Корутунду.

1. Изилденген аймактын булганышынын негизги очогу болуп, тоо-кен комбинаты жана калдык сактоочу жерлер аныкталды. Топурак катмарында оор металдардын концентрациясын ЧНКга салыштырганда жогору жана чогултулган үлгүлөрдүн булгануучу ордунун аралыктарынын алыстыгына жараша болооору белгиленди.
2. Калдыктарды сактоочу жайдагы топурактын үлгүлөрүндө айрым оор металлдардын: Pb, Sb, Zn, Sn, Cd, жогорку концентрациясы табылган. Суунун үлгүлөрүндө табылган оор металдардын ичинен бир гана Sb нын жогорку концентарциясын көрсөттү.
3. Изилденген үлгүлөрдөгү оор металдар сууга салыштырмалуу топуракта жогорку концентрацияны көрсөттү. Изилдөөнүн жыйынтыгы боюнча калдык төгүлгөн жерлердин экологиялык компоненттерге тескери техногендик таасирлерин азайтууда мониторинг жүргүзүп, сунуш жана иш чараларды иштеп чыгуу зарыл экендигин көрсөтөт.

Колдонулган адабияттар.

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PRODUCTION OF N-OXIDES OF PYRIDYLACETYLENE AMINES OBTAINED FROM 2-METHYL-5-ETHYNYLPYRIDINE

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Annotation: This work reports the investigation of reactions between 2-methyl-5-ethynylpyridine N-oxide and a range of aliphatic and aromatic amines, leading to the formation of pyridylacetylene amine N-oxides. The synthesis route involved the dehydrogenation of 2-methyl-5-ethylpyridine to yield 2-methyl-5-vinylpyridine, followed by bromination and dehydrobromination to afford 2-methyl-5-ethynylpyridine. Its oxidation with perhydrol in acetic anhydride provided 2-methyl-5-ethynylpyridine N-oxide. Subsequently, the Mannich reaction was employed for the preparation of N-oxides of pyridylacetylene amines. The effects of solvents (methanol, ethanol, and n-dioxane), the use of a catalyst, and reaction time on product yields were systematically examined. The structural features and composition of the synthesized pyridylacetylene amine N-oxides were confirmed through IR and PMR spectroscopic analysis.

Keywords: 2-methyl-5-ethynylpyridine, methylamine, ethylamine, piperidine, morpholine, catalyst, solvents, pyridylacetylene amine N-oxides.

ПРОИЗВОДСТВО N-ОКСИДОВ ПИРИДИЛАЦЕТИЛЕНОВЫХ АМИНОВ, ПОЛУЧЕННЫХ ИЗ 2-МЕТИЛ-5-ЭТИЛЕНИЛПИРИДИНА

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Аннотация: В данной работе представлены исследования реакций между N-оксидом 2-метил-5-этинилпиридина и рядом алифатических и ароматических аминов, приводящих к образованию N-оксидов пиридилацетиленовых аминов. Синтетический путь включал дегидрирование 2-метил-5-этилпиридина с получением 2-метил-5-винилпиридина, последующее бромирование и дегидробромирование с образованием 2-метил-5-этинилпиридина. Его окисление пергидролем в уксусном ангидриде привело к получению N-оксида 2-метил-5-этинилпиридина. Далее реакция Манниха была использована для синтеза N-оксидов пиридилацетиленовых аминов. Систематически исследовалось влияние растворителей (метанол, этанол и n-диоксан), применения катализатора и времени реакции на выход продуктов. Структурные особенности и состав синтезированных N-оксидов пиридилацетиленовых аминов были подтверждены методами ИК- и ПМР-спектроскопии.

Ключевые слова: 2-метил-5-этинилпиридин, метиламин, этиламин, пиперидин, морфолин, катализатор, растворители, N-оксиды пиридилацетиленовых аминов

INTRODUCTION

At present, a wide range of biologically active compounds has been synthesized on the basis of pyridine and its homologues, including herbicides of both continuous and selective action, insecticides, fungicides, and bactericides, which are broadly applied in agriculture [1]. Among them, compounds such as nitropyrin and 2-chloro-6-(trichloromethyl)pyridine serve as nitrogen fertilizer stabilizers in soils. Furthermore, a number of clinically important pharmaceuticals have been developed from these derivatives, such as ftivazide, saluzide, and metazid, which are used in the treatment of tuberculosis. In addition, pyridine derivatives have also found application as vitamins, monomers and polymers, lubricating oil additives, corrosion inhibitors for metals, suspension stabilizers, extractants, dyes, and analytical reagents [2].

The synthesis and chemical transformations of numerous pyridine bases (PB) have been investigated in detail in several studies [3–9]. Nevertheless, some classes of pyridine derivatives remain poorly explored or have not been studied at all.

It is well established that heteroaromatic N-oxides represent important intermediates in the synthesis of substituted pyridine derivatives and other nitrogen-containing heterocycles. Their enhanced reactivity in electrophilic and nucleophilic substitution reactions, compared to the corresponding non-oxidized bases, makes them valuable in synthetic chemistry [10]. Various approaches have been employed for the preparation of N-oxides of nitrogen heterocycles. For instance, the N-oxide of diethyl ester of 3,4-pyridinedicarboxylic acid was synthesized using a urea–hydrogen peroxide complex in the presence of acetic anhydride [11].

The range of applications of N-oxides is broad. For example, solid-state complexes of N-oxides of 2,6-lutidines, 2-picoline, and pyridine with succinic acid (2:1 composition) were characterized by IR spectroscopy, and according to their electrical properties, they were classified as protonic semiconductors [12]. It has also been reported that pyridine N-oxides can form unstable molecular complexes with alkali and alkaline earth metal cations [13, 14], which arise in aqueous solutions containing a large excess of complexing ions. When interacting with SiO_2 in aqueous medium, pyridine N-oxides yield unstable pentacoordinated silicon complexes containing an O–Si bond [15]. The formation of such complexes accounts for the interesting observation that N-oxides significantly enhance the solubility of SiO_2 in water.

Solid complexes of $ZnCl_2$ with pyridine and methylpyridine N-oxides (1:2 ratio) display distinct crystal structures depending on temperature. The electron-donating effect of a methyl substituent considerably increases the O–Zn bond enthalpy compared with the unsubstituted analogue. In many instances, N-oxides also demonstrate a much stronger catalytic activity than their corresponding unoxidized bases, with numerous examples of their catalytic potential reported in the literature.

Despite the significant interest in pyridine N-oxides, data on acetylene-containing pyridine base N-oxides remain scarce. The present study is therefore devoted to the synthesis of several representatives of this class of compounds.

RESEARCH METHODS

The objects of the present study include 2-methyl-5-ethynylpyridine N-oxide, a series of aliphatic and heteroaromatic amines, paraformaldehyde, perhydrol, acetic anhydride, and an AHCO catalyst (Al_2O_3 – 70.0%, Fe_2O_3 – 25.0%, Cr_2O_3 – 5.0%). Initially, 2-methyl-5-ethylpyridine was subjected to dehydrogenation at 380 °C in the presence of the AHCO catalyst to afford 2-methyl-5-vinylpyridine. Subsequent bromination followed by dehydrobromination yielded 2-methyl-5-ethynylpyridine, which was then oxidized with perhydrol in acetic anhydride medium to give 2-methyl-5-ethynylpyridine N-oxide.

The aminomethylation of 2-methyl-5-ethynylpyridine N-oxide was carried out via the Mannich reaction with dimethylamine, diethylamine, piperidine, morpholine, benzoxazolone, and nitrobenzoxazolone. For this purpose, the calculated quantities of 2-methyl-5-ethynylpyridine N-oxide, finely powdered paraformaldehyde, the selected amine, n-dioxane, and a catalytic amount of anhydrous copper acetate or copper chloride were placed into a round-bottom flask equipped with a reflux condenser. The reaction mixture was heated on a water bath at 90–95 °C with stirring for 4–6 hours. After cooling, the reaction mass was diluted with water and treated with a 10% potassium carbonate solution. The organic phase was separated, while the aqueous layer was additionally extracted with chloroform. The combined organic layer and chloroform extracts were dried over potassium carbonate, followed by removal of n-dioxane by water-bath distillation, and the residue was purified by vacuum distillation.

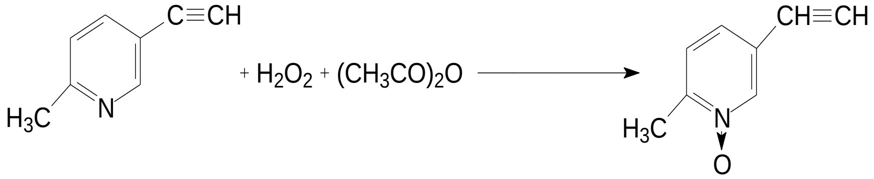
RESULTS

Further transformations of 2-methyl-5-ethylpyridine (2-M-5-EP), the primary product of the interaction between crotonaldehyde (CA) and ammonia, have attracted considerable interest [1, 2]. In particular, the feasibility of employing newly developed catalysts for the heterogeneous dehydrogenation of 2-M-5-EP to obtain the valuable monomer 2-methyl-5-vinylpyridine (2-M-5-VP) has been demonstrated. The process was carried out at 360–390 °C in the presence of a catalyst, with the most effective results achieved using the AHCO catalyst (Al_2O_3 – 70.0%, Fe_2O_3 – 25.0%, Cr_2O_3 – 5.0%), which afforded 2-M-5-VP in a yield of 41.05%.

On the basis of 2-M-5-VP, 2-methyl-5-ethynylpyridine (2-M-5-ENP) was subsequently synthesized, representing an important starting reagent for the preparation of numerous novel pyridine derivatives [3, 4]. The yield of 2-M-5-ENP under these conditions reached 60–65%.

The next stage involved exploring the synthesis of pyridylacetylenic amines (PAA) derived from 2-M-5-ENP under Mannich reaction conditions. Interest in these compounds is largely driven by their established biological activity [5-12].

As has been emphasized previously, N-oxides of pyridine bases (PB) are of significant practical relevance [13–15]. Guided by this, we investigated the synthesis of such derivatives based on 2-M-5-ENP. For this purpose, peracetic acid was generated in situ via the interaction of perhydrol with acetic anhydride, and the reaction proceeded according to the following scheme:



The yield of the obtained N-oxide was **63.1%**. Subsequently, the reactivity of the synthesized 2-M-5-ENP N-oxide was examined in its interaction with dimethylamine, diethylamine, piperidine, morpholine, benzoxazolone, and nitrobenzoxazolone. These reactions were found to proceed both in the absence and in the presence of catalysts, demonstrating the pronounced reactivity of 2-M-5-ENP N-oxide.

A number of physicochemical parameters of the synthesized compounds were determined (Table 1).

The **IR spectra** of the obtained products were recorded using a UR-22 spectrophotometer within the spectral range of 400–4000 cm^{-1}. For this purpose, solid samples were analyzed in the form of KBr pellets.

The **PMR spectra** were obtained on a C-60HI spectrometer in deuterochloroform solution.

Table 1

Selected physicochemical characteristics 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, such as *2-methyl-5-(3'-dimethylaminopropyn-1'-yl-1') pyridine N-oxide* and *2-methyl-5-(3'-diethylaminopropyn-1'-yl-1') pyridine*, intensive absorption bands are observed in the region of **1630 cm^{-1}**, corresponding to the **HC=CH bond**. The signals at **1270 cm^{-1}** are attributed to the **tertiary amino group**, while those at **1468 cm^{-1}** belong to the **–CH_2 groups**. Absorptions characteristic of the **C≡C bond** are weakly manifested in the range of **2220–2100 cm^{-1}**, whereas in the **850 cm^{-1}** region, bands corresponding to the **N→O group** are detected. Additionally, peaks at **1480–1440 cm^{-1}** are indicative of the pyridine ring. The spectrum of the second N-oxide also displays a broad band at **3400–3100 cm^{-1}**.

For the **IR spectra of heterocyclic PAA N-oxides**, absorption bands appear in the region of **1090–1080 cm^{-1}**, associated with the **piperidine group**, and at **1120–1080 cm^{-1}**, corresponding to the **morpholine group**. Additional characteristic bands are present at **1270 cm^{-1}, 1580–1530 cm^{-1}, 2200–2100 cm^{-1}, and 2900–2790 cm^{-1}**, reflecting the structural fragments of these compounds.

In the spectra of heteroaromatic PAAs, including *2-methyl-5-(3'-N-benzoxazolonylpropyn-1'-yl-1') pyridine* and *2-methyl-5-(3'-N-(6"-nitrobenzoxazolonyl)propyn-1'-yl-1') pyridine*, intense absorption is noted near **1630 cm⁻¹** (HC=CH bond). Bands at **1270 cm⁻¹** are associated with **tertiary amino groups**, while **1468 cm⁻¹** corresponds to **CH_2 groups**, and absorptions at **1800–1750 cm⁻¹** are attributed to the **C=O group**. In the nitro-containing compound, distinct absorptions near **1400 cm⁻¹** confirm the presence of the **NO_2 group**. The **C≡C bond** absorptions are weakly represented at **2220–2100 cm⁻¹**.

The **PMR spectra** of aliphatic PAA N-oxides display strong signals within **6.5–8.5 ppm**, characteristic of protons in the **α, β, and γ positions of the pyridine nucleus**. Signals at **2.7–2.8 ppm**, **3.2–3.3 ppm**, and **2.2–2.6 ppm** are assigned to **methyl group protons** in various positions.

In the **PMR spectrum** of *2-methyl-5-(3'-piperidinopropyn-1'-yl-1') pyridine N-oxide*, resonances appear at **5.6–7.4 ppm**, corresponding to protons in the pyridine ring (α, β, γ positions). Additional peaks at **2.2–2.5 ppm** are attributed to the **methylene group** and piperidine ring protons, while signals at **1.1–1.4 ppm** correspond to **methyl protons**.

For *2-methyl-5-(3'-morpholinopropyn-1'-yl-1') pyridine N-oxide*, the PMR spectrum shows intense resonances at **6.6–8.5 ppm** (pyridine α, β, γ protons), signals at **3.3–3.8 ppm** assigned to **methylene and morpholine protons**, and peaks at **2.2–2.8 ppm** corresponding to **methyl protons**.

To clarify the **influence of solvent nature** on these reactions under comparable conditions, the **aminomethylation of 2-M-5-ENP N-oxide** was carried out **without a catalyst** in methanol, ethanol, and *p*-dioxane at their respective boiling points. In each case, the corresponding **target PAAs were successfully synthesized** (Table 2).

Table 2

Effect of amine type and solvent nature on the synthesis of PAA N-oxides in the absence of catalysts (reaction time – 8 h; molar ratio of N-oxide:2-M-5-ENP:PFA: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

Analysis of the data presented in **Table 3** indicates that the yields of all PAA N-oxides reach their **maximum within 6–7 hours**, after which they remain virtually unchanged. This stabilization can be attributed both to the accumulation of target products and to the reduction in the concentration of initial reactants in the reaction medium.

The synthesis of PAA N-oxides is significantly enhanced in the presence of the **Cu_2Cl_2 catalyst** (2.5% of the total mass of the reaction mixture) (**Table 4**). This effect, as noted earlier, is associated with the formation of **active transition π-complexes** between Cu^{2+} ions and the C≡C bond of the starting pyridylacetylene derivative.

For instance, during the aminomethylation of **2-M-5-ENP N-oxide** with **dimethylamine** in *n*-dioxane at its boiling point, the yield of compound (a) reaches **83.3%,** which is **21.2% higher** than under catalyst-free conditions. A comparative evaluation of the Mannich reactions with other amines revealed similar trends: the yields of compounds (b), (c), (d), (e), and (g) increased by **20.6%, 45.9%, 43.4%, 17.5%, and 18.9%,** respectively, when the catalyst was applied.

Overall, depending on the nature of the amine used, the presence of **Cu_2Cl_2** ensures product yields in the range of **64.7–83.3%,** confirming the pronounced catalytic effect of copper(I) chloride on these transformations.

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

The analysis of the data presented in Table 5 demonstrates that, under comparable conditions, an increase in the concentration of PFA and amines leads to a proportional rise in the yields of compounds (b), both in the presence and absence of Cu_2Cl_2. This clearly indicates that both initial components play an equally important role in the synthesis of the corresponding PAA N-oxides. The catalytic effect of Cu_2Cl_2, contributing to the enhanced yields observed in these processes, has been discussed earlier.

It should be emphasized that all the synthesized PAA N-oxides represent crystalline substances of white or light-yellow coloration. They exhibit high solubility in a wide range of organic solvents, including methanol, ethanol, chloroform, dimethylformamide, and dimethyl sulfoxide, as well as in water.

CONCLUSION

In the present work, a series of N-oxides of pyridylacetylenic aliphatic and heteroaromatic amines were successfully synthesized via the Mannich reaction.

The structure and composition of the obtained PAA N-oxides were confirmed by IR and PMR spectroscopic analysis. A systematic study was carried out on the influence of the nature of amines, solvents, catalysts, reaction duration, and molar ratios of initial reagents on the efficiency of PAA N-oxide formation.

Under optimized conditions—namely, in an *n*-dioxane medium, in the presence of copper chloride catalyst, at 90 °C with a reaction duration of 8 hours—the following yields of PAA N-oxides were obtained:

- 2-methyl-5-(3'-dimethylaminopropyn-1'-yl-1')pyridine N-oxide – 83.3%
- 2-methyl-5-(3'-diethylaminopropyn-1'-yl-1')pyridine N-oxide – 82.3%
- 2-methyl-5-(3'-piperidinopropyn-1'-yl-1')pyridine N-oxide – 83.4%
- 2-methyl-5-(3'-morpholinopropyn-1'-yl-1')pyridine N-oxide – 76.6%
- 2-methyl-5-(3'-N-benzoxazolonylpropyn-1'-yl-1')pyridine N-oxide – 64.7%
- 2-methyl-5-(3'-N-(6-nitrobenzoxazolonyl)propyn-1'-yl-1')pyridine N-oxide – 68.3%

These results confirm that the applied methodology is effective for the synthesis of diverse PAA N-oxides and allows regulation of product yields through variation of reaction parameters.

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ОЧИСТКА ВОДЫ ОТ ИОНОВ ХРОМА С ПОМОЩЬЮ БЕНТОНИТОВ И ТЕРМОДИНАМИКА АДСОРБЦИИ

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