Synthesis andpropertiesof4-piperidilo-(butin-2) ol derivatives

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ABSTRACT

In articledescribed syntheses, characteristic and biological activity of 4-piperidilo-(butin-2) ol. Determined physico-chemical constants and proved chemical structures of synthesized compounds by IR- and N-PMR methods. Organized quantum-chemical calculations and shown reactionary centers of the molecules.

KEYWORDS: *selective method, physical and chemical constants, 3D structure, distribution charges, electron density, center of reactivity, IR and PMR spectroscopy.*

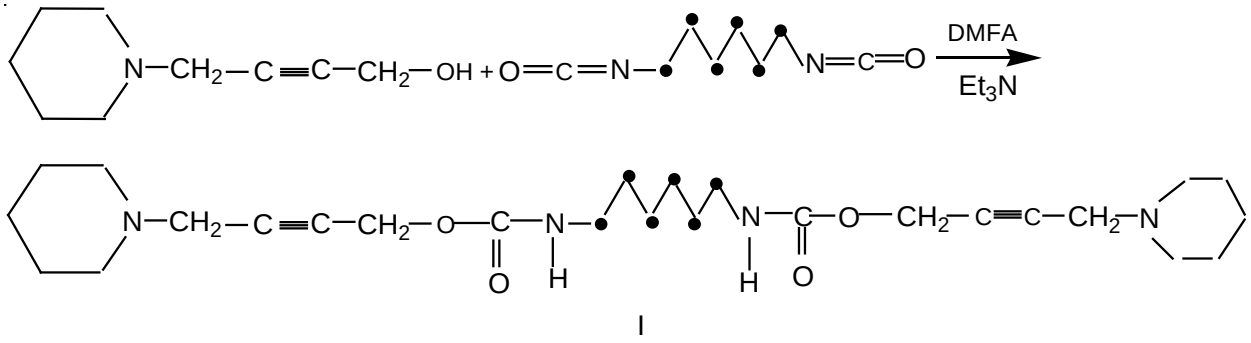
INTRODUCTION

In world scientific literature, monographs and patent literature [1-10] show that the synthesis technology of selective, high-performance, low-cost, low-toxic, the promising piperidil- (butyn-2) -1-ol is the subject of active research laboratories and companies in several foreign countries.

The objective of this work is to reduce the toxicity of labor input, environmentally friendly produce bio-growth regulators, low cost process for the preparation, accessibility, even without electricity costs and high yields, providing growth of regulated properties for technical and vegetable crops (cotton, tomatoes, cucumbers, corn, soybean and etc.).

EXPERIMENTAL

The task is achieved the purpose of non-waste technology environmentally friendly synthesis of previously undescribed organic growth regulators N_1N^I-hexamethylenebis [(4-piperidilo- (2-butyne) carbamate, according to the following reaction scheme:



I

The composition and the resulting compound confirmed identity physicochemical characteristics, by elemental analysis,N-PMR and IR spectroscopy are listed in Table 1.

Table 1. Physico-chemical characteristics of the N_IN^I- hexamethylenebis [(4-piperidilo- (2-butyne) carbamate]

output %	T., C^0	R_t	Gross formula	Elemental analysis,%						mm
				Calcul.			found.			
				C	H	N	C	H	N	
96,8	203-204	0,81	$C_{26}H_{42}N_4O_4$	65,82	8,86	11,81	65,66	8,69	11,64	474

Preparation I was prepared by the following steps: mixing, washing, drying, cleaning, which takes time of about 2.5-3.0 hours.

The IR spectrum of the compound has strong absorption bands, given in Table 2 and Table 3.

Table 2. IR Spectral data ofN_IN^I -hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] R-CH_2-C≡C-CH_2OCONH$(CH_2)_6$NHCOO-CH_2-C≡C-CH_2R

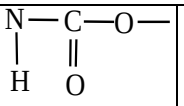
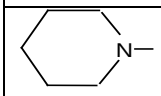
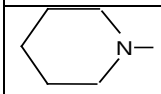
where:R	IR spectrum, ν, sm^{-1}						
		-NH-CH_2-	-C≡C-		-$(CH_2)_6$	-NH-	-O-CH_2
	1670	1418	2216	1284	752-718	3290	1016

Table 3. H-NMR Spectral data of N_IN^I -hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] R-CH_2-C≡C-CH_2OCONH$(CH_2)_6$NHCOO-CH_2-C≡C-CH_2R

where:R	H-NMR spectrum, δ,м.д		
	-N-CH_2	NH	piperidyl
	3,22	4,50	1,23-2,43

RESULTS AND DISCUSSIONS

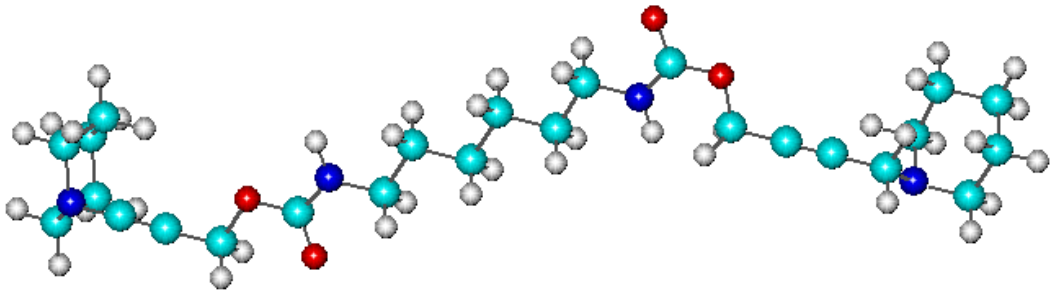
The result of applying the methods of quantum chemistry is the information density of electron states in the distribution of the electron density, the potential surfaces and reaction calculations of various spectroscopic values. Currently, quantum chemistry methods are cheaper, accessible and universal method of studying the electronic structure of molecules. However, it should be understood that, nevertheless, it is impossible to completely abandon the expensive experimental methods of research material.

Investigated 3D structure and charge distribution of the electron density of 4 - [(piperidyl-butyn-2) 1-ol] and N_IN^I -hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] widespread program Hyper Chem semi-empirical quantum-chemical method RMZ.

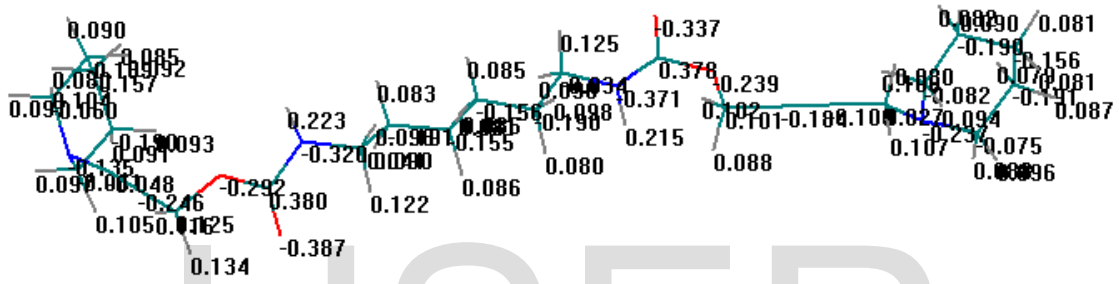
As an example, we give the results of studying the geometry and electronic structure of molecules of 4 - [(piperidyl-butyn-2) 1-ol] and N_IN^I -hexamethylenebis [(4-piperidilo- (2-butyne) carbamate].

Research and charge distribution of the electron density on the atoms in the molecule N_IN^I -hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] (Figure 1) showed that the molecule has a symmetry, the highest number of negative charge and the electron density is concentrated oxygen

atoms. Moreover, both the oxygen atom have the same activity in the reaction to produce the bis [(4-piperidilo- (2-butyne) carbamate].



a



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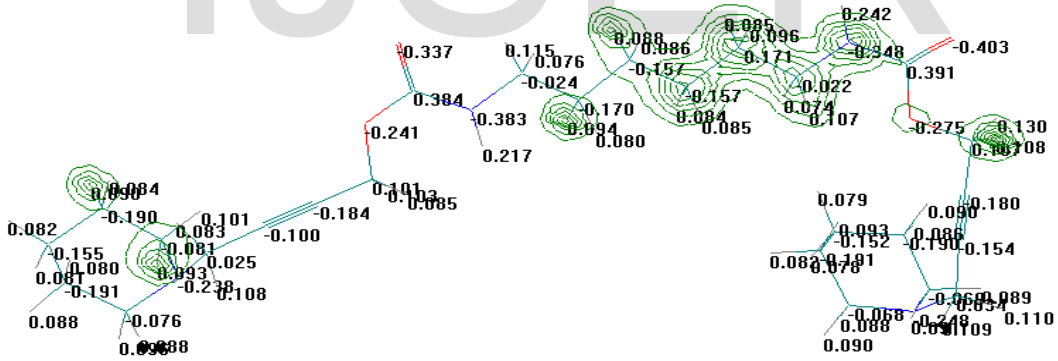

c

Fig.1 (a), 3D structure of the distribution of charges in a molecule (b) and the electron density distribution (c) for the atoms in the molecule bis [(4-piperidilo- (2-butyne) carbamate]

Distribution charges in the molecule and the electron density distribution over the atoms in the molecule bis [(4-piperidilo- (2-butyne) carbamate], we also carried out quantum chemical calculations (total energy, energy of formation, heat of formation, the energy of the electron energy of the nucleus, the dipole moment, charge oxygen atom), the results of which are presented in Table 4.

Thus, based on the results of studies of the structure, the charge distribution of the electron density and energy characteristics of selected molecules semiempirical quantum-chemical method, the reaction centres identified molecules used, which in turn are used for the specific coordination of the molecules of the reaction components.

Table 4. Quantum chemical calculations of the studied compounds

Nature of compound	The total energy, kcal/mol	Formation energy, kcal/mol	The heat of formation, kcal/mol	The dipole moment (D)	The charge of the oxygen atom
The initial substance					
[Piperidyl-4 (2-butyne) 1-ol]	-43482,09	-2507,4919	-15,3930	1,349	-0,318
hexamethylenediisocyanate	-51742,65	-2385,28	-47,8256	0,000109	0,211
The synthesized compound					
N_IN^I-hexamethylenebis [(4-piperidilo- (2-butyne) carbamate]	-135141,67	-7146,8486	-100,282	2,106	-0,292

To identify organic growth stimulant active derivatives, NINI-hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] the drug has been tested in the laboratory of phyto-toxicology atInstitute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Biotest served vegetable seeds and cotton primary cracking was conducted by Y.V.Rakitin method. This method allows fairly quickly determine the degree of physiological activity of chemical compounds, which is detected by stimulation or inhibition of the germination of plant seeds, as well as to change the length of the roots and the length of the stems. The drugs tested by steeping the seeds in solutions of various concentrations, followed by germination in Petri control seeds were soaked in distilled water.

It is found that when the cotton seed clasp NINI-hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] at 0.1, 0.01, 0.001%, the drug increases germination ahead control study medication for bio growth stimulating activity of cotton. This showed that the drug promotes development of seedling root system at a concentration of 0.001%, the possibility of accelerated seed on the fifth day to several times higher than the control (Table 5).

Table 5. The results of bioassay of cotton on the concentration of the drug

Drug name	Concentration,%	Seed germination, after 5 days,%	The effect of drug on the growth of plantfor 10 days. %	
			Root	stem
N_IN^I- hexamethylenebis [(4-piperidilo- (2-butyne) carbamate]	0,1	80	117,8	106,6
	0,01	80	134,9	117,4
	0,001	80	129,4	109,7
Control-H_2O	-	80	100	100
Roslyn	0,75	80	102,6	96,3

The drug I N_IN^I-hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] at a concentration of 0.001% (ie at a dilution of 7500 times) bio stimulated germination of cotton seedlings to 161% higher than the control, as well as root growth to 129.4% and the growth of the stem at 109.7% higher than the control (table 5).

Preparation of bis [(4-piperidilo- (2-butyne) carbamate] at a concentration of 0.001% (ie at a dilution of 7500 times the bio stimulated germination of tomato seedlings 140% above control, as well as root growth and 133.4% the growth of the stem at 121.2% above control) Table 6.

Table 6. The results of bioassay tomato on the concentration of the drug

The name of drug	Concentration,%	Seed germination, after 5 days,%	The effect of drug on the growth of plantfor 10 days. %	
			Root	Stem
N_IN^I- hexamethylenebis [(4-piperidilo- (2-butyne) carbamate]	0,1	43	118,8	109,6
	0,01	49	126,7	117,8
	0,001	52	133,4	121,2
Control-H_2O	-	30	100	100
Roslyn	0,75	40	100	100

Experiments on "Uzbekistan-740" cucumber varieties have shown that the drug is actively influenced the growth of the root system of seedlings (Tab. 6)

Table 6. **The results of bioassay of cucumber on the concentration of the drug**

The name of drug	Concentration,%	Seed germination, after 5 days,%	The effect of drug on the growth of plantfor 10 days. %	
			Root	Stem
N_IN^I- hexamethylenebis [(4-piperidilo- (2-butyne) carbamate]	0,1	100	118,4	111,1
	0,01	100	126,7	117,8
	0,001	100	139,6	122,5
Control-H_2O	-	100	100	100
Roslyn	0,75	100	101,3	98,7

CONCLUSION

Thus, a new drug N_IN^I- hexamethylenebis [(4-piperidilo- (2-butyne) carbamate] in a concentration of 0.001% is the most highly biorosto stimulant for vegetables and cotton in the laboratory and is recommended for further more in-depth study in the field Navoi and Kashkadarya regions.

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