Original Article

Synthesis Of New Propargyl Ester Derivatives And Biostimulation Activity Of 4-(Bis(2-Hydroxyethyl)Amino)But-2-Yn-1-Yl Butyrate

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Abstract

The proposed article relates to synthesis and the study of new propargyl ester derivatives, and biostimulation activity of 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate. Aminomethylation reactions of the obtained propargyl ester with ethanolamine and paraform were carried out in the presence of a catalyst. Copper salts were used as a catalyst. The structure of the new propargyl ester derivatives was established by IR and ^{1}H-, ^{13}C- NMR spectroscopy. Comparative tests show that the test derivative of the drug 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate showed a higher growth-promoting activity.

Keywords: propargyl ester, 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate**,** propargyl butyrate, synthesis, 1,4-dioxane, Mannich reaction, aminomethylation, paraform, copper (I) chloride, copper (II) chloride, copper (II) acetate, hexane, ethyl acetate, diethanolamine.

I. INTRODUCTION

Currently, the world considers it an important task to create targeted methods for the synthesis of highly effective biologically active compounds and successfully apply them in various areas of the national economy. In this regard, it is important to create inexpensive and effective local preparations, further improving their physicochemical and biological properties.

II. SIGNIFICANCE OF THE SYSTEM

The paper mainly focuses on how the chemistry derivatives 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl. The study of literature survey is presented in section III, materials and methods, and the experimental results of the study proposed methodology and discussion is explained in section IV, section V covers Conclusion.

III. LITERATURE SURVEY

In recent years, according to the analysis of articles in prestigious scientific journals of the world, many experimental works have been carried out on the introduction of triple bonds into natural and synthetic organic substances, polymers and the implementation of their chemical modifications [1, 3, 21].

Substances with a triple bond and their derivatives are used in pharmaceuticals as anti-inflammatory, antibacterial, antifungal agents; in agriculture as herbicides, insecticides, fungicides; in industry they are used as anti-corrosion agents for metals [2, 4-7, 9, 11, 14-15, 17, 20].

It is known that the introduction of triple bonds into a molecule increases the biological activity of a substance, reducing its toxicity. In addition, there are many chemical reactions that occur with terminal alkynes (cyclization, halogenation, hydrogenation, combination, exchange), which allows you to study in depth the chemical properties of this substance. We have done a lot of work on the synthesis of compounds with a triple bond and their chemical transformations, and several new biologically active substances have been identified [8, 10, 12-14, 16, 18-21]. The research results given in these sources indicate that the study of the aminomethylation process and the biological activity of new derivatives obtained on the basis of the Mannich reaction of propargyl carboxylic acid esters with secondary amines is relevant.

IV. PROPOSED METHODOLOGY AND DISCUSSION

MATERIALS AND METHODS

Solvents: used ethyl acetate, ethyl alcohol, hexane, acetone, benzene, toluene. The structure of the compounds has been reliably analyzed based on the results obtained using modern IR, ^{1}H and ^{13}C NMR spectroscopy. The progress of the reactions was monitored by thin layer chromatography (TLC). The eluent was a mixture of hexane : ethyl acetate - 7:1. The melting points of the synthesized compounds were determined on a MEL-TEMP instrument (USA).

EXPERIMENTAL PART

Synthesis of 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6).

1.26 g (1.34 ml, ρ = 0.94 g/ml, 0.01 mol) of propargyl butyrate, 0.45 g (0.015 mol) of paraform (1), 1.05 g (0.96 ml, ρ= 1.097 g/ml 0.01 mol) diethanolamine, 0.05 g (0.27 mmol) copper (II) acetate and 25.0 ml 1,4-dioxane. The flask was placed in an oil bath, a reflux condenser was connected and refluxed at the boiling point of dioxane (101-102°C) for 6 hours. The progress of the reaction was monitored by TLC. After cooling, the reaction mixture was poured with 30 ml of 5% hydrochloric acid and extracted twice with 30 ml of petroleum ether. After separating the petroleum ether layer, the aqueous part was neutralized with ammonia. Then it was extracted twice more with 30 ml of petroleum ether. Part of the petroleum ether was separated and dried over sodium sulfate. After separation, the petroleum ether was distilled using a rotary evaporator. The product (6) weighing 2.05 g was obtained with a yield of 84%. T_{boil} = 172-174 ^{o}C (8 mm Hg). R_f = 0.66 (hexane:ethyl acetate - 5:1 system).

^{1}H NMR (400 MHz, $CDCl_3$): 0.88 (3H, t, J=7.34, $C\underline{H_3}$-), 1.32 (2H, m, CH_3-$\underline{CH_2\text{-}}$), 1.59 (2H, t, J=5.88., -CH_2-$\underline{CH_2\text{-}}$), 2.31 (2H, t, J=7.28, -$\underline{CH_2}$-CH_2-OH), 2.44 (2H, t, J=7.14, $\underline{\text{- }CH_2}$-$CH_2$-OH), 2.70 (2H, t, J=5.26.,-$\underline{CH_2}$-N<), 2.98(2H, s,$\underline{\text{- OH}}$), 3.61 (4H, t, J=5.20, - CH_2-$\underline{CH_2}$-OH), 4.65 (2H, t, J=4.10, -O-$\underline{CH_2}$-)

^{13}C NMR ($CDCl_3$): 15.24, 21.11, 38.54, 48.78, 56.15, 60.21, 62.80, 85.47, 85.734, 170.11.

IR-spectrum (KBr, v, cm^{-1}): 2958 (CH_3), 2932, 2873 (CH_2), 1742 (>C=O), 1244 (-C-N-), 1166 (-C-O-C-), 1109 (-C-OH).

Synthesis of 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl valerate (7).

Similarly, 1.40 g (1.52 ml, ρ= 0.92 g/ml, 0.01 mol) propargyl valerate, 0.45 g (0.015 mol) paraform (1), 1.05 g (0.96 ml, ρ= 1.097 g/ml 0.01 mol) diethanolamine, 0.05 g (0.27 mmol) copper(II) acetate, and 25.0 ml 1,4-dioxane. The product (7) was obtained with a mass of 2.11 g and a yield of 82%. T_{bp}=176-178 ^{o}C. (8 mm Hg) R_f=0.68 (hexane:ethyl acetate system - 5:1).

^{1}H NMR (400 MHz, $CDCl_3$): 0.88 (3H, t, J=7.18, $C\underline{H_3}$-), 1.26 (4H, m, CH_3-$\underline{(CH_2)_2\text{-}}$), 2.12 (2H, t, J=5.36, -$CH2_2$-$\underline{CH_2\text{-}CH2\text{ -}}$), 2.46 (4H, t, J=6.14,-$\underline{CH_2}$-$CH_2$-OH), 2.81 (2H, t, J=6.18, -$\underline{CH_2}$-N<), 3.06 (2H, s, $\underline{\text{- OH}}$), 3.58 (4H, t, J=5.26, - CH_2-$\underline{CH_2}$-OH), 4.81 (2H, t, J=5.41, -O-$\underline{CH_2}$-)

^{13}C NMR ($CDCl_3$): 16.24, 22.65, 26.37, 38.35, 49.86, 58.04, 59.88, 61.16, 75.87, 85.48, 175.24.

IR spectrum (KBr, ν, cm^{-1}): 2961 (-CH_3), 2923, 2867 (-CH_2-), 1742 (>C=O), 1243 (-C-N-), 1159(-C-O-C-), 1112 (-C-OH),

Synthesis of 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl capronate (8).

By analogy with the above methods of synthesis, 1.54 g (1.69 ml, ρ = 0.91 g / ml, 0.01 mol) of propargyl valerate, 0.45 g (0.015 mol) of paraform (1), 1.05 g (0.96 ml, ρ= 1.097 g / ml, 0.01 mol) diethanolamine, 0.05 g (0.27 mmol) copper(II) acetate, and 25.0 ml 1,4-dioxane. Product (8) weighing 2.16 g was obtained with a yield of 80%. T_{bp}=184-185°C. (8 mm Hg) R_f=0.69 (hexane:ethyl acetate - 5:1 system).

^{1}H NMR (400 MHz, $CDCl_3$): 0.87 (3H, t, J=7.16, -$C\underline{H_3}$-), 1.22 (6H, m, CH_3-$\underline{(CH_2)_3\text{-}}$), 2.15 (2H, t, J=5.44, -CH_2-$\underline{CH_2\text{-}}$), 2.49 (4H, t, J=6.16, -$\underline{CH_2}$-CH_2-OH), 2.83 (2H, t, J=6.14, -$\underline{CH_2}$-N<), 3.07 (2H, s, , $\underline{\text{-OH}}$), 3.66 (4H, t, J=5.28, -CH_2-$\underline{CH_2}$-OH), 4.76 (2H, t, J=5.23, -O-$\underline{CH_2}$-)

^{13}C NMR ($CDCl_3$): 15.21, 20.81, 25.74, 31.71, 38.36, 49.81, 57.26, 59.98, 60.86, 85.97. 84.69, 176.12.

IR spectrum (KBr, v, cm^{-1}): 2961 ($-CH_3$), 2926, 2874 ($-CH_2-$), 1751 (>C=O), 1241 (-C-N-), 1168 (-C-O-C-), 1109 (-C-OH).

Synthesis of 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl enanthate (9).

Synthesis was carried out according to the method described in previous cases. For this, 1.68 g (1.83 ml, ρ= 0.916 g/ml, 0.01 mol) propargyl valerate, 0.45 g (0.015 mol) paraform (1), 1.05 g (0.96 ml, ρ= 1.097 g/ml, 0.01 mol) diethanolamine , 0.05 g (0.27 mmol) of copper(II) acetate, and 25.0 ml of 1,4-dioxane. Product (9) weighing 2.30 g was obtained with a yield of 81%. T_{bp}=196-198 ºC. (8 mm Hg) R_f=0.72 (hexane:ethyl acetate - 5:1 system).

^{1}H NMR (400 MHz, $CDCl_3$): 0.87 (3H, t, J=7.08, $C\underline{H_3}-$), 1.21 (6H, m, $CH_3-\underline{(CH_2)_3-}$), 1.44 (2H, m, $-CH_2-\underline{CH_2-}$), 2.16 (2H, t, J=5.38, $-CH_2-\underline{CH_2-}$), 2.47 (4H, t, J=6.84, $-\underline{CH_2}-CH_2-OH$), 2.81 (2H, t, J=6.12, $-\underline{CH_2}-N<$), 3.06 (2H, s, , $\underline{-OH}$), 3.67 (4H, t, J=5.32, $-CH_2-\underline{CH_2}-OH$), 4.75 (2H, t, J=5.06, $-O-\underline{CH_2}-$)

^{13}C NMR ($CDCl_3$): 15.14, 20.92, 26.86, 31.44, 39.47, 49.83, 57.15, 58.94, 61.17, 86.16. 84.68, 175.57.

IR spectrum (KBr, ν, cm^{-1}): 2959 ($-CH_3$), 2931, 2872 ($-CH_2-$), 1743 (>C=O), 1232 (-C-N-), 1167 (-C-O-C-), 1108 (-C-OH),

Synthesis of 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl caprylate (10).

Synthesis was carried out by a similar method. We took 1.82 g (2.02 ml, ρ= 0.90 g/ml, 0.01 mol) propargyl valerate, 0.45 g (0.015 mol) paraform (1), 1.05 g (0.96 ml, ρ= 1.097 g/ml 0.01 mol) diethanolamine, 0.05 g (0.27 mmol) of copper (II) acetate and 25.0 ml of 1,4-dioxane. Product (10) weighing 2.36 g was obtained with a yield of 79%. T_{bp}=211-213ºC. (8 mm Hg) R_f =0.74 (hexane:ethyl acetate - 5:1 system).

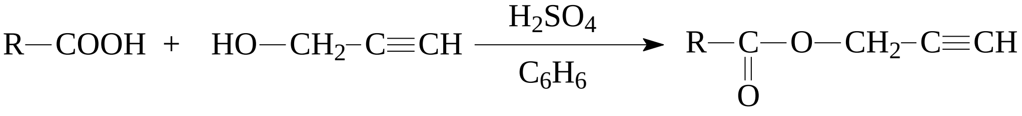
^{1}H NMR (400 MHz, $CDCl_3$): 0.84 (3H, t, J=7.18, $-C\underline{H_3}-$), 1.22 (8H, м, $CH_3-\underline{(CH_2)_4-}$), 1.48 (2H, м, $-CH_2-\underline{CH_2-}$), 2.12 (2H, t, J=5.36, $-CH_2-\underline{CH_2-}$), 2.34 (4H, t, J=7.16, $-\underline{CH_2}-CH_2-OH$), 2.77 (2H, t, J=5.34, $-\underline{CH_2}-N<$), 3.01 (2H, s, , $\underline{-OH}$), 3.65 (4H, t, J=5.20, $-CH_2-\underline{CH_2}-OH$), 4.72 (2H, t, J=4.12, $-O-\underline{CH_2}-$)

^{13}C NMR ($CDCl_3$): 14.71, 21.11, 25.26, 29.59, 31.72, 38.79, 48.75, 56.48, 59.85, 62.66, 85.17, 84.71, 174.27.

IR spectrum (KBr, ν, cm^{-1}): 2960 ($-CH_3$), 2933, 2871 ($-CH_2-$), 1746 (>C=O), 1239 (-C-N-), 1165 (-C-O-C-), 1110 (-C-OH).

The results obtained and their analysis.

Analyzing the literature published in recent years, the aim of this work was the synthesis of some propargyl esters of a saturated monocarboxylic acid, carrying out aminomethylation reactions with diethanolamine and paraform. First, at the first stage, the corresponding propargyl esters (3) were synthesized from monocarboxylic acid and propargyl alcohol. The main method for the synthesis of a propargyl ester of a carboxylic acid is the mutual esterification of an acid and an alcohol. By this method, an equimolar mixture of a monobasic saturated carboxylic acid and propargyl alcohol was heated in the presence of sulfuric acid. Monobasic normal carboxylic acids were used in the reaction: butyric acid, valeric, caproic, enanthic, caprylic acids. The resulting products were purified by distillation at 8-9 mm Hg. using a vacuum pump and obtained the corresponding propargyl esters in high yield (1-5). The resulting ether was washed in water, dried and distilled in vacuo. The reaction equation is the following:



$$R-COOH + HO-CH_2-C\equiv CH \xrightarrow[C_6H_6]{H_2SO_4} R-\underset{\underset{O}{\|}}{C}-O-CH_2-C\equiv CH$$

$$R= C_3H_7, C_4H_9, C_5H_{11}, C_6H_{13}, C_7H_{15}$$

The structure of the resulting esters was studied by IR and NMR spectroscopy, and the structures of the obtained substances were confirmed (Fig. 1).

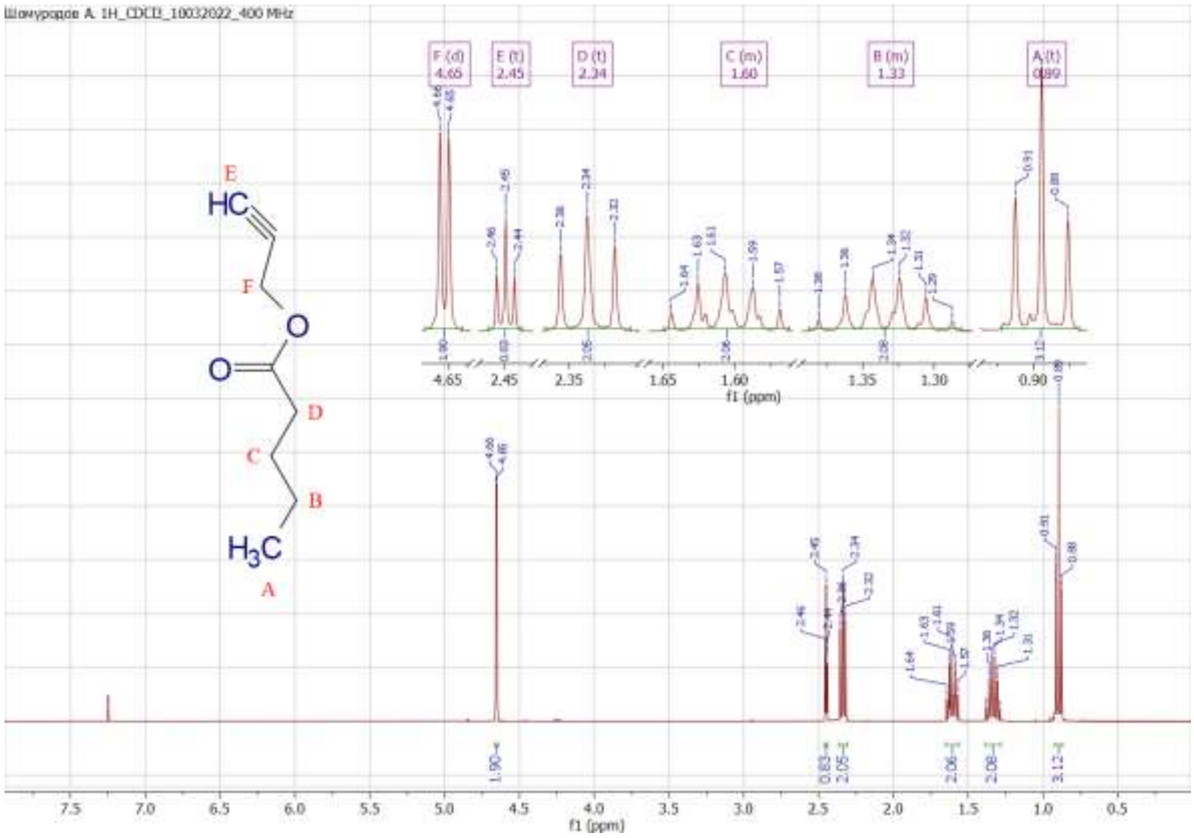
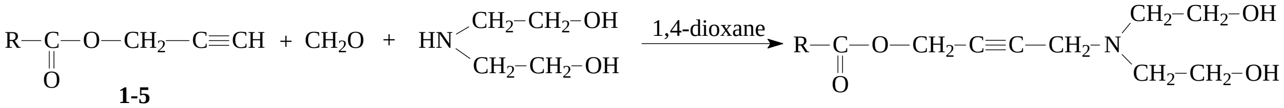


Figure 1: ^{1}H NMR spectrum of propargyl valerate (2).

At the second stage, the reactions of aminomethylation of propargyl esters of diethanolamine and the paraform by the Mannich reaction were carried out. The aminomethylation reaction was carried out in the medium of a mixture of the corresponding esters:diethanolamine:paraform in the ratio 1:1:1.5 mol, in a solvent medium, by heating at a temperature of 80-110°C, for 6-8 hours. Toluene, acetonitrile, and 1,4-dioxanes were used as solvents in the reaction. Copper salts were used as a catalyst: copper (I) chloride, copper (II) chloride, and copper (II) acetate. The reaction was carried out at the boiling temperature of solutions of toluene, acetonitrile, 1,4-dioxane at different ratios of catalysts and different time durations. The highest yield was achieved when copper(II) acetate was used as a catalyst by boiling in 1,4-dioxane for 6 hours. As a result, the corresponding aminomethylated derivatives (6-10) were isolated.



$$R-\underset{\underset{O}{\|}}{C}-O-CH_2-C\equiv CH + CH_2O + HN\begin{matrix}CH_2-CH_2-OH\\ CH_2-CH_2-OH\end{matrix} \xrightarrow{\text{1,4-dioxane}} R-\underset{\underset{O}{\|}}{C}-O-CH_2-C\equiv C-CH_2-N\begin{matrix}CH_2-CH_2-OH\\ CH_2-CH_2-OH\end{matrix}$$

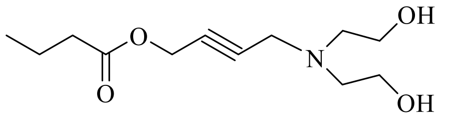
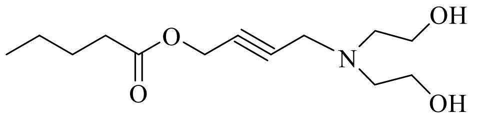
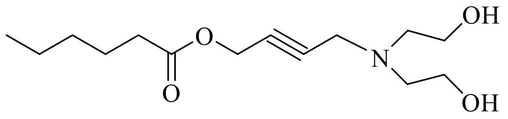
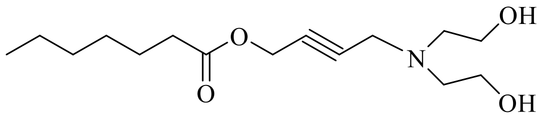
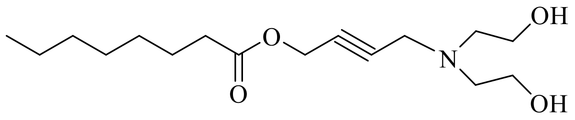
1-5

R= **1,6** C_3H_7, **2,7** C_4H_9, **3,8** C_5H_{11}, **4,9** C_6H_{13}, **5,10** C_7H_{15}

After the end of the experiments, the reaction mixture was cooled, the main product was isolated with petroleum ether. The substances were dried and purified by vacuum distillation. Due to the fact that the substances were mainly in a liquid state of aggregation, it was somewhat difficult to isolate them in their pure form. Some physical constants of the obtained substances were studied (Table 1).

Table 1: Yield of synthesized substances, some of their physical constants.

№	Structure formula	R_f value (hexane:ethyl acetate 7:1)	Boiling point. 8 mm. mercury wall, ^{0}C	Reaction yield, %

6		0.66	172-174	84
7		0.68	176-178	82
8		0.69	184-185	80
9		0.72	196-198	81
10		0.74	211-213	79

The structure of the obtained substances was studied by IR and ^{1}H NMR spectroscopy. Thus, when analyzing the ^{1}H NMR spectrum of 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) (Fig. 2), the protons of the butyric acid residue exhibit signals in the form of a two-proton triplet in the region of 0.88 ppm (3H, t, J=7.34, -C$\underline{H_3}$-), 1.32 (2H, m, CH_3-$\underline{CH_2}$-), 1.59 (2H, t, J=5.88, -CH_2-$\underline{CH_2}$-), methylene groups adjacent to the triple bond in in the middle of the molecule show a signal in the region of 2.70 ppm. (2H, t, J=5.26, -$\underline{CH_2}$-N<), 4.65 (2H, t, J=4.10, -O-$\underline{CH_2}$-) as a two-proton triplet, methylene group protons in the diethanolamine residue in the region of 2.31 ppm. (2H, t, J=7.28, -$\underline{CH_2}$-CH_2-OH), 2.44 ppm (2H, т, J=7.14, $\underline{\text{-}CH_2}$-CH_2-OH), 3.61 ppm. (4H, t, J=5.20, -CH_2-$\underline{CH_2}$-OH) show signals in the form of a two-proton triplet and a four-proton triplet, and the protons of the hydroxyl group in the remainder diethanolamine in the region of 2.98 ppm. (2H, s, $\underline{\text{-}OH}$), show a signal in the form of a two-proton singlet.

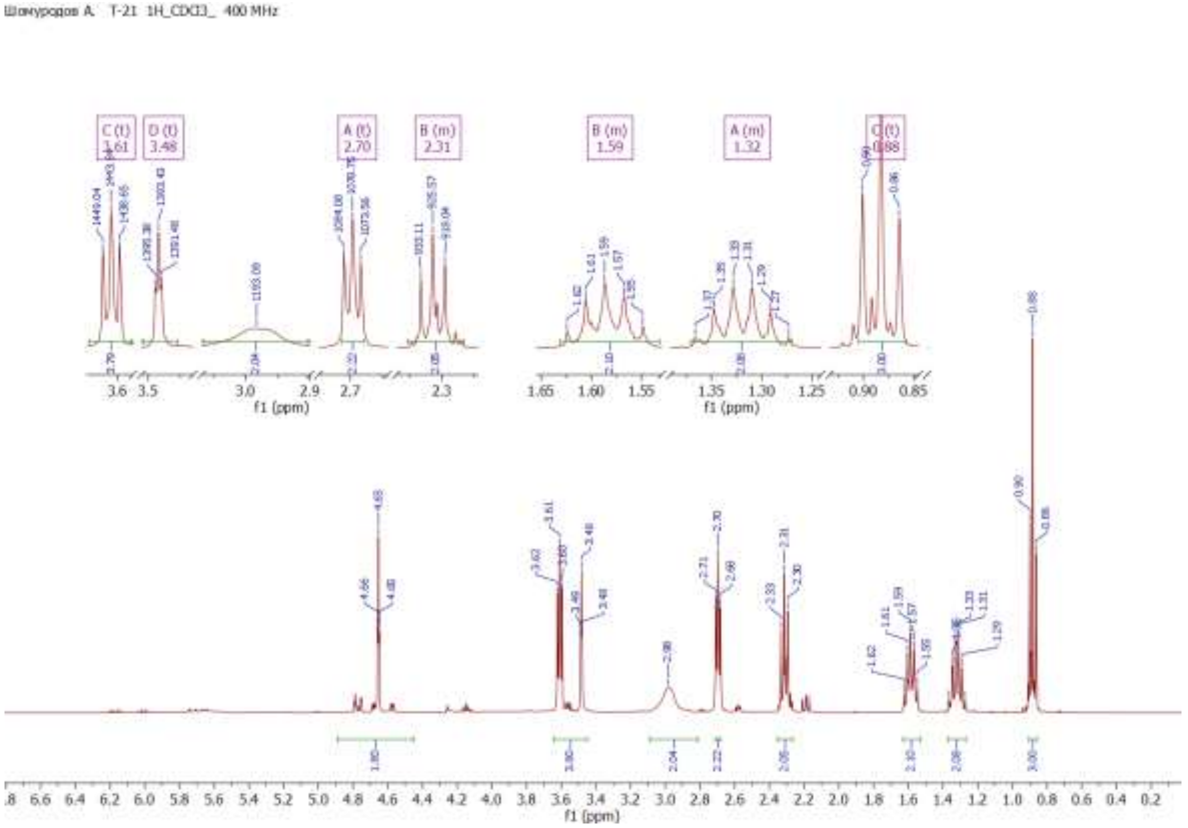


Figure 2: ^{1}H NMR spectrum of 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6).

Analysis of the IR spectra of the substance (6) showed (Fig. 3) that the vibration frequencies of the methyl group ($-CH_3$) of the molecule show signals in the region of 2958 cm^{-1}, the methylene group ($-CH_2-$) in the region of 2932, 2873 cm^{-1}, vibrations carbonyl group (>C=O) in the region of 1742 cm^{-1}, vibrations of the (C-N) group in the region of 1244 cm^{-1}, vibrations of the carbon-oxygen-carbon (C-O-C) bond in the region of 1166 cm^{-1}, vibrations of the bond carbon-hydroxyl group (-C-OH) in the region of 1109 cm^{-1} (Fig. 3). The structures of other new substances (7-10) were also analyzed by IR and NMR spectroscopy.

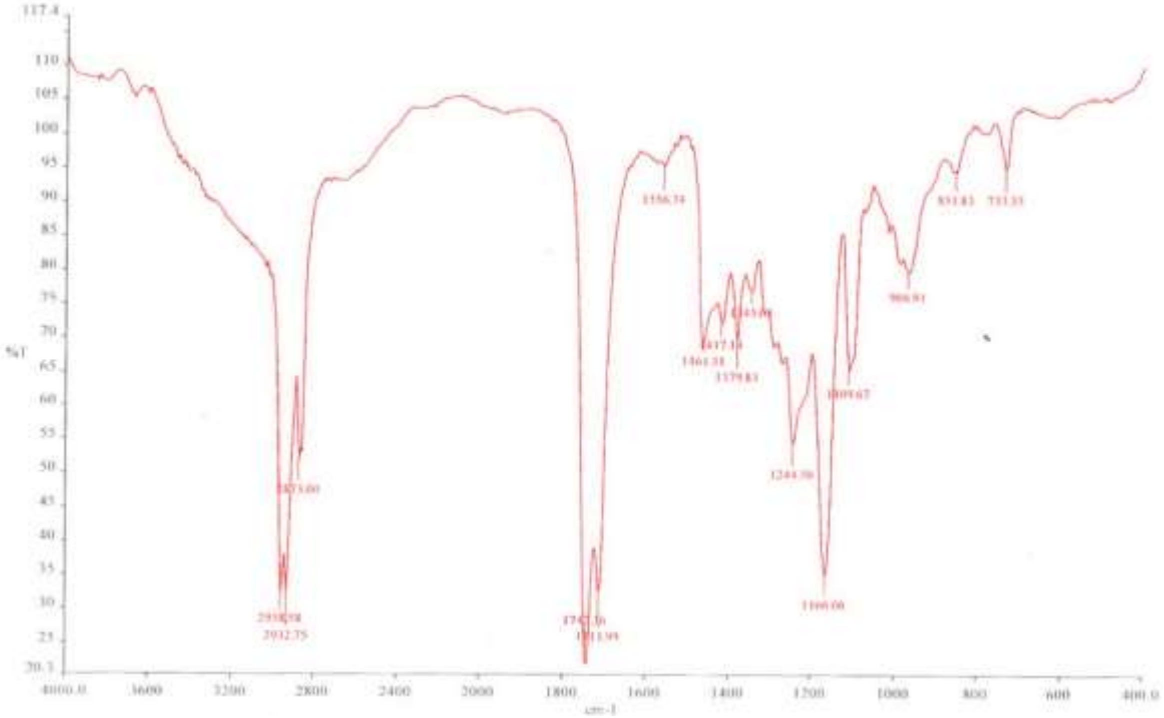


Figure 3: IR spectrum of 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6)

According to the analysis of the results of the obtained spectra, it was proved that the structure of the synthesized substances is fully consistent with the corresponding structures.

V. CONCLUSION AND FUTURE WORK

A number of propargyl esters were synthesized by esterification of monocarboxylic acids and propargyl alcohol. The obtained esters were aminomethylated with paraform and diethanolamines. Toluene and 1,4-dioxanes of acetonitrile were used as solvents. And copper (II) salts were used as catalysts. The highest reaction yield was achieved when a copper(II) acetate catalyst was used and heated in a 1,4-dioxane solution by heating for 6 hours. The result of the synthesis is obtaining derivatives of 4-(bis(2-hydroxyethyl)amino)but-2-yn-1-yl, which retained the residue of monocarboxylic acids. The proposed methods of synthesis, which undoubtedly have a number of advantages, will be able to find their application in the targeted synthesis of such substances in the future.

To identify the growth-promoting activity of compounds 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6), tests were carried out in the laboratory of the Institute of Plant Chemistry of the Academy of Sciences of the Republic of Uzbekistan in laboratory conditions, vegetable seeds served as biotests.

The experiments used cucumbers of the Orzu variety and tomatoes of the Yusupovsky variety. The preparations were dissolved in dioxane, the presowing method of seed sowing was used for 18-20 hours. The concentrations used were 0,1; 0,01; 0,001; 0,0001 and 0,00001 %. The repetition of the experiments is 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.

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 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 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 after 3, 5, 7 and 10 days after inoculation (tables 2-3).

Table 2. The effect of the preparation 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) on the germination of seeds and the growth of seedlings of cucumbers varieties "Orzu"

Experiences A drug	Concentration, %	Germination, %	Cucumbers	
			Root growth	Stem growth
Control - water	without	100,0	100,0	100,0
4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6)	0,1	100,0	107,7	108,3
	0,01	100,0	110,2	112,5
	0,001	100,0	114,6	111,9
	0,0001	100,0	120,2	115,2
	0,00001	100,0	128,1	117,5
“Rostlin” (famous)	0,75-1,0	100,0	102,7	101,8

Table 3. The effect of the drug 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) on seed germination and growth of seedlings of tomato varieties " Yusupovsky".

Experiences A drug	Concentration, %	Germination, %	Tomatoes	
			Root growth	Stem growth
Control - water	without	50,0	100,0	100,0
4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6)	0,1	50,0	107,8	117,2
	0,01	57,7	116,5	120,4
	0,001	59,2	122,9	124,5
	0,0001	47,3	116,7	108,1
	0,00001	54,8	119,5	107,6
“Rostlin” (famous)	0,75-1,0	52,6	101,3	100,5

Comparative tests also show that the test drug 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) showed higher growth-promoting activity at a lower concentration (from 7,5 to 75000 times) than currently used in many agriculture industries of Uzbekistan, the drug "Rostlin".

The drug 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) on tomatoes, similar to previous cultures, showed a very high biological activity of 119,5 % at a concentration of 0,00001 % (even 75,000 times diluted).

The drug 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) on cucumber culture also showed biological activity at a concentration of 0,00001 % (i.e. 75,000 times diluted), promoted root growth by 128,1 %, somewhat lower - stem growth 117,5 % higher than control and the well-known drug "Rostlin" (concentration 0,75-1,0 %).

Thus, the low-toxic (LD_{50} = 2250 mg / kg) preparation 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) showed high stimulating properties on the seeds of tomato and cucumbers at 0,00001 % concentration.

The results of determining the growth-promoting activity of compound 4-(Bis(2-hydroxyethyl)amino)but-2-yn-1-yl butyrate (6) are presented in tables 2-3.

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