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SYNTHESIS AND PROPERTIES OF 1-(PHENYL) – AZO – (2^1 – HYDROXY-3^1-CHLORO-PROPYLOXY) NAPHTHOL-2

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ABSTRACT

The article describes the synthesis of derivatives of azo-compounds by the interaction of aniline with nitrososarcosine reagents. Synthesized anti-1^1 (phenyl -azo) -naphthol-2, bis [1- (phenyl) -azo- (2-naphthoxy)] -propanol-2^1, chemical properties are studied, the biostimulating properties of the obtained compounds are tested.

KEYWORDS: azo compounds, aniline, diazotization, phenyl diazonium, alkylation, biological activity.

I. INTRODUCTION

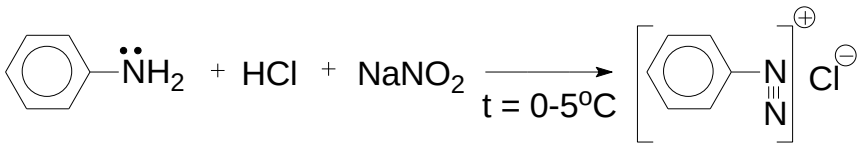
At present practically all branches of the national economy use substances of azo-compound, which are considered important reagents for coloring synthetic, natural and chemical fibers, plastics, film, paper, wooden objects, food products, leather raw materials, etc. [1-5].

II. MATERIALS AND METHODS

The object of the study are the derivatives of azo compounds, the study of the process and the conditions for their synthesis. IR spectral analyzes were obtained with a Spekord-75. It was studied the biostimulating effect of the preparation on the germination and growth of seedlings of agricultural crops. Staining of polymers is carried out according to GOST20-282-04.

III. RESULTS AND DISCUSSION

To create new technologies for the derivatization of azo compounds, reactions of aniline with nitrosating reagents ($NaNO_2$ + HCl) are carried out. The reactions are carried out at a reduced temperature (0-5 ° C). The scheme of the chemical reaction is as follows:



The mechanism of the reaction of diazotization is very complex and little studied. We consider that the intermediate product for carrying out our reaction is N-nitrosamine:

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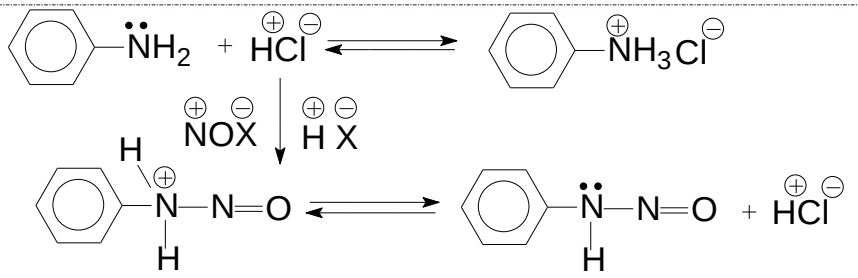
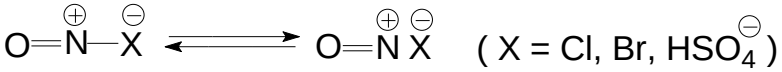
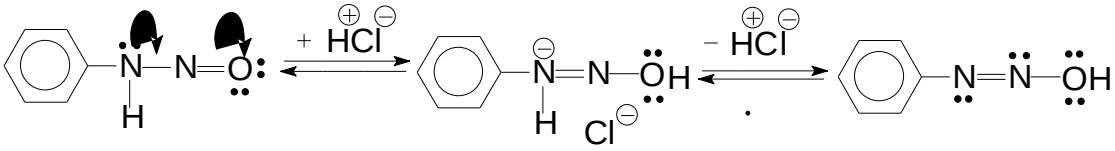


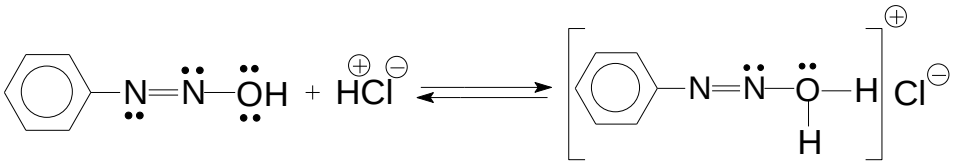
Then, upon interaction with $NaNO_2$ acid it forms HNO_2 nitrous acid, which, in our opinion, passes into the nitrosyl derivativesin an acidic environment:



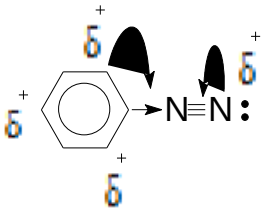
Further, N-nitrosamine of aniline being in an acidic environmenteasilypasses into its tautomeric form - diazohydroxide of aniline:



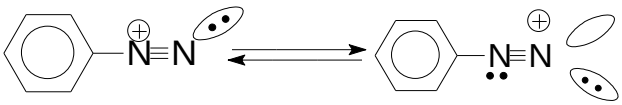
which in an acidic environment turns into a phenyldiazonium cation.



Diazonium salts are usually obtained in aqueous solutions, and directly used for further reactions with naphthol-2. Phenyldiazonium salts are colorless crystalline substances soluble in water. The compounds of phenyldiazonium salts are unstable and easily decompose, since the phenyldiazonium ion is a conjugate ion, which a strong interaction between the electronic phenyl system and the triple bond of the diazonium group is realized in. The chemical interaction can be represented by the following scheme:

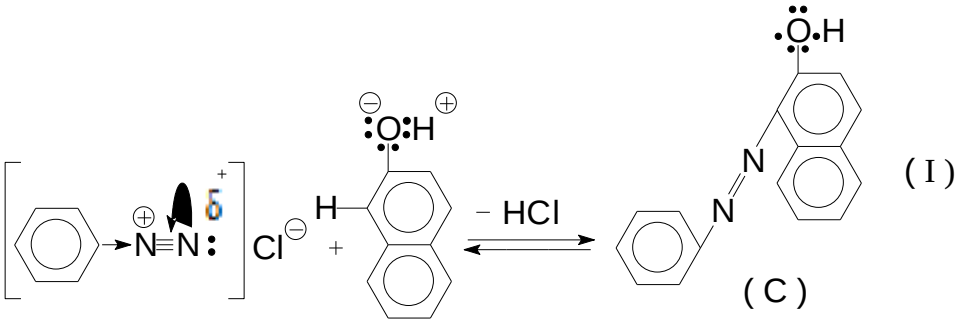


Apparently, both nitrogen atoms are in the state of Sp - hybridization, and one of the atoms has a positive charge. This causes the electronic displacement in the anilindiazonium grouping:



In this scheme, the second nitrogen atom acquires a certain positive charge. This can be represented by a mesomeric boundary structure, in which the positive charge is completely localized on a given nitrogen atom. The second nitrogen atom is the main center of the reaction in the transformation of diazonium salts.

The diazonium group belongs to the strongest EA groupings in organic compounds, it has a greater effect compared to the nitrogroup, therefore, the phenyldiazonium ion is a strong electron acceptor.
Also, the cation of phenyldiazonium, as an electrophilic particle with ED reagents, can form a complex with charge transfer and further- the product of addition at the nitrogen atom:

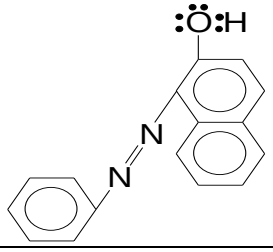


anti-1[1] (phenyl-azo) -naphthol-2

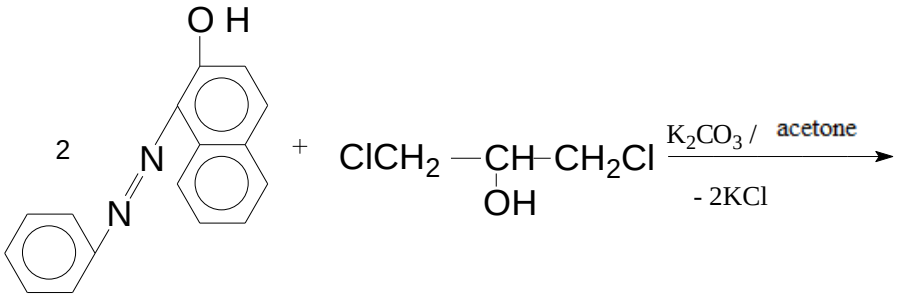
Phenyldiazonium salts react with nucleophilic ions (C) to form diazo derivatives, in which azo compounds are formed, i.e. there is a reaction of electrophilic substitution S_E in naftalina cycle.
Physicochemical parameters of the derivative of anti-α-(phenylazo) -β-naphthol are given in Table 1.

Table 1

Physical and chemical characteristics of the dye (I)

Structural formula	Output, %	T_{PL}, °C	R_f	Gross formula	Elemental analysis, %		IR- spectra, cm^{-1}			M_M
					Calculated	Found	-N=N-	-OH		
					N	N				
	89,4	95	0,71	$C_{16}H_{17}N_2O$	11,29	11,14	1584	3319	748	248

The structure of the obtained azo-dye (I) is established due to analytical data and IR spectroscopy. In the IR spectrum of the azo-dye in the region of 1584 cm^{-1}, there are observed characteristic for -N=N – group absorption bands, 3319 cm^{-1} for (-OH).
To prove the presence of hydroxyl groups, we carried out alkylation reactions using 1,3-dichloropropanol-2 in a known manner according to the following scheme:



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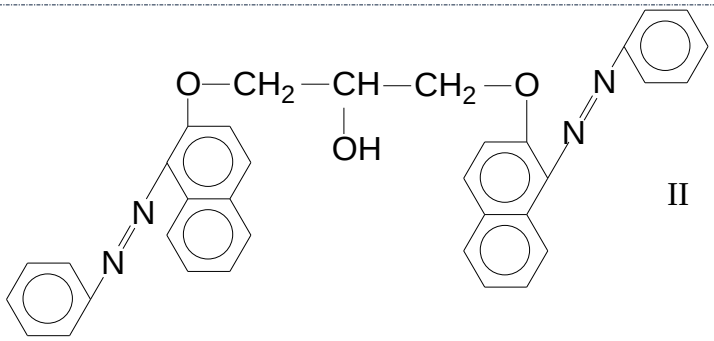


II

Bis - [1- (phenyl) -azo- (2-naphthoxy)] -propanol-2[1]

Synthesized bis - [1- (phenyl) -azo- (2-naphthoxy)] -propanol-2[1] with 88 % yield. This substance is a colored powder with a melting point of 147-148 °C.

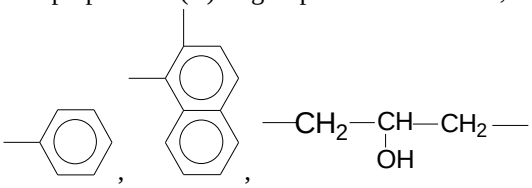
The structure of the obtained compounds is confirmed by elemental analysis and IR, UV - spectroscopy. The IR spectrum of the preparation (II) contains characteristic absorption bands in the region of 1400-1500 cm^{-1} and 1200 cm^{-1} corresponding to the -N = N- groups of the azo compound. The deformation valence vibrations of some aromatic groups are manifested in the form of strong bands between ordinary bands, deformation vibrations in the region of 3000-3300 cm^{-1} and 1100-1500 cm^{-1}, the character of substitution is determined by strong absorption below 900 cm^{-1}.

During the study of the IR spectrum dimer parameters 1051-920 cm^{-1} correspond to the-OH group and 1134 cm^{-1} asymmetric valence oscillations -C-O-C-correspond to the group of ethers. The UV spectrum of bis - [1-(phenyl)-azo-(2 –naphthoxy)]-propanol–2[1] have characteristic absorption bandsin the range of 203-208, 224-228, 260-265, 300-320, 400-440 and 470-490 nm, that corresponds to phenyl-azo-naphthol-propanol.

In the spectrum there is a band in the region of 207 nm causing a single-substituted benzene ring, and in the region of 226 nm there is a band of naphthalene ring, at 264 nm there is a band of azo-group phenyl-azo-naphthoxy-propanol-2[1]. In the area of 416 nm, only one large band is manifested, which undergoes a batochromic shift to 481 nm phenyl-azo-naphthoxy - propanol-2[1].

The condensed nucleus of naphthalene displaces the specific for aromatic compounds maximum of absorption to the long-wave region and increases its intensity. The absorption band in the region of 207 nm is due to the excitation of electrons of a single-substituted benzene ring.

The presence in the molecule of the preparation (II) of groups such as -N = N-, -O-CH_2



and others together contributes to an increase of the biological activity of synthesized by us compounds.

There were carried out tests for the initial assessment of the growth-regulating effect of the chemical preparation (II) on the germination and growth of crop seedlings in the laboratory conditions. The tests were conducted with the purpose of the experimental determination of the sensitivity of the cultures to the action of the drug and establishing the optimal effective concentration.

The preparation is a slightly soluble in water colored powder (II). It is known that those concentrations, under the action of which there is maximum stimulation significantly different from the control one, can be taken as stimulating ones. The biological activity of the preparation (II) was studied in triple repetition and in three concentrations (0,01, 0,001, 0,0001%), while it was dissolved with a DMFA solvent.

Table 2

Biotest of tomatoes (Temp)

Name of the preparation	Concentration, %	Seed germination, after 5 days, %	Growth of seedlings on the 10 day, %	
			root	stem
Control	H_2O; w/t	70,0	100,0	100,0
Bis - [1- (phenyl) -azo- (2-naphthoxy)] -propanol-2[1] (II)	0,01	60,0	110,0	147,0
	0,001	78,0	121,5	165,0
	0,0001	86,0	106,0	130,0

The study of the preparation (II) for growth-stimulating activity on tomato seeds showed that at a concentration of 0,001% it increased by 65% compared to the control one (table.2).

Table 3

Biotest of cucumbers (variety "Uzbekistan")

Name of the preparation	Concentration, %	Seed germination, after 5 days, %	Growth of seedlings on the 10 day, %	
			root	stem
Control	H_2O; w/t	71,3	100,0	100,0
Bis - [1- (phenyl) -azo- (2-naphthoxy)] -propanol-2^1 (II)	0,01	93,0	106,1	108,9
	0,001	71,3	104,3	125,0
	0,0001	85,9	108,8	107,7

The study of the preparation (II) for the growth-stimulating activity on cucumber seeds showed that at a concentration of 0,001% it increased by 25% compared to the control one (table.3).

Table 4

Biotest of cotton (variety "6524")

Name of the preparation	Concentration, %	Seed germination, after 5 days, %	Growth of seedlings on the 10 day, %	
			root	stem
Control	H_2O; w/t	87,5	100,0	100,0
Bis - [1- (phenyl) -azo- (2-naphthoxy)] -propanol-2^1 (II)	0,01	100,0	106,1	105,4
	0,001	100,0	104,6	107,1
	0,0001	100,0	116,5	110,4

The study showed that the studied preparation(II) for cotton is the most effective for growth-regulating activity in the laboratory conditions.
Further in-depth study in the field is needed.

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