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CATALYTIC SYNTHESIS OF PYRIDINES BASED ON THE CROTON FRACTION

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The dimensional mismatch of gold with the silicon crystal lattice is due to the ionic and covalent radii of 1.37 \AA , 1.50 \AA compared to the covalent radius of silicon 1.17 \AA . Therefore, gold in silicon is unlikely to be located in the nodes of the crystal lattice. Electronic correspondence implies the possibility of the formation of a stable chemical bond between the impurity atom and the surrounding atoms at the sites of the crystal lattice. For the electronic correspondence of gold with a node of the crystal lattice of silicon, 3 electrons are not enough. Therefore, gold atoms cannot be located in the nodes of the silicon crystal lattice because of the dimensional and electronic mismatch with the silicon crystal lattice. On the other hand, if gold impurity atoms are found in the nodes of the silicon crystal lattice, the charge carriers of the impurity gold ion should have an effective mass density in the state of the corresponding band, that is, the acceptor level should be bound to the top of the valence band, and the donor level with the bottom of the conduction band ... In the cited works [2, 7], the acceptor level $E_c - 0,54 \text{ eV}$ is observed in the upper half of the band gap of silicon, and the donor level $E_v + 0,35 \text{ eV}$ in the lower half of the band gap. Here it is appropriate to assert [1] that several complexes of monovalent gold are known that are stable in aqueous solution; the most important of them are $\text{Au}(\text{CN})_2^-$, AuCl_2^- . The cyanide complex is highly stable; it is formed by the interaction of AuCN with an excess of cyanide or, more often, by the dissolution of gold in alkali metal cyanide in air or in the presence of hydrogen peroxide. Crystalline compounds of the $\text{Au}(\text{CN})_2$ type can be obtained. The nature of gold tends to form complexes of gold with carbon-based compounds such as $\text{Au}(\text{CN})_2$. It can be assumed that the nature of the acceptor level of gold in silicon with a level of $E_c - 0,54 \text{ eV}$ is associated with the formation of a spatially separated vapor of gold with a carbon complex such as CO , CO_2 , or others.

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Abstract:

Background. *The presence in the molecule of pyridine bases of nitrogen atoms having the properties of the bases determines the specificity of the products of this class and gives ample opportunities for their use in various industries. . The chemistry of pyridine bases is associated with such general theoretical issues as the electronic structure of the cycle, aromaticity, the effect of substituents on the reactivity of heterocycles in nucleophilic substitution reactions, and*

tautomerism. At the same time, systematic data on the reactions of alkylation with dihalo-alkanes, acylation under low-temperature poly-condensation, and complexation were not available to create a complete picture of the reactivity of pyridine bases.

Materials and methods. *The aim of the work is to develop cheap and convenient methods for the preparation of pyridine bases, to search for effective and selective process catalysts, and to determine the degree of anticorrosion protection of metals of the obtained products.*

Results and discussions. *The catalytic synthesis of nitrogen heterocyclic compounds using the croton fraction and ammonia was studied. New multifunctional catalysts based on iron, zinc, cadmium and chromium oxides were proposed, the selectivity for the yield of pyridine bases was determined from 58.0 - 88.0 %, while the conversion of the croton fraction was 98 %.*

Conclusion. *The obtained pyridine bases were tested as corrosion inhibitors; it was determined that with an increase in the amount of methyl pyridine in the mixture, the degree of protection increases. Nowadays, methods have been developed for the technical synthesis of alkyl-pyridines from various organic compounds. All alkyl-pyridine production plants in the world use only carbonyl compounds and ammonia as raw materials.*

Keywords: *pyridine, pyridine bases, hetero-cyclization, catalyst, catalysis, selectivity, conversion, croton fraction, ammonia, corrosion inhibitor, degree of protection*

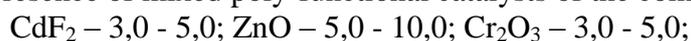
Introduction. The great interest currently shown in the chemistry of heterocyclic compounds and, in particular, derivatives of pyridine and pyridine bases is associated with the peculiar structure and properties of these amino-azoles. The chemistry of pyridine bases is associated with such general theoretical issues as the electronic structure of the cycle, aromaticity, the effect of substituents on the reactivity of heterocycles in nucleophilic substitution reactions, and tautomerism. At the same time, systematic data on the reactions of alkylation with dihalo-alkanes, acylation under low-temperature poly-condensation, and complexation were not available to create a complete picture of the reactivity of pyridine bases. Therefore, the study of the synthesis and structure of pyridine bases is important both for the development of heterocycle chemistry and for the theory of organic chemistry [1-3].

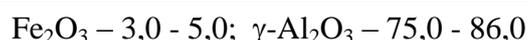
The presence of basic nitrogen in alkyl pyridines gives this class of compounds a specific ability, as a result of which they are widely used in various industries. The main consumers of alkyl pyridines are the chemical, including polymer, and pharmaceutical industries.

2 - and 4-methylpyridines and 2-methyl-5-ethylpyridine are used as raw materials for the production of 2 - and 4-vinylpyridines and 2-methyl-5-vinylpyridine, which are used to make latexes for impregnation of soft cord, ion exchange resins, film photographic materials, etc. Many physiologically active drugs used in agriculture and healthcare are produced on the basis of pyridine and methyl pyridines. Derivatives of 3-and 4-methylpyridines are effective agents for the treatment of tuberculosis. Due to the ever-increasing demand for alkyl pyridines and their limited production in the coke-chemical industry, many industrialized countries have developed and implemented the production of synthetic alkyl pyridines. The resources of alkyl pyridines extracted from coal tar are very limited. In addition, coal tar contains more than 70 pyridine isomers in its composition, and the isolation of individual bases in pure form is a very time-consuming process. In addition, coal tar contains mainly pyridine and 2-methyl pyridine in its composition.

Method of research. The paper uses chromium-mass spectroscopy: Agilent Technology GC 6890/MS 5973N chromate-mass spectrometer using a 30m×0.25 mm capillary column with 5 % phenyl-methyl-siloxane in dimethyl-siloxane, carrier gas-hydrogen, injector temperature-280°C, source temperature-230°C, quadrupole temperature MS - 180°C, when programming the temperature of the column thermostat from 100 to 280°C, the temperature rise rate of 10°C min, the sample size of 1 µl., in the mode without flow division, in addition, standardized test methods were studied to determine the physical, mechanical and technological properties, etc.

Experimental part. The reaction of hetero-cyclization of the Croton fraction with aniline and o-aminophenol in the presence of mixed poly-functional catalysts of the composition, % wt:





The catalysts were prepared by suspension, molding, washing, drying, and calcination. Aluminum hydratoxide was used as a carrier, (PPP-33%). 3 - 5% solutions of hydrofluoric and acetic acids were used as a peptirating agent. The textural characteristics of the developed catalysts were determined (table 1).

Table 1

Physical, chemical and operational characteristics of the developed catalysts

№	Composition, % by weight.	Specific surface area mg / h	Mechanical strength, MPa	Service life before regeneration, hour	Productivity, g / kg-kat-hour
1.	CdF ₂ - 3,0; ZnO - 5,0; Cr ₂ O ₃ - 5,0; Al ₂ O ₃ - 87,0	225	6,8	68,0	81,0
2.	CdF ₂ - 3,0; ZnO - 7,0; Fe ₂ O ₃ - 5,0; Al ₂ O ₃ - 83,0	168	7,1	76,0	92,0
3.	CdF ₂ - 5,0; ZnO - 5,0; Cr ₂ O ₃ - 3,0; Fe ₂ O ₃ - 3,0; Al ₂ O ₃ - 84,0	240	7,8	96,0	101,0
4.	CdF ₂ - 5,0; ZnO - 5,0; Cr ₂ O ₃ - 5,0; Fe ₂ O ₃ - 3,0; Al ₂ O ₃ - 82,0	225	7,3	72,0	98,0
5.	CdF ₂ - 5,0; ZnO - 10,0; Cr ₂ O ₃ - 3,0; Fe ₂ O ₃ - 5,0	196	8,0	120	122
6.	CdF ₂ - 5,0; ZnO - 5,0; Cr ₂ O ₃ - 5,0; Fe ₂ O ₃ - 5,0; Al ₂ O ₃ - 80,0	184	8,2	184	130

The condensation reaction of the croton fraction with ammonia and amines was carried out under flow conditions in a stainless steel reactor of size $d \times l = 25 \times 1000$ mm with external electric heating and a 100 cm³ bulk volume of the catalyst.

The reaction of the croton fraction with ammonia in the presence of catalyst № 6 was studied at temperatures of 360-420 °C. It was found that the main reaction products are a mixture of 2 - and 4-methylpyridine (up to 50 %), 2-methyl-5-ethylpyridine (up to 26%). As by - products, acetonitrile, a mixture of di-and tri-methyl-pyridine, resin, and water are formed.

Research results and discussions. Methods for the synthesis of pyridines and alkyl-pyridines are very diverse. Thus, in [4], methodological aspects of the synthesis of pyridine bases by the reaction of cross-metathesis of olefins are proposed, which provides a fast and efficient method in which the initial raw material uses α , β -unsaturated derivatives of 1,5-dicarbonyl.

Chinese scientists [5] propose a method for producing aryl-pyridines by the interaction of acetophenones and 1,3-diaminopropane, which is catalyzed by copper.

It is proposed to synthesize [6] a pharmacologically significant 2,4,5-tri-substituted pyridine by region-selective hydro-amination of alkynes using N-silylamine using a bis-(amidate)-bis-(amido) precatalyst)- titanium (IV), adding α , β -unsaturated carbonyls to the crude mixture, followed by oxidation. This method synthesized 47 examples of pyridines with a good yield containing variable structure substitution.

S.I. Sherbinina and others [7] proposed a photoredox combination of α,α -difluoro- β -iodocetones with silyl esters, catalyzed by iridium salts under irradiation with a blue led followed by condensation in a single vessel with ammonium acetate, with the formation of various substituted 3-fluoropyridines.

The reaction of vinylazides with monocyclic cyclopropanols produces pyridines in the presence of Mn salts, whereas reactions with bicyclic cyclopropanols lead to the formation of 2-azabicyclo [3.3.1] non-2-EN-1-ol derivatives using Mn catalytic agents [8].

In [9], the reaction of 2-allyl-2H-azyryns promoted by DBU was studied, in which 1-azatrienes are formed, then electrocyclicized to pyridines with very good yields. The reaction demonstrates a wide volume of substrate and transfers various substituents. In addition, the synthesis of pyridines from oximes in one vessel was obtained by the formation of 2H-azyryns.

The catalytic production of mixtures of pyridine bases, vapor-phase catalytic condensation of acetylene with ammonia, or a mixture of acetylene with methanol was considered very promising. Depending on the starting products, mixtures of only pyridine and 3-methylpyridine or only 2 - and

4-methylpyridine are obtained. Various catalysts were proposed for the process: oxides of heavy metals-molybdenum, cadmium, tungsten, vanadium, chromium, zinc, iron, thorium, and aluminum. Some authors used various heavy metal salts for cyclization of acetylene with ammonia: chromate, molybdate, tungstate, and cadmium vanadate on acid clay, as well as cadmium phosphate on fuller soil [10,11].

The reaction of synthesis of pyridine and isomeric methylpyridines from acetylene and ammonia (methanol) in the presence of a cadmium calcium phosphate catalyst by acetylene hydration was studied [12,13]. It was found that when a mixture of acetylene and ammonia is passed in the ratio $C_2H_2: NH_3 = 1: 2$ mol at a temperature of 420-440 °C with a total volume rate of 100 $hour^{-1}$, a catalysate is formed that contains 2-methyl-pyridine-56.3 %, 4-methyl-pyridine-31.4 %.

Carrying out the process in the presence of methanol leads to the formation of a mixture of pyridine and isomeric methyl-pyridines. In this case, the content of pyridine reaches 27.0 %; 2-methylpyridine-22.0 %; 3-methylpyridine-22.0 %, a cadmium-calcium-phosphate catalyst with constant activity works for up to 22 hours, after which it requires regeneration. Its total service life does not exceed 100 hours.

Nowadays, methods have been developed for the technical synthesis of alkyl-pyridines from various organic compounds. All alkyl-pyridine production plants in the world use only carbonyl compounds and ammonia as raw materials.

The process of producing a mixture of pyridine and 3-methyl-pyridine from carbonyl compounds and ammonia is a well-studied process. "Navoiyazot" JSC has established production of acetaldehyde with a capacity of 20 thousand tons per year and formaldehyde – 7 thousand tons per year. In this regard, it was interesting to study the process of co-production of pyridine and 3-methylpyridine from acetaldehyde, formaldehyde and ammonia.

The reaction of hetero-cyclization of carbonyl compounds with ammonia (and amines) refers to complex parallel-sequential reactions, including reactions of nucleophilic addition of ammonia to the triple bond of isomerization, dehydro-cyclization, etc. The combination of all these processes is possible in the vapor phase in the presence of catalysts with poly-functional properties. Based on this, the selection of the composition of the catalysts took into account the role of each component in the reaction of formation of pyridine and methyl-pyridines.

Elements with extremely filled d^{10} -(Cu^{+1} , Ag^{+1} , Au^{+2} , Hg^{+2} , Zn^{+2} , Cd^{+2} , Pt^{+2} , Ni^0 , etc.) are able to organize π -complexes with acetylene compounds, i.e., if these activation elements are present in the catalyst, the acetylene component is characterized, on the one hand, by its deformation due to partial displacement of a pair of π - electrons of acetylene to the free s-orbits of the ion and, on the other hand, by the formation of a so-called dative or a donor π bond consisting in the transition of electrons from the d-orbit of an ion to the loosening orbit of acetylene. The transfer of electrons from the π ligands to the metal and from the metal to the π ligand can lead to a noticeable polarization of the unsaturated molecule. The formation of a metal-carbonyl group donor-acceptor bond leads to a decrease in the electron density on carbon atoms $-C = O$ bond. This increases the electro-philicity of the multiple bond and its ability to interact with nucleophilic reagents.

Indeed, the elements and molecules of d^{10} -orbitals proved to be effective catalysts in the reactions of acetylene transformations. The use of zinc oxide and chromium is due to the fact that they are widely used in the synthesis and dehydrogenation of methanol and dehydro-cyclization processes.

Since iron oxides are used as a contact in the synthesis of ammonia, we assumed that the presence of the latter in the catalyst composition should contribute to the adsorption and activation of ammonia during the synthesis of pyridine bases.

We have studied the influence of temperature, volume velocity, ratio of initial reagents, height of the catalyst layer, etc. output of target products and conversion of the croton fraction:



The study of flow rate on yields of products, conversion of crotonic fraction and the selectivity of the process showed that with the increase in flow rate of ammonia from 150 to 300 hr⁻¹, crotonic fraction from 0.1 to 0.7 hr⁻¹ and gradually decreases the yield of pyridines. This shows that the process takes place in the internal diffusion region.

The influence of temperature was studied in the range of 360-430 °C (table 2).

Table 2

Effect of temperature on the yield of target products.
Croton fraction: ammonia = 1 : 2; V_{total} = 200 hr⁻¹, catalyst № 6

Temperature, °C	Content in the catalyst, %			Σ of by-products	Conversion of croton fraction
	2-methylpyridine-	4-methylpyridine-	2-methyl-5-ethylpyridine-		
360	14,0	8,0	8,0	12,0	45,0
370	18,0	12,0	11,0	14,0	56,0
380	22,0	16,0	14,0	18,0	64,0
390	32,0	22,0	20,0	26,0	85,0
400	38,0	25,0	25,0	12,0	95,0
410	36,0	24,0	26,0	14,0	97,0
420	37,0	23,0	25,0	15,0	97,0
430	33,0	22,0	20,0	25,0	98,0

As can be seen from the table data, the reaction does not go below 360 °C. As the temperature increases from 360 °C to 400 °C, the yield of the target products gradually increases, reaching a maximum at 400 °C. A further increase in temperature leads to a decrease in the yield of target products due to the occurrence of side reactions – tarring, polymerization, decomposition, etc.

Table 3

**Determination of the inhibitory properties of the obtained mixtures
by gravimetric method depending on the concentration
(T = 70 °C, solution-1% NaCl + 1% H₂SO₄, pH of the medium = 5.3)**

Inhibitors	Inhibitor Concentration %	Experiment time					
		15 days			30 days		
		K, g/m ² ·days	γ	E, %	K, g/m ² ·days	γ	E, %
Without inhibitor	-	398,86	-	-	354,04	-	-
mixture №1	0,1	74,93	6,92	82,21	58,96	7,88	83,41
mixture №2		81,41	18,11	80,82	77,31	20,09	81,82
mixture №3		101,25	41,25	75,43	87,33	48,35	79,84
mixture №4		55,44	9,84	87,85	51,32	11,48	90,73
mixture №5		6,24	60,81	99,65	4,25	102,41	99,75
mixture №6		8,19	58,84	98,80	6,09	97,69	99,03
mixture №7		27,68	12,45	93,37	25,05	14,43	95,46
mixture №8		78,66	20,13	81,06	75,58	24,32	83,25
Without inhibitor	-	398,81	-	-	355,01	-	-
mixture №1	1,0	72,43	8,84	85,32	70,45	9,63	86,41
mixture №2		75,38	20,15	82,93	73,18	21,48	83,09
mixture №3		88,46	43,85	80,64	85,95	50,51	82,55
mixture №4		58,53	12,81	91,36	61,58	12,83	92,64
mixture №5		4,03	68,74	99,81	3,35	104,52	98,89
mixture №6		8,04	61,43	98,92	5,89	98,08	98,02
mixture №7		26,03	15,05	95,88	16,03	15,81	86,44
mixture №8		73,04	23,48	85,47	28,41	26,44	87,03

In order to find ways of application, the obtained products were tested as corrosion inhibitors in hydrochloric acid (tables 3 and 4).

As can be seen from these tables, the degree of corrosion protection of the mixture №5, №6 and №7 in an acidic environment (H_2SO_4) at a temperature equal to 50 °C, at a concentration of 0.1 % becomes in the range of 86.2 ÷ 92.4 %. When the concentration is increased to 1.0 % and the temperature is up to 70 °C, the degree of corrosion protection is 99.4 ÷ 91.4 % in the aisles.

Table 4

**Determination of the inhibitory properties of the obtained mixtures
by the gravimetric method depending on the temperature
(inhibitor concentration 1.0 %, solution-3 % Na_2S + 3 % H_2SO_4 , medium pH = 6.1)**

Inhibitors	Temperature, °C	Experiment time					
		15 days			15 days		
		K, g/m ² ·days	γ	E, %	K, g/m ² ·days	γ	E, %
Without inhibitor	50	142,37	-	-	101,75	-	-
mixture №1		39,79	6,18	83,85	36,95	6,85	84,58
mixture №2		43,56	18,93	80,54	40,81	17,18	83,45
mixture №3		44,05	19,02	80,63	42,34	5,64	81,39
mixture №4		6,29	39,98	97,51	5,18	58,41	98,15
mixture №5		2,89	95,13	99,82	1,93	168,44	99,89
mixture №6		3,58	78,25	98,91	2,83	105,35	99,05
mixture №7		19,25	13,96	93,44	17,52	21,14	95,51
mixture №8		9,58	25,65	96,82	8,14	39,59	97,08
Without inhibitor	70	264,34	-	-	238,27	-	-
mixture №1		44,02	8,84	80,36	43,89	9,04	80,41
mixture №2		44,28	19,05	80,48	43,85	18,59	80,54
mixture №3		45,01	20,01	80,09	49,59	21,08	79,42
mixture №4		39,97	17,45	83,49	40,79	17,69	83,09
mixture №5		3,51	79,01	98,43	2,72	105,35	99,35
mixture №6		2,84	95,38	99,81	1,93	169,58	99,18
mixture №7		19,52	12,89	93,82	17,58	10,93	95,28
mixture №8		44,08	18,06	80,09	48,81	19,08	79,24
Etalon-Nalco	60	38,93	6,99	85,71	30,27	8,05	87,58

The value of the corrosion potential in relation to the potential of a normal electrode in a background solution of 50 °C is -0.585 V (in an acidic environment), and in the H_2S environment it is -0.600 V. If mixture №7 is placed in an acidic environment, the corrosion potential reaches -0.505 V, for mixture №5 this indicator is -0.285 V, so there is a shift in the polarization curves. When the mixture №5 is introduced into the solution, the value of the electrode potential changes in the positive direction, which indicates a slowdown in the anode reaction. This condition indicates that a thin protective film is formed on the metal surface with fragments of inhibitors present in the composition. With a change in the potential, the value of the corrosion current simultaneously changes, this indicates the effect of a mixed mechanism on the process of metal corrosion of the inhibitor.

Conclusion. The catalytic reaction for the production of pyridine and pyridine bases using croton aldehyde (croton fraction) and ammonia as feedstock was studied. New poly-functional catalysts for the synthesis of pyridine and pyridine bases have been developed. It is shown that catalysts containing iron, zinc, aluminum, cadmium, and chromium oxides are poly-functional and provide 58.0 - 88.0 % pyridine selectivity, while the conversion of the Croton fraction and ammonia reaches 98 %. It was found that the use of extraction phosphoric acid as a peptizing component increases the mechanical strength of the catalyst by an order of magnitude.

The resulting mixtures of pyridine bases were tested as corrosion inhibitors. It was found that an increase in the amount of methyl-pyridines positively affects the protective properties of the inhibitor and at a concentration of 1.0% is more than 99.0 %.

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FORMALIZATION OF THE PROCESS OF SWITCHING PULSE ELEMENTS IN THE STUDY OF COMPLEX DISCRETE SYSTEMS

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Abstract:

Background. This article discusses the issues of a formalized description of the modes of operation of pulsed elements in the study of complex discrete systems that include several pulse elements with different characteristics and work asynchronously and out of phase, as well as being the basis of automated control systems for technological processes. Processes in one-dimensional and multidimensional systems containing several impulse elements operating in asynchronous and out-of-phase order are compared. There are providing basic concepts in the study of automation of control systems, such as a pulse element, parameters of real pulses, complex systems, discretization, the value of a pulse element.

The materials and methods deals with the formation of switching processes and the calculation of transient functions in one-dimensional and multidimensional pulse systems, revealing difficulties in various discrete systems are shown. Also in this part, the most general case of modulation of the first kind is considered, and the possibility of applying and extending this approach to modulations of other kinds is shown.

Results. In order to formalize various processes, a scheme is proposed, according to which a multidimensional system is considered, which contains impulse elements of various types, operating asynchronously and not in phase. An algorithm for the formation of the switching process of impulse elements in the study of complex discrete systems, based on the event-driven modeling method, is also proposed.

Conclusion. it was concluded that the proposed switching matrix is, as it were, a dispatcher of the computational process, monitoring the change in states in the system under study and initiating, if necessary, certain parts (modules) of the computational program (software package).