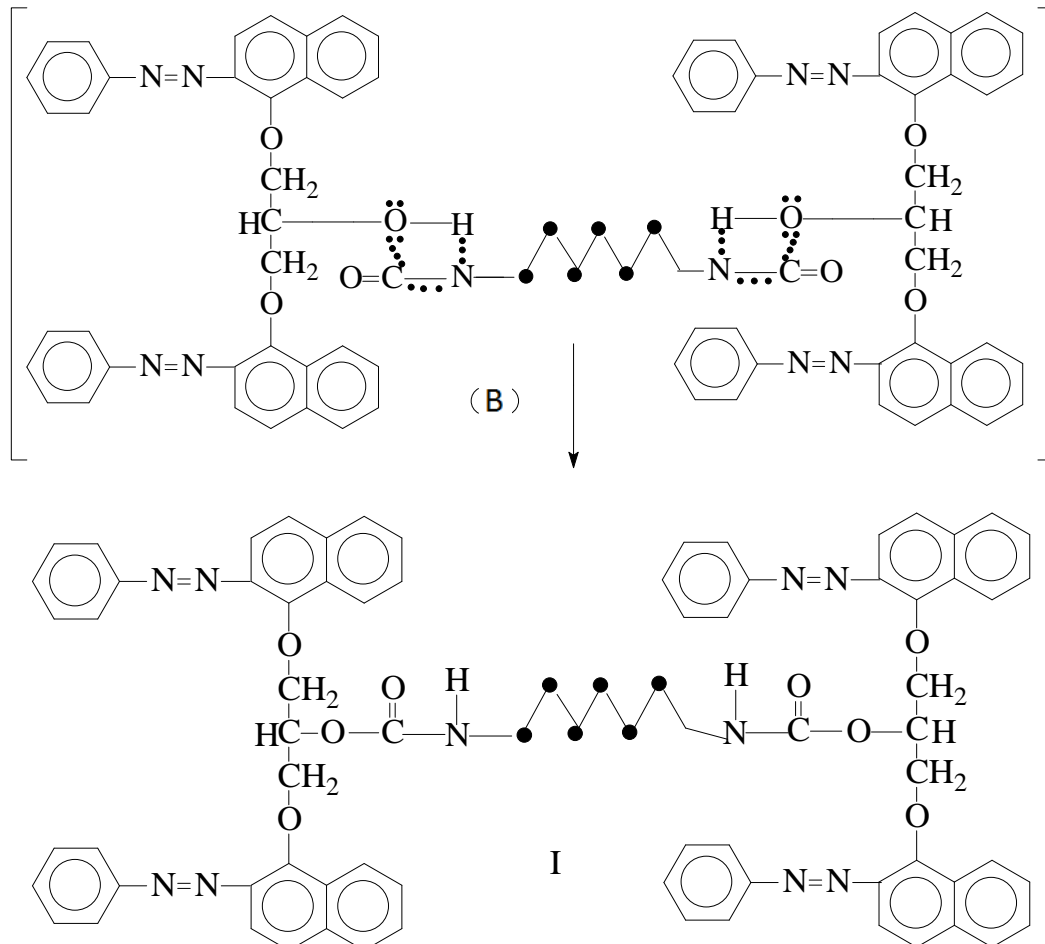
The physico-chemical characteristics of bis-carbamate derivatives are apparently due to the high density and easy mobility of the conjugate (—N=C=O) electron group, which leads to an increase in the positive charge in the carbon atom of the isocyanate group, facilitating the attack of this atom by a nucleophilic agent, and also touching on the issue whether this happens by increasing the positive charge on the carbon atom or by stabilizing the transition state.

Table 1. Physical-chemical characteristics of the preparation I

	Preparation	Yield, %	MT, °C	R _f	Brutto formula	Elemental analysis, %						M _M
						Calculated			Camputed			
						C	H	N	C	H	N	
I	I	92,4	234-235	0,74	C ₇₈ H ₆₈ N ₁₀ O ₈	73,23	5,32	10,95	72,01	5,07	10,84	1278

However, in our cases $\text{—}\ddot{\text{O}}\text{H}$ a hydroxyl group having a free pair attacks the electrophilic center in the isocyanate molecule to formation of intermediate product (B), which then regroups into the final reaction product. Based on our assumptions and literature data, the probably mechanism of the interaction of bis- [2,2¹-(phenyl-azo) -1,1¹-(naphtholo)]-glitceryl-2,2¹-dioxy-β - ola with hexamethylenediisocyanate can be represented by the scheme:




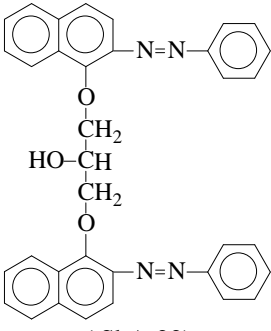




We purify the preparation (ShA-22) using preparative thin-layer chromatography on Al_2O_3 in the system ($HCOOH:CH_3COCH_3:CCl_4=0.5:5.0:0.15$).

To prove the structure of bis-[2,2¹-(phenyl-azo)-1,1¹-(naphthyl)-glyceryl-2,2¹-dioxy-β-ol] (ShA-22), the method of IR- and UV-spectroscopy is used (table2).

Table 2. IR and UV spectra of the preparation (ShA-22)

Structural formula	IR-spectra, ν , cm^{-1}					UV-spectra, nm		
	—OH	—N=N—	—O—CH ₂				—N=N—	Preparation (I) ShA-22
 (ShA-22)	3390	1550	1282	710-690	207	226	264	481

The UV spectrum of bis -[2,2¹-(phenyl-azo)-1,1¹-(naphtholo)-glyceryl-2,2¹-dioxy - β-ol] (ShA-22) has characteristic absorption bands in the regions of 203-208 , 224-228, 260-265, 300-320, 400-440 and 470-490 nm, which corresponds to the structure and name. A band appears in the spectrum in the region of 207 nm, it is caused by a monosubstituted benzene ring, and a naphthalene ring appears in the region of 226 nm, and the azo groups of the phenyl azo-2 naphtholglycidylate appear at 264 nm. The condensed nucleus of naphthalene shifts the absorption maximum specific to aromatics to the long-wavelength region and increases its intensity. The epoxy-containing part of ShA-22 does not contain absorption bands in the long-wavelength part of the spectrum caused by the π-π* transition, which indicates the absence of a double bond in its molecule. The absorption band in the region of 207 nm is due to the excitation of electrons of a monosubstituted benzene ring.

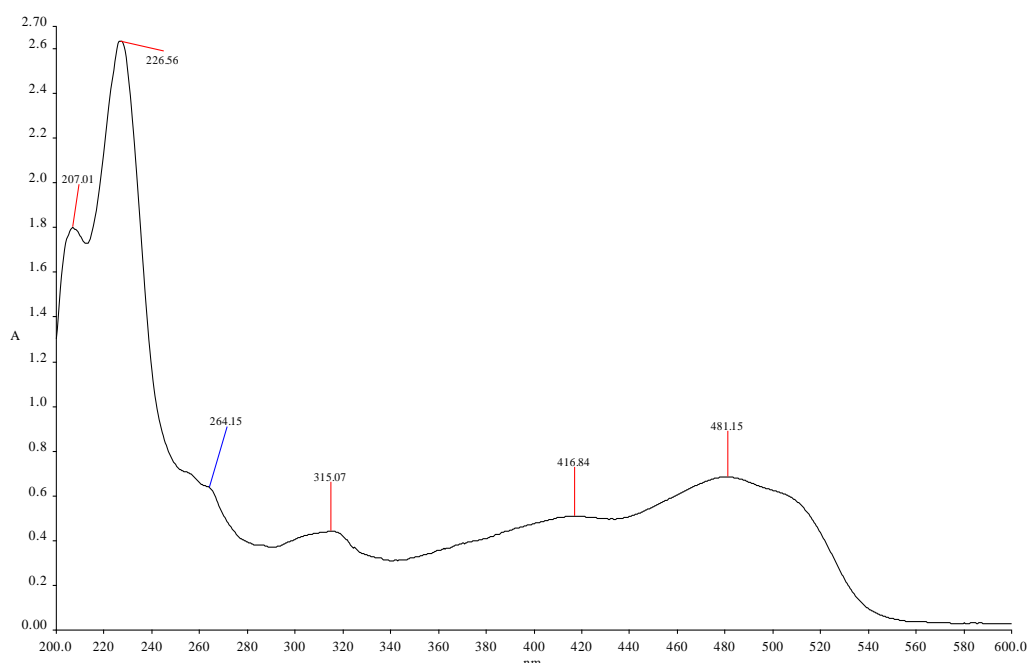


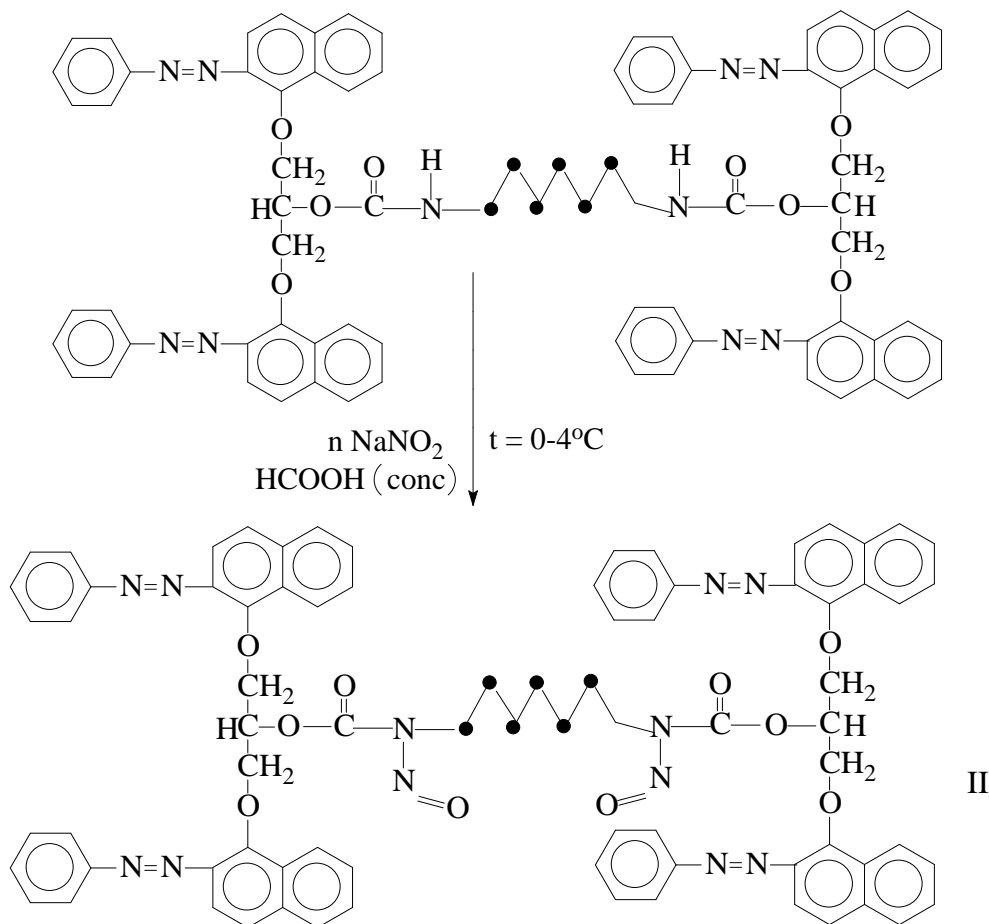
Fig.1: UV spectrum of bis - [2,2¹ - (phenyl-azo) - 1,1¹ - (naphtholo) - glyceryl- 2,2¹ - dioxy-β-ol] (ShA-22)

Chemical transformations of derivatives N,N¹-hexamethlen-bis¹ -{bis² -[2,2¹ - (phenyl-azo)-1,1¹ - (naphtholo)-glyceryl-2,2¹ -dioxy - β -ol]- carbamate} (I).

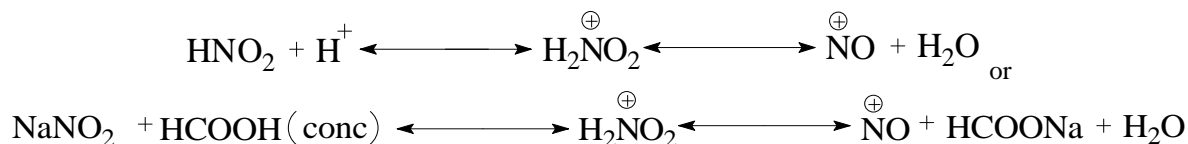
Reactions of N,N¹ - dinitrozoation of the preparation (I).

Reactions of N-N¹ dinitrozoation (I) are comparatively little studied in the world literature [19–34].

According to the literature data and the data of our own research, the nitrogen atoms react connecting directly with the polymethylene chain $\text{-(CH}_2\text{)-}$ by N-nitrosation. As a result of the reaction of N, N¹-dinitrozoation N, N¹-hexamethylene-bis¹-{bis²-[2,2¹-(phenyl-azo)-1,1¹-(naphtholo)-glyceryl-2,2¹-dioxy-β-ol]-carbamate} (I) with NaNO₂ (in excess) in 98 % HCOOH at a temperature of 0-4 °C, N, N¹ -dinitroso substituted (I) were obtained in the yield of 86,4 %. N, N¹ -dinitrozoation proceeds by the electrophilic substitution mechanism (S_E).



The attacking agent is nitrosonium —NO^{\oplus} . Since nitrous acid, which is the most common nitrosating agent, does not exist in its free form, sodium nitrite and strong acid (HCOOH) are used to carry out the process. The resulting nitrous acid, by adding a proton, generates an ion —NO^{\oplus} :



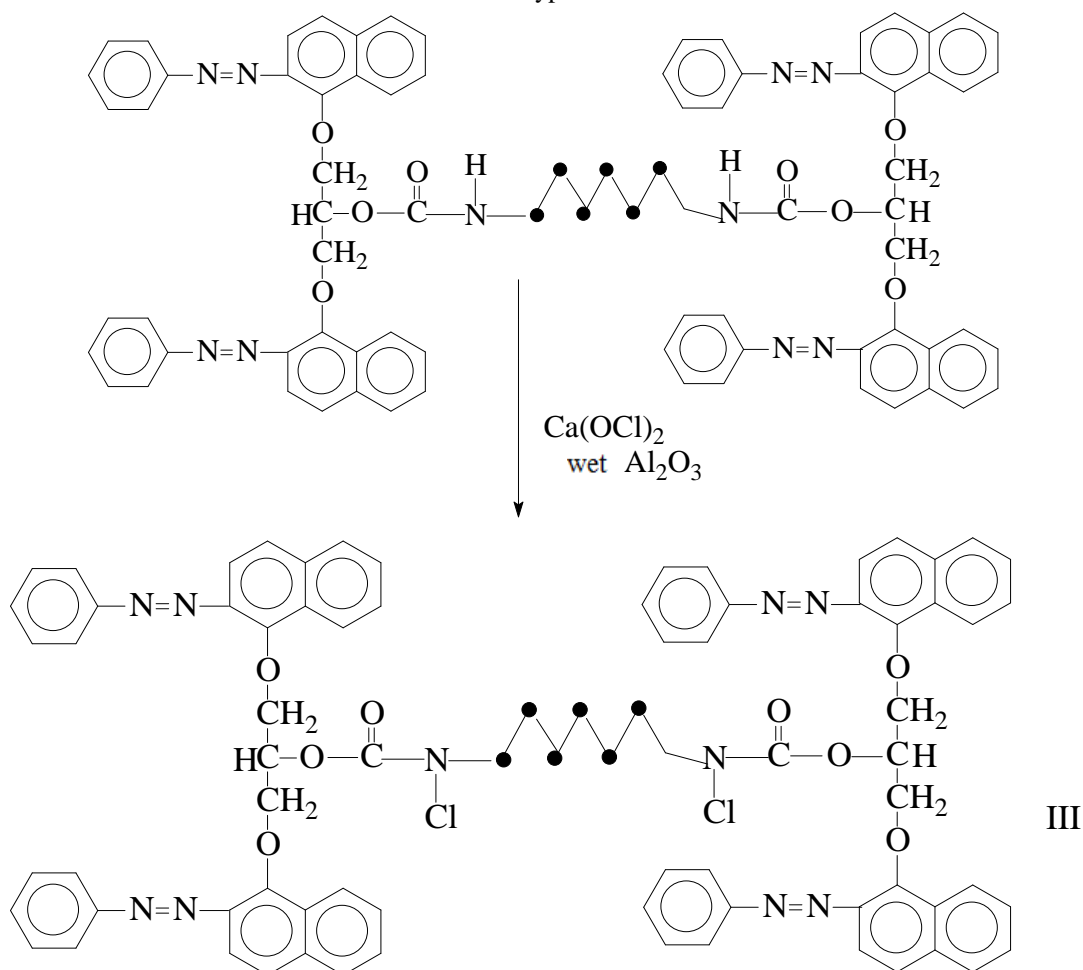
N, N¹-dinitrosation is carried out while cooling the reaction mixture. Increasing the temperature is undesirable because it reduces the yield of the target product, and sometimes affects the direction of the reaction. Identification of N, N¹-dinitroso compounds is carried out in the absorption bands of N—N=O groups. The characteristic band in the region of 1500-1420 cm⁻¹ for N—N=O groups.

Obtaining of N, N¹-dichlorination of the preparation (I).

Derivatives of N, N¹-hexamethylene-bis¹-{bis²-[2, 2¹-(phenyl-azo)-1, 1¹-(naphtholo)-glyceryl-2, 2¹-dioxy-β-ol]-carbamate} (I) are the most valuable raw material for the further synthesis of various biologically active compounds used in

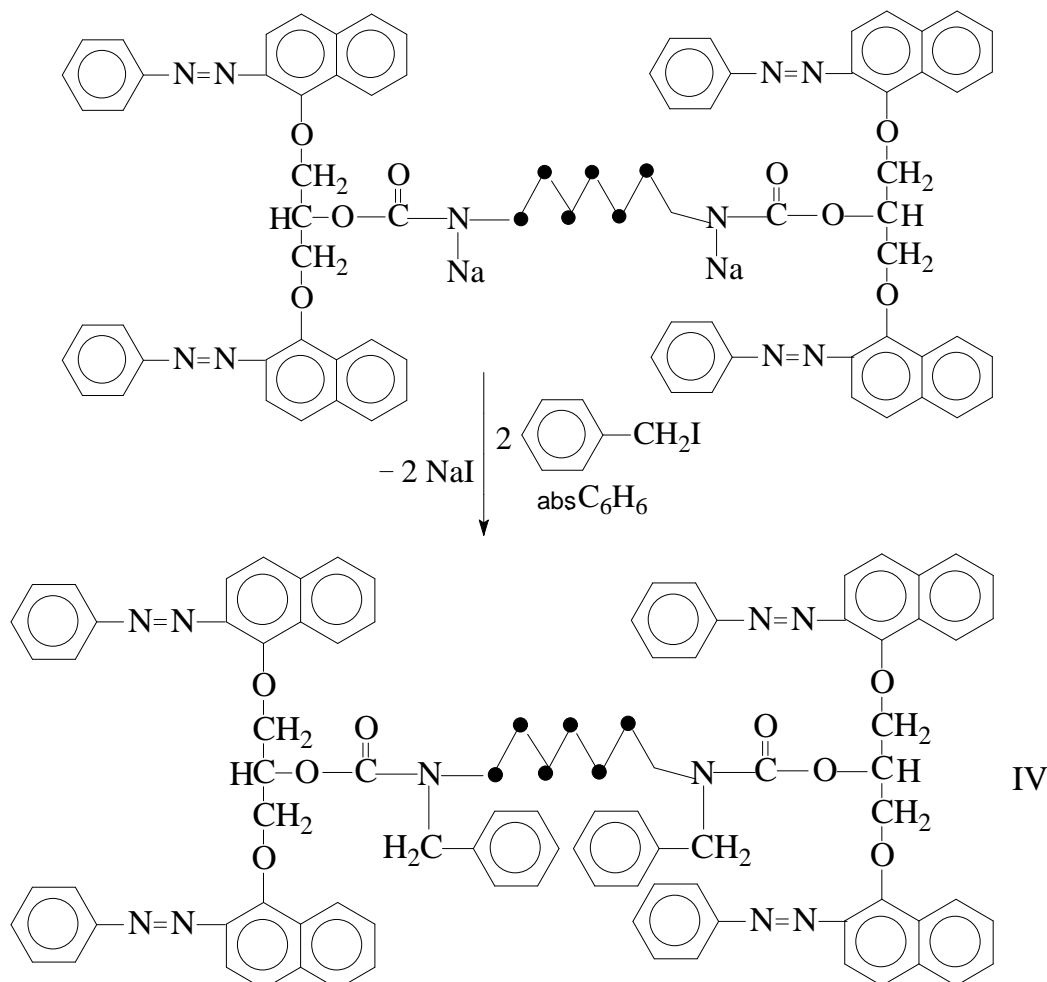
engineering, agriculture, and also have a high reaction center of N-H groups for carrying out reactions of nucleophilic and electrophilic substitutions.

We have developed an efficient, affordable, cheap, stable and environmentally friendly method for carrying out N,N¹ dichlorination of a bis-carbamate derivative with calcium hypochlorite on wet aluminum oxide:



The yield of compounds (III) – 90,4 %; melting temperature = 197-198 °C. Obtaining of N,N¹-dibenzyl substituted derivatives of the preparation (I).

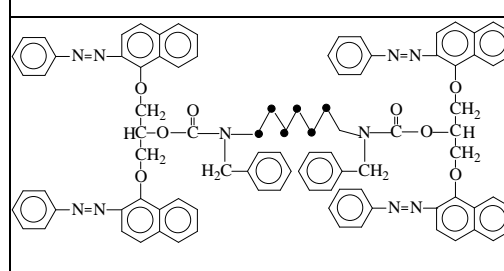
Dibenylation on N-H groups in bis-carbamates (I) by benzyl iodides is of undoubted interest for establishing the reactivity of containing compounds (I). The reaction was carried out by benzylation by the interaction of N,N¹-disodium derivatives of N,N¹-hexamethylene-bis¹-{bis²-[2,2¹-(phenyl-azo)-1,1¹-(naphtholo)-glyceryl-2,2¹-dioxy-β-ola]- carbamate} with benzyl iodide in anhydrous benzene. At temperature of 28-32 °C and with stirring, benzyl iodide was added dropwise over 3,0-3,5 hours according to the following reaction scheme:



The course of the benzylation reaction exclusively on the nitrogen atom N, N¹ - is explained, apparently, by the relatively easy dissociation of sodium in this atom, due to the presence of neighboring carbonyl groups. The yield of the preparation (IV) was 93,4%.

Physical-chemical parameters (IV) are shown in table 4.

Table 4. Physical-chemical parameters of compounds (IV).

Structural formula	Yield, %	MT, °C	R _f	Brutto formula	Elemental analysis, %						
					Calculated			Computed			
					C	H	N	C	H	N	
	93,4	244-245	0,71	C ₉₂ H ₈₀ N ₁₀ O ₈	76,03	5,51	9,64	75,93	5,44	9,82	M _M 1452

V. EXPERIMENTAL RESULTS**Synthesis of N,N¹-hexamethylene-bis¹-{bis²-[2,2¹ - (phenyl-azo)-1,1¹-(naphtholo)- glyceryl-2,2¹ -dioxy-β-ol]- carbamate}.**

In a three-necked flask equipped with a reflux condenser, a thermometer and a stirrer, 11,04g (0,02 mol) bis-[2,2¹ - (phenyl-azo)-1,1¹ - (naphtholo)-glyceryl-2,2¹ -dioxy-β-ol] is put and 20 ml of triethylamine, 40 ml of DMF are added, at room temperature of 30-32 °C while stirring, 3,6 ml (0,01 mol) of hexamethylene-diisocyanate is added dissolving in 10 ml of DMF. The reaction mixture is stirred for 3 hours at a temperature of the reaction mixture of 35-40 °C. After the time, the contents of the flask are transferred to a glass, water is added. The precipitate is washed with TLC. After drying, a colorless powder is obtained. Yield (I) – 12,05 g (92,4% of theoretical); Melting temperature = 234-235 °C. R_f = 0,74.

Computed, %: C 72,01; H 5,07; N 10,84

Calculated for C₇₈H₆₈N₁₀O₈, % : C 73,23; H 5,32; N 10,95

IR-spectra, cm⁻¹ ; $\begin{array}{c} \text{H} \\ | \\ \text{---N---C---O---} \\ || \\ \text{O} \end{array}$ (1573); N—H (3370); COO (1718); $\text{---(CH}_2\text{)}_n\text{---}$ (756-724); NH—CH₂ (1431).

Synthesis of N, N¹-dinitroso, N, N¹-hexamethylene-bis¹ - {bis² - [2,2¹ - (phenyl-azo) -1,1¹ - (naphtholo) - glyceryl-2,2¹ -dioxy - β -ol] - carbamate}.

In a three-necked flask equipped with a reflux condenser, a thermometer and a stirrer, 12,8 g (0,01 mol) of N,N¹ - hexamethylene-bis¹ - {bis² - [2,2¹ - (phenyl-azo) -1,1¹ - (naphtholo) - glyceryl-2,2¹ -dioxy-β-ol] - carbamate} dissolved in 130 ml of dissolved in 100 ml of formic acid. With constant stirring at a temperature of 0-4 °C in portions 0,5 g of sodium nitrite is added in excess for 3,5-4,0 hours. After completion of the reaction, the contents are poured into a one-liter jar, 250 ml of cold water are added. At the same time it starts to precipitate. The precipitate is filtered off, washed with benzene and dried. The individuality of N, N¹ -dinitroso, N, N¹ -hexamethylene-bis¹-{bis²-[2,2¹-(phenyl-azo)-1,1¹-(naphtholo)-glyceryl-2,2¹-dioxy-β-ol]-carbamate} TLC was installed on the plates "Silifol". The yield is 83,6 %; Melting temperature = 248-250 °C (decomp).

Computed, % : C 70,21; H 4,80; N 12,48

Calculated for C₇₈H₆₆N₁₂O₁₀, % : C 70,37; H 4,96; N 12,63

Synthesis of N, N¹ -dichloro, N, N¹ -hexamethylene-bis¹ - {bis² - [2,2¹ - (phenyl-azo) -1,1¹ - (naphtholo) - glyceryl-2,2¹ -dioxy - β -ol] - carbamate}.

12,8 g (0,01 mol) N, N¹-hexamethylene-bis¹ - {bis² - [2,2¹ - (phenyl-azo) -1,1¹ - (naphtholo) - glyceryl-2,2¹ -dioxy-β-ol] - carbamate} 60 ml of CCl₄, 25 g of wet alumina and 6,0 g of calcium hypochlorite are added dropwise at a temperature of 40 °C for 1 hour. Then the reaction mass is left for 24 hours, filtered, the residue is washed with ether, alcohol. It is dried and N, N¹-dichloro-, N, N¹-hexamethylene-bis¹-{bis²- 2,2¹-(phenyl-azo)-1,1¹-(naphtholo) -glyceryl-2,2¹-dioxy-β-ol]-carbamate} is obtained. The yield of the product (III) -12,17, (90.4% of theoretical); Melting temperature = 197-198 °C. R_f = 0.68; M_m = 1341.

Computed, % : C 69,64; H 4,79; Cl 15,21; N 10,36

Calculated for C₇₈H₆₆Cl₂N₁₀O₈, % : C 69,79; H 4,92; Cl 5,29; N 10,44

To prove the structure of N, N¹- dichloride I (III), elemental analysis was carried out with silver salts (AgNO₃ solution).

Field tests on the growth-promoting activity of N, N¹-hexamethylene-bis¹ - {bis²-[2,2¹-(phenyl-azo)-1,1¹-(naphtholo)-glyceryl-2,2¹ -dioxy - β -ol] - carbamate}(I).

Laboratory and field tests of this preparation (I) were carried out as stimulants for the growth of cotton, cucumber and tomato plants. For comparison, the well-known stimulator "Rostlin" was used. It was established that N, N¹-



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hexamethylene-bis¹-{bis²-[2,2¹-(phenyl-azo)-1,1¹-(naphthol)-glyceryl-2,2¹-dioxy- β-ol] -carbamate} has a growth-promoting activity, at a concentration of 0,001 % is the most highly efficient growth promoter for vegetable crops and cotton in plot conditions.

Comparative tests have shown that our preparation (I) exceeds the "Rostlin" stimulator in the germination of cotton seeds and the development of its root system by almost 2, 5 times.

VI. CONCLUSION AND FUTURE WORK

Further in-depth study in the field conditions on S.Azamov's farm in Kasbinsk district of Kashkadarya region, as well as in a number of farms in Andijan and Navoi regions of Uzbekistan, was recommended: from April to November 2019-2020.

An additional 11,205 tons of cotton was harvested, which is about 4,3 billion sums only for cotton production. Similarly, interesting results were obtained on cucumbers, sunflowers, soybeans, corn and tomatoes.

Thus, the preparation (I), recommended by us in a solution of 0,001% of concentration, exceeds many known preparations in biostimulating activity and is even less toxic (I) (LD₅₀ = 3910 ppm / kg).

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