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SYNTHESIS OF SOME N-OXIDES OF PYRIDYLACETHYLENE AMINES BASED ON 2-METHYL-5-ETHYNYLPYRIDINE

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Abstract: The article presents the results of studies of the reaction of 2-methyl-5-ethynylpyridine N-oxide with certain aliphatic and aromatic amines to produce pyridylacetylene amine N-oxides. 2-Methyl-5-ethylpyridine was converted by dehydrogenation to 2-methyl-5-vinylpyridine, and 2-methyl-5-ethynylpyridine was obtained by its bromination and dehydrobromination. Synthesized 2-methyl-5-ethynylpyridine N-oxide by oxidation of 2-methyl-5-ethynylpyridine with perhydrol in acetic anhydride. The synthesis of N-oxides of pyridylacetylene amines by the Mannich reaction was carried out. The influence of solvents on the yield of the final product: methanol, ethanol and n-dioxane, a catalyst, and also the duration of the reaction was studied. The structure and composition of the synthesized N-oxides of PAA were established using IR and PMR spectroscopy.

Keywords: 2-methyl-5-ethynylpyridine, methyl amine, ethylamine, piperidine, morfolino, catalyst, solvents, N-oxides of pyridylacethylene amines.

INTRODUCTION

Currently, herbicides of continuous and selective action, insecticides, fungicides and bactericides have been obtained from pyridine and its homologues and are used in agriculture. [1].

Preparations such as nitropyrin and 2-chloro-6-(trichloromethyl)pyridine are nitrogen fertilizer stabilizers in the soil. Based on these compounds, widely used drugs have also been developed - ftivazide, saluzide and metazid, used in the treatment of tuberculosis diseases. Pyridine derivatives, to a certain extent, have found application as vitamins, monomers and polymers, oil additives, metal corrosion inhibitors, suspension stabilizers, extractants, dyes and analytical reagents. [2].

The synthesis and various reactions of many pyridine bases (PB) have been studied in some detail in a number of works [3–9]. However, some representatives of pyridine derivatives have so far remained insufficiently studied or not considered at all.

It is well known that heteroaromatic N-oxides are widely used as the most important intermediates in the synthesis of substituted pyridine derivatives and other nitrogen-containing heterocycles. This is facilitated by the higher activity of N-oxides compared to non-oxidized bases in both electrophilic and nucleophilic substitution reactions [10]. Synthesis of N-oxides of nitrogen-containing heterocycles was carried out by various methods. For example, to obtain N-oxide of 3,4-pyridinedicarboxylic acid diethyl ester, a complex of urea with H_2O_2 and acetic anhydride was used [11].

The scope of N-oxides is wide. For example, solid complexes of N-oxides of 2,6-lutidines, 2-picoline, and pyridine with succinic acid with a composition of 2:1 were studied by IR spectroscopy. According to their electrical properties, these complexes belong to the class of protonic semiconductors [12]. There is information about the possibility of formation of unstable molecular complexes of pyridine N-oxides with alkali and alkaline earth metal cations [13, 14]. These complexes are formed in an aqueous solution with a significant excess of complexing ions. When interacting with SiO_2 in an aqueous medium, pyridine N-oxides give unstable complexes of pentacoordinated silicon with an O–Si bond [15]. The appearance of such complexes explains the interesting fact that N-oxides significantly increase the solubility of SiO_2 in water.

Solid complexes of ZnCI2 with N-oxides of pyridine and methylpyridines with a composition of 1:2 are characterized by a difference in crystal structures depending on temperature. The electron-donating effect of the methyl group significantly affects the enthalpy of the O–Zn bond, significantly increasing it compared to the unsubstituted compound [16]. In many cases, N-oxides exhibit a much greater catalytic effect than the corresponding non-oxidized bases. A large number of examples of the catalytic activity of N-oxides are given in [17, 18].

There are very few data in the literature on the study of acetylene-containing N-oxides of pyridine bases. This study is aimed at the synthesis of some N-oxides of acetylene-containing pyridine bases.

RESEARCH METHODS

The objects of study are 2-methyl-5-ethynylpyridine N-oxide, some aliphatic and heteroaromatic amines, paraformaldehyde, perhydrol, acetic anhydride, AHCO(AI2O3(70.0%)+ Fe2O3(25.0%)+Cr2O3(5, 0%)) catalyst. First, by dehydrogenation of 2-methyl-5-ethylpyridine in the presence of an AHLC catalyst at a temperature of 380°C, 2-methyl-5-vinylpyridine was obtained, which, by subsequent bromination and dehydrobromination, was converted into 2-methyl-5-ethynylpyridine, which was oxidized with perhydrol in acetic anhydride medium and converted it to N-oxide-2-methyl-5-ethynylpyridine.

Aminomethylation was carried out according to the Mannich reaction of N-oxide-2-methyl-5-ethynylpyridine with dimethyl-, diethylamines, piperidine,

morpholine, benzoxazolone and nitrobenzoxazolone. To carry out the reactions, the calculated amounts of N-oxide-2-methyl-5-ethynylpyridine, dry powdered PFA, the corresponding amine, n-dioxane, and anhydrous copper acetate or copper chloride catalyst are placed in a round-bottomed flask equipped with a reflux condenser. The mixture was heated on a water bath (90-95°C) for 4-6 hours with stirring. After cooling, the mixture was diluted with an appropriate volume of water and 10% potash solution, and the organic part was separated from the aqueous. The latter was additionally extracted with chloroform. The organic part and chloroform extracts were combined and dried over potash, the solvent (n-dioxane) was distilled off on a water bath, and the residue was subjected to vacuum distillation.

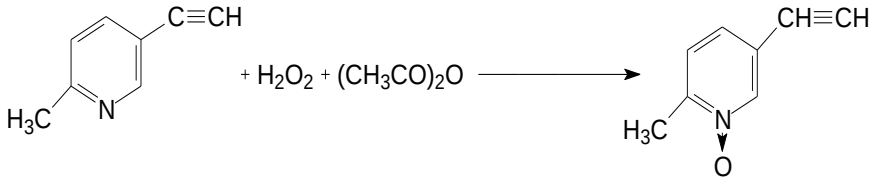
RESULTS

The further transformations of 2-methyl-5-ethylpyridine (2-M-5-EP), the main product of the interaction of crotonaldehyde (CA) with ammonia, were of great interest [19, 20]. Thus, the possibility of using the developed catalysts in the process of obtaining the valuable monomer 2-methyl-5-vinylpyridine(2-M-5-VP) by heterogeneous dehydrogenation of 2-M-5-EP has been elucidated. The process was carried out at temperatures of 360-390 °C in the presence of a catalyst. In this case, the best results were obtained with the use of a catalyst for the reaction of AHCO(AI_2O_3(70.0%)+Fe_2O_3(25.0%)+Cr_2O_3(5.0%)) catalyst. In its presence, the yield of 2-M-5-VP reaches 41.05%.

Then, based on 2-M-VP, 2-methyl-5-ethynylpyridine (2-M-5-ENP) was synthesized, which, in turn, is an important initial reagent in the preparation of numerous new pyridine derivatives [3, 4]. Its output is in the range of 60-65%.

Next, we investigated the possibility of synthesizing some pyridylacetylenic amines (PAA) based on 2-M-5-ENP by a known method under the conditions of the Mannich reaction. Great interest in such PAAs is primarily due to their known biological activity [21].

As already noted, PB N-oxides are of great practical interest [13–18]. Based on this, we studied the possibility of synthesizing such compounds based on 2-M-5-ENP. For this, the necessary peracetic acid was obtained directly during the interaction of perhydrol with acetic anhydride. The reaction proceeds according to the scheme:



The output of the resulting N-oxide 63.1%.

Next, we studied the interaction of the resulting 2-M-5-ENP N-oxide with dimethyl- and diethylamines, as well as piperidine, morphaline, benzaxozolone, and nitrobenzaxolone. These processes can also proceed both in the absence and in the presence of catalysts, which indicates the high reactivity of 2-M-5-ENP N-oxide.

Some physicochemical data of the synthesized compounds were determined (Table 1).

The IR spectra of the formed compounds were recorded on a UR-22 spectrophotometer in the region of 400-4000 cm^{-1}. In this case, the spectra of liquid solids were taken in the form of tablets with potassium bromide.

The PMR spectra of the samples were taken on a C-60HI instrument in a solution of deuterochloroform.

Table 1

Some physicochemical data of the synthesized compounds

№	Compound	Molecular mass, %	$T_{boiling}$, °C	N, %	
				Found	Computed
1	2-methyl-5-(3'dimethylaminopropin-1'-yl-1'-) pyridine N-oxide (a)	190	120,3-121,0	14,58	14,73
2	2-methyl-5-(3'diethylaminopropyn-1'-yl-1'-) pyridine N-oxide (b)	218	129,1-130,0	12,69	12,84
3	2-methyl-5-(3'-piperidino-propyn-1'-yl-1')pyridine N-oxide (c)	230	197,1-197,6	12,03	12,17
4	2-methyl-5-(3′-morpholino-propyn-1′-yl-1′)pyridine N-oxide (d)	232	201,2-202,0	11,96	12,06
5	2-methyl-5-(3′-N-benzoxazolonyl-propyn-1′-yl-1′)pyridine N-oxide (e)	280	248,5-249,0	9,87	10,00
6	N-Oxide 2-methyl-5-(3′-N-(6-nitrobenzoxozo lonyl-propyn-1′-yl-1′)pyridine (l)	309	235,1	13,14	13,59

In the IR spectra of aliphatic PAA N-oxides, in particular, 2-methyl-5-(3'-dimethylaminopropin-1'-yl-1') pyridine N-oxide and 2-methyl-5-(3'-diethylaminopropin-1'-yl-1')-pyridine, intense bands appear in the region of 1630 cm^{-1}, characteristic of the HC=CH group; peaks at 1270 cm^{-1} belong to the tertiary amino group; 1468 cm^{-1} - to $–CH_2$ groups. The bands characteristic of the C≡C bond are weakly manifested at 2220–2100 cm^{-1}, and in the region of 850 cm^{-1} there are bands assigned to the N → 0 group 1480–1440 cm^{-1}, which are characteristic of the pyridine nucleus. In addition, the spectrum of the last N-oxide also has a broad band at 3400-3100 cm^{-1}.

In the IR spectra of N-oxides of heterocyclic PAA, absorption bands are observed in the region 1090-1080 cm^{-1} related to the piperidine group, and the bands at 1120-1080 cm^{-1} correspond to the morpholino group. There are also bands at 1270, 1580-1530, 2200-2100 and 2900-2790 cm^{-1}, which are characteristic of other fragments of these compounds.

In the IR spectra of synthesized heteroaromatic PAAs, in particular, 2-methyl-5-(3'-N-benzoxazolonylpropin-1'-yl-1') pyridine and 2-methyl-5-(3'-N–(6"-nitrobenzoxazolonyl)-propyn-1'-yl-1') pyridine, intense absorption bands are observed in the region of 1630 cm^{-1} - for the HC=CH bond, the bands at 1270 cm^{-1} belong to the tertiary amino group; at 1468 cm^{-1} - to CH_2 groups; bands at 1800-1750 cm^{-1} - to the C=0 group. In the spectrum of a nitro-containing compound, there are also characteristic absorptions in the region of 1400 cm^{-1}, attributed to the nitro group. The bands characteristic of the C≡C bond appear weakly in the region of 2220-2100 cm^{-1}.

In the PMR spectra of N-oxides of aliphatic PAAs, there are intense signals in the region of 6.5–8.5 ppm, which are characteristic of protons in the α, β, and γ positions of the pyridine ring. Signals at 2.7-2.8 ppm; 3.2-3.3 ppm and 2.2-2.6 ppm correspond to the protons of the methyl group in the α, β, γ positions.

In the PMR spectrum of 2-methyl-5-(3'-piperidinopropyn-1'-yl-1-1-1) pyridine N-oxide, there are signals in the region of 5.6-7.4 ppm, which are characteristic of protons in α, β, γ - positions of the pyridine nucleus, signals at 2.2-2.5 ppm belong to the protons of the methylene group, as well as to the protons of the piperidine ring, and at 1.1-1.4 ppm. correspond to the protons of the methyl group.

In the PMR spectrum of 2-methyl-5-(3'-morpholinopropyn-1'-yl-1') pyridine N-oxide, intense signals are observed in the region of 6.6–8.5 ppm, which are characteristic of protons in α, β, γ -positions of the pyridine ring. Signals at 3.3-3.8 ppm correspond to the protons of the methylene group, as well as to the protons of morpholine and at 2.2-2.8 ppm. - characteristic of the protons of the methyl group.

First, in order to elucidate the influence of the nature of solvents under comparable conditions on the ongoing reactions, the aminomethylation of 2-M-5-ENP N-oxide was carried out in the absence of a catalyst in methanol, ethanol, and p-dioxane at their boiling temperature. As a result, the corresponding target compounds were synthesized in all cases (Table 2).

Table 2

Influence of the nature of amines and solvents on the formation of PAA N-oxides in the absence of catalysts (reaction time 8 hours, ratio of N-oxide, 2-M-5-ENP, PFA and amine 1.0:1.5:1.5)

Compound	Compound yield in solvent medium, %		
	methanol	ethanol	p-dioxane
2-methyl-5-(3'dimethylaminopropyn-1'-yl-1')pyridine(a) N-oxide (a)	67,6	63,8	62,1
N-oxide of 2-methyl-5-(3'-diethylaminopropyn-1'-yl-1'-)pyridine(b)	67,3	63,1	61,8

2-methyl-5-(3'-piperidinopropyn-1'-yl-1')pyridine N-oxide (c)	44,3	40,2	37,5
N-oxide - 2-methyl-5-(3'-morpholinopropyn-1'-yl-1')pyridine (d)	41,7	37,3	33,2
2-methyl-5-(3,-N-benzox-zolonyl propyn-1'-yl-1')pyridine N-oxide (e)	51,3	49,3	47,2
2-Methyl-5-(3'-N-(6-nitrobenzoxazolonylpropin-I'-yl-1') pyridine N-oxide (l)	53,5	51,7	49,4

It follows from the data obtained (Table 2) that the dependence of the formation of products on the nature of the amines taken and the solvents used is almost the same.

Next, we studied the effect of the duration of work on the yield of target products (Table 3)

Table 3

Yield of PAA N-oxides depending on the reaction time (at the boiling point of p-dioxane)

Reaction time, hours	Yields of compounds, %					
	a	b	c	d	e	i
4	40,9	41,2	28,3	23,1	35,1	36,3
5	47,3	47,8	29,9	25,8	37,2	38,9
6	55,3	56,3	34,1	29,8	39,5	41,2
7	57,9	59,1	35,3	32,3	41,9	44,0
8	62,1	61,8	37,5	33,2	43,7	46,9
9	62,2	61,7	37.8	33.5	46.9	48,7

From the results presented in Table 3, it follows that the yields of all PAA N-oxides reach their maximum, mainly within 6-7 hours, and then remain practically constant. The latter is obviously due to the influence of both the resulting products and a decrease in the concentration of the initial reagents in the reaction medium.

The processes of synthesis of PAA N-oxides are significantly intensified in the presence of the Cu_2CI_2 catalyst (2.5% of the total mass of the reaction mixture) (Table 4). This is due, as already mentioned above, to the formation of active transition complexes (π-complexes) between Cu^{2+} and C≡C ions by the bond of the initial pyridylacetylene compound. For example, in the case of aminomethylation of 2-M-5-ENP N-oxide with dimethylamine in n-dioxane at the boiling point of the latter, 83.3% of substance (a) is formed, i.e. 21.2% more than when the reaction is carried out in the absence of a catalyst. A similar comparison of the data on the conducted Mannich reactions with other amines showed that the yields of the

synthesized substances (b), (c), (d), (e) and (g) in the presence of a catalyst increase by 20.6, respectively; 45.9; 43.4; 17.5 and 18.9%. In general, in these processes, the yields of the products obtained in the presence of Cu_2CI_2, depending on the nature of the amines used, range from 64.7 to 83.3%.

Table 4

Dependence of yields of PAA N-oxides in the absence and presence of a catalyst (Cu_2CI_2). (The duration of the reaction is 8 hours, at the boiling point of p-dioxane; the ratio of N-oxide 2-M-5-ENP, PFA and amine 1.0:1.5:1.5)

Compound	Yields of compounds, %	
	In the absence of a catalyst	In the presence of a catalyst
2-methyl-5-(3'dimethylaminopropyn-1'-yl-1')pyridine(a) N-oxide (a)	62,1	83,3
N-oxide of 2-methyl-5-(3'-diethylaminopropyn-1'-yl-1'-)pyridine(b)	61,8	82,3
2-methyl-5-(3'-piperidinopropyn-1'-yl-1')pyridine N-oxide (c)	37,5	83,4
N-oxide - 2-methyl-5-(3'-morpholinopropyn-1'-yl-1')pyridine (d)	33,2	76,6
2-methyl-5-(3,-N-benzox-zolonyl propyn-1'-yl-1')pyridine N-oxide (e)	47,2	64,7
2-Methyl-5-(3'-N-(6-nitrobenzoxazolonylpropin-I'-yl-1') pyridine N-oxide (l)	49,4	68,3

The molar ratio of the initial components of the reaction mixture also has a certain effect on the formation of PAA N-oxides. These processes were carried out both in the absence and in the presence of the Cu_2CI_2 catalyst in boiling n-dioxane for 8 hours. In both cases, the mole fraction of 2-M-5-ENP N-oxide was 1.0. The results obtained for the reaction of aminomethylation of 2-M-5-ENP N-oxide with diethylamine are presented in Table 5.

Table 5

The dependence of the yield of substances (b) on the molar ratios of diethylamine with other initial reagents

Molar ratio		Yields of compounds, %	
PFA	Diethylamine	In the absence of a catalyst	In the presence of a catalyst
Substance (b)			
1.0	1.0	49,7	67,6
1.3	1.0	51,6	69,1
1.3	1.3	56,6	74,4
1.5	1,3	61,6	81,8
1,5	1.5	61,6	82,3

It follows from the data in Table 5 that, under comparable conditions, with an increase in the content of PFA and amines, the yields of substances (b) both in the presence of Cu_2CI_2 and in its absence increase symbatically. This unambiguously indicates that the initial components are equally responsible for the synthesis of the corresponding PAA N-oxides. The reasons for the increase in product yields in the studied processes in the presence of Cu_2CI_2 have already been discussed.

It should be noted that all synthesized PAA N-oxides are white or light yellow crystalline substances, easily soluble in most organic solvents, for example, methanol, ethanol, chloroform, dimethylformamide and dimethyl sulfoxide, as well as in water.

Conclusion

Some N-oxides of pyridylacetylenic aliphatic and heteroaromatic amines were synthesized by the Mannich reaction.

The structure and composition of the synthesized PAA N-oxides were determined using IR and PMR spectroscopic methods.

The effect of the nature of amines, solvents, catalyst, reaction time, and molar ratio of initial components on the formation of PAA N-oxides was studied. Under the optimal condition, i.e. in an n-dioxane medium in the presence of a copper chloride catalyst in a reaction time of 8 hours at a temperature of 90 °C, the yields of PAA N-oxides are as follows: 2-methyl-5-(3'-dimethylaminopropyn-1'-yl-1') -pyridine (83.3%); 2-methyl-5-(3'-diethylaminopropyn-1'-yl-1')-pyridine N-oxide (82.3%); N-oxide-2-methyl-5-(3'-piperidinopropin-I'-yl-1')pyridine (83.4%); N-oxide 2-methyl-5-(3'-morpholinopropin--I'-yl-1')pyridine (76.6%); N-oxide-2-methyl-5-(3'-N-

benzoxazolonyl-propyn-1'-yl-1')pyridine (64.7%) and N-oxide-2-methyl-5-/3', - N -(6-nitrobenzaxozolonyl propyn-1'-yl-I')/pyridine (68.3%)

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