



ACTUAL PROBLEMS OF MODERN SCIENCE, EDUCATION AND TRAINING

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ACTUAL PROBLEMS OF NATURAL SCIENCES

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SYNTHESIS OF ACETYLENIC DIOLS BASED ON THE REACTION OF TERMINAL ACETYLENIC ALCOHOLS WITH ALIPHATIC KETONES

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Annotatsiya. Ushbu ishda ilk bor $\text{CaO}/\text{NH}_3/\text{Et}_2\text{O}$ katalitik sistema yordamida molekulasida alifatik, alitsiklik, aromatik va geterotsiklik o‘rinbosarlar saqlagan terminal atsetilen spirtlarining atseton, metilbutilketon va metiluchlamchibutylketonlar bilan nukleofil birikish reaksiyasi asosida yuqori biologik faollikka ega bo‘lgan atsetilen diollari sintezi o‘rganilgan. Atsetilen diollarining sintez qilish jarayoni va mahsulot unumiga turli omillar– harorat, reaksiya davomiyligi, katalizator va erituvchilar tabiati, reagent va substratlarning miqdorlari, reaksiyada hosil bo‘ladigan oraliq va qo‘shimcha mahsulotlar turlari va miqdorlari tizimli ravishda o‘rganilgan. Olingan natijalar asosida nukleofil birikish jarayonining eng muqobil sharoitlari aniqlangan hamda reaksiya mexanizmlari taklif etilgan. Sintez qilingan atsetilen diollari identifikatsiyalangan, ularning xususiy konstantalari, tuzilishi, tozaligi va tarkibi zamonaviy fizik-kimyoviy tadqiqot usullari yordamida isbotlangan.

Kalit soʻzlar: atsetilen diollari, alifatik ketonlar, nukleofil birikish, reaksiya mexanizmi, mahsulot unumi.

Аннотация. На основании влияния строения, природы и химической активности выбранных молекул ацетиленовых спиртов и кетонов на ход реакции и выход продуктов разработан относительный ряд продуктивности выходов ацетилен диолов. Систематически изучены различные факторы, влияющие на процесс синтеза и выход продуктов ацетилен диолов - температура, продолжительность реакции, природа катализаторов и растворителей, количества реагентов и субстратов, виды и количества образующихся в реакции промежуточных и побочных продуктов. На основании полученных результатов определены наиболее оптимальные условия процесса нуклеофильного сочетания и предложены механизмы реакции. Идентифицированы синтезированные ацетилендиолы, доказаны их собственные константы, структура, чистота и состав с помощью современных физико-химических методов исследования.

Ключевые слова: ацетилен диолы, алифатические кетоны, нуклеофильное присоединение, механизм реакции, продукт.

Abstract. In this work, the synthesis of highly biologically active acetylene diols was studied for the first time based on the reaction of the nucleophilic coupling of terminal acetylene alcohols with aliphatic, alicyclic, aromatic and heterocyclic substituents in their molecule with acetone, methyl butyl ketone and methyl tert-butyl ketones using the $\text{CaO}/\text{NH}_3/\text{Et}_2\text{O}$ catalytic system. Based on the influence of the structure, nature and chemical activity of selected molecules of acetylene alcohols and ketones on the course of the reaction and the yield of products, a relative series of productivity yields of acetylene diols has been developed. Various factors influencing the synthesis process and the yield of acetylene diol products have been systematically studied - temperature, reaction duration, nature of catalysts and solvents, amounts of reagents and substrates, types and amounts of intermediates and by-products formed in the reaction. Based on the results obtained, the most optimal conditions for the nucleophilic coupling process were determined and reaction mechanisms were proposed. The synthesized esters have been identified, and their partial constants, structure, purity and composition have been proven using modern physicochemical methods.

Keywords: acetylene diols, aliphatic, ketones, nucleophilic coupling, reaction mechanism, product yield.

Introduction

The presence of three bonds in the structure of acetylene alcohols, a hydroxyl group and mobile hydrogen, the ability of representatives of this class to enter into electrophilic and nucleophilic reactions in various processes increases their properties, they are considered an important reagent in organic synthesis. Today, acetylene alcohols are widely used as additional components in the production of vitamins, pigments, antibiotics and hormones.

Literature Review

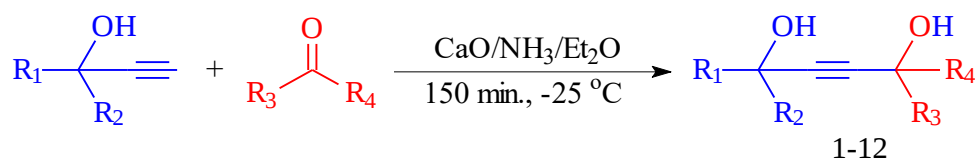
Acetylenic alcohols possess several reactive centers within their molecular structure, which makes them of great interest to organic chemists [1-5]. Specifically, the presence of the $-C\equiv CH$ group in their molecules enables them to undergo various nucleophilic, electrophilic, radical, and cyclization reactions with different reagents, allowing for the formation of diverse organic compounds [6-7]. Nowadays, extensive research is being conducted on acetylenic alcohols and their derivatives in the context of their biological activity, their occurrence in natural products, their pharmacological significance, and their applicability in other scientific fields.

The development of new technologies for the synthesis of acetylenic alcohols and their derivatives is actively pursued in modern progressive countries. These studies involve systematic investigations aimed at understanding the biological activities of acetylenic alcohols, including their occurrence in aliphatic, aromatic, cyclic, and heterocyclic scaffolds, as well as the development of synthetic methodologies for their production [8-11]. R.E.Minto and his scientific team have discovered the presence of acetylenic diols as natural products in organisms such as plants, marine organisms and certain species of fungi [12]. For instance, acetylenic diols obtained from the mushroom *Clitocybe catinus* using a reagent derived from potassium carbonate and methanol exhibited significant biological activity against sarcoma [13]. The Favory reaction has been employed to synthesize second- and third-generation acetylenic diols by nucleophilic addition of acetylene to the carbonyl group, followed by oxidative coupling [14]. G.Wang and J.Dou successfully synthesized 2,4,7,9-tetramethyldecyne-5-diol-4,7, which possesses a complete dynamic surfactant behavior and can be utilized in the field of proton-absorbing black and white developer production [15]. Brazilian researchers investigated the synthesis of acetylenic diols using propargyl alcohols and aldehydes derived from aliphatic, cyclic, aromatic, and heterocyclic substrates with the assistance of $CeCl_3/nBuLi$. The process was carried out in tetrahydrofuran at $-40\text{ }^{\circ}\text{C}$ for 4 hours, resulting in a maximum product yield [16].

Research Methodology

In this study, as the research object in the form of a selected molecule in the $CaO/NH_3/Et_2O$ complex catalytic system, terminal acetylenic alcohols - 1-ethynylcyclopentanol, 3-methylheptyn-1-ol-3, 2-phenylbutyn-3-ol-2 and 2-(pyridin-4-yl)-butyn-3-ol-2 with aliphatic, alicyclic, aromatic, and heterocyclic substituents were investigated. Based on their reactions with acetone, methyl butyl ketone, and methyl ethyl ketone, the following acetylene diols were synthesized: 1-(3-hydroxy-3-methylbutyn-1-yl)cyclopentanol (1), 1-(3-hydroxy-3-methylheptyn-1-yl)cyclopentanol (2), 1-(3-hydroxy-3,4,4-trimethylpentyn-1-yl)cyclopentanol (3), 2,5-dimethylnonyne-3-diol-2,5 (4), 5,8-dimethyldodecin-6-diol-5,8 (5), 2,2,3,6-tetramethyldecin-4-diol-3,6 (6), 2-methyl-5-phenylhexyn-3-diol-2,5 (7), 5-methyl-2-phenylnonyn-3-diol-2,5 (8), 5,6,6-trimethyl-2-phenylheptyn-diol-2,5 (9), 2-methyl-5-(pyridin-4-yl)hexyn-3-diol-2,5 (10), 5-methyl-2-(pyridin-4-yl)nonyn-3-diol-2,5 (11), and 5,6,6-trimethyl-2-(pyridin-4-yl)heptyn-3-diol-2,5 (12). The general scheme and

mechanism of the reaction were proposed based on literature sources and research results [17-18].



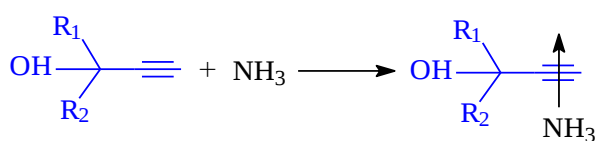
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| 1. $\text{R}_1\text{R}_2 = \text{cPt}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{Me}$ | 7. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Ph}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{Me}$ |
| 2. $\text{R}_1\text{R}_2 = \text{cPt}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{Bu}$ | 8. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Ph}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{Bu}$ |
| 3. $\text{R}_1\text{R}_2 = \text{cPt}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{'Bu}$ | 9. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Ph}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{'Bu}$ |
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| 5. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Bu}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{Bu}$ | 11. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Py}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{Bu}$ |
| 6. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Bu}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{'Bu}$ | 12. $\text{R}_1 = \text{Me}$, $\text{R}_2 = \text{Py}$, $\text{R}_3 = \text{Me}$, $\text{R}_4 = \text{'Bu}$ |

Analysis and Results

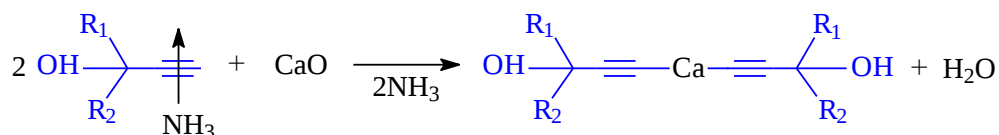
It is known that ketones, as one of the oxo compounds, possess unique reactivity. This is attributed to the distribution of electron density within the ketone molecule and the difference in relative electronegativity between carbon and oxygen atoms. The electron density of the carbonyl group in the ketone molecule is drawn towards the oxygen atom with higher electronegativity, resulting in a partial negative charge (δ^-) on the oxygen atom and a partial positive charge (δ^+) on the carbon atom, exhibiting electrophilic characteristics. This leads to the polarization of the C-C bonds adjacent to the carbonyl group in the ketone molecule, and ketones can undergo nucleophilic attacks by other molecules. Additionally, the stability of the carbon-oxygen bond in ketones is higher compared to the C=C (double bond), meaning that the energy of the C-O bond is higher than that of the two carbon-carbon bonds, in contrast, the energy of the C=C double bond is lower than the energy of two carbon-carbon bonds. The mentioned conditions facilitate the formation of complex molecules or ions with a carbonyl group, and these conditions make it easier for them to participate in various reactions.

Terminal acetylenic alcohols, in the context of the movement of hydrogen in the sp-s bond, combine with metals to form strong nucleophilic reagents, resulting in the formation of acetylene diols in a suitable manner as a result of the attack of the carbonyl group on the carbon atom in the selected ketone molecule.

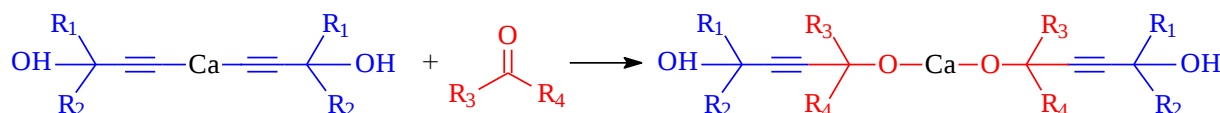
Initially, ammonia interacts with the triple bond in the acetylenic alcohol molecule, increasing the mobility of the hydrogen atom in the triple bond, and as a result, the acetylenic alcohol undergoes deprotonation due to the easy replacement of the hydrogen position with a metal atom, creating favorable conditions for their combination.



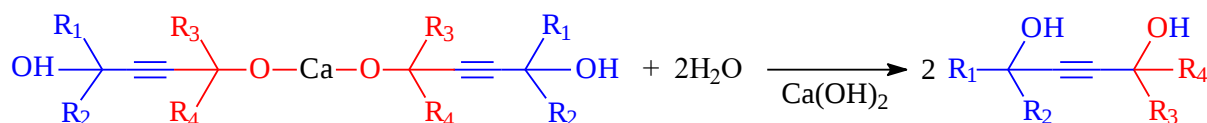
In the subsequent stage of the reaction, under the catalytic influence of the intermediate complex CaO, a strong nucleophilic reagent - acetylide is formed [21]. In this process, with the assistance of unpaired electrons of the undissociated nitrogen atom in the ammonia molecule, a proton from the catalyst, which has moved away from the solvent due to the interaction with oxygen, reacts with water molecules in the system, resulting in the formation of a hydroxide ion.



In the subsequent stage of the reaction, the calcium salt of acetylenic alcohol, which has a very high catalytic activity when reacting with 4-methylpentanone-2, is formed, resulting in the production of calcium alkoxylate of acetylene diol, which readily undergoes hydrolysis.



The resulting acetylene diol alkoxylate undergoes hydrolysis in an ammonia solution, producing acetylene diol and calcium hydroxide. [22-23].



In the catalytic system of CaO/NH₃/Et₂O, the reactivity and phase structure of terminal acetylenic alcohols and the nature of substituents on the ketone molecules were investigated as a result of their influence on the reaction activity and phase arrangement for the purpose of obtaining the desired products. The conditions of chemical processes were systematically analyzed, including the synthesis of acetylene diols, such as temperature, reaction continuity, the nature and quantity of the solvent and catalyst, as well as the initial molar quantities of the starting materials.

As a preliminary investigation, the effect of the amount of CaO, which was chosen as the catalyst, on the selectivity of the nucleophilic addition reaction between acetylenic alcohols and ketones was studied (Figure 1). The molar quantities of CaO were varied from 0,01 to 0,1 in this case.

As observed from the figure, when CaO was taken in the amount of 0,025 moles, it exhibited maximum catalytic activity. Consequently, a selective environment was created for the deprotonation of terminal acetylenic alcohols and their subsequent addition reaction with the oxygen of the carbonyl group in hydrogen ketone molecules. By mutually influencing the reagent and substrate, a new sp-sp³ bond was formed, leading to the synthesis of acetylene diols with maximum yield (1- 78,2%, 2- 75,4%, 3- 71,3%, 4- 76,3%, 5- 68,8%, 6- 62,0%, 7- 85,3%, 8- 82,0%, 9- 79,8%, 10- 70,1%, 11- 63,4%, 12- 58,2%).

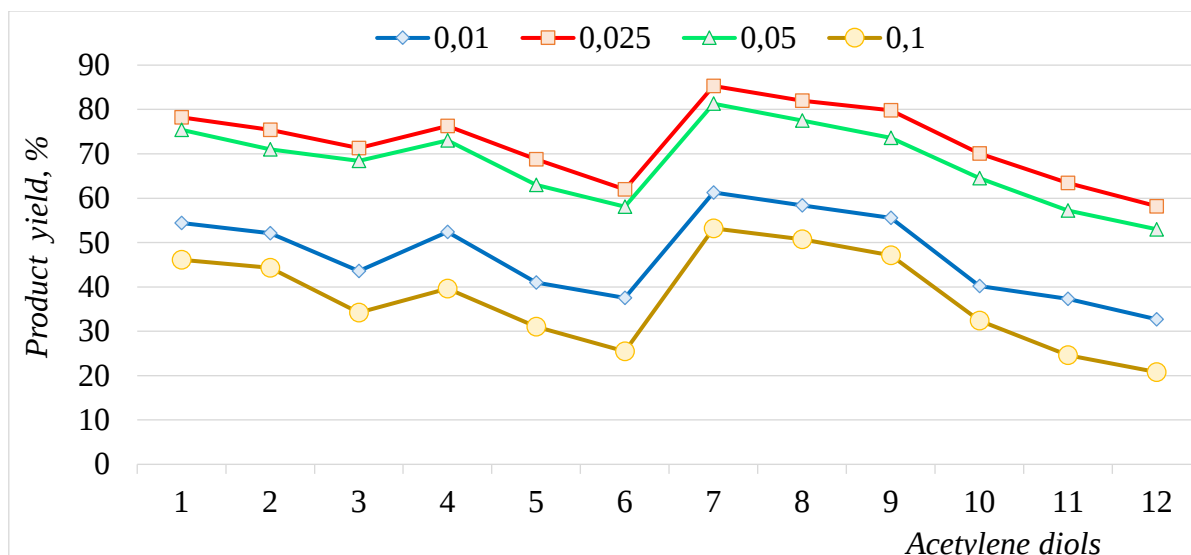


Figure 1. The effect of the amount of CaO on the yield of acetylene diols (temperature: -25 °C, solvent: diethyl ether, reaction duration: 150 minutes).

However, when the catalyst amount was increased to 0,05 or 0,1 moles, additional reactions occurred during the process. These included the production of calcium alkoxylates by the interaction of the synthesized acetylene diols with other catalysts, as well as the occurrence of vinyl oxy ethers, short-term dehydration, internal molecular cyclization, transformation, dimerization, condensation, and enolization reactions in the presence of phenylacetylene in the system. These additional and intermediate compounds resulted in a significant decrease in the yield of acetylene diols due to their consumption.

In order to investigate the reaction continuity and determine the corresponding conditions for the process, the reactions were carried out in intervals ranging from 90 minutes to 180 minutes. The results are presented in Table 1.

Table 1. Effect of reaction duration on the yield of acetylene diols (temperature - 25 °C, solvent: diethyl ether, CaO amount: 0,025 moles).

Acetylene diols	Product yield, %			
	90 minutes	120 minutes	150 minutes	180 minutes
1	43,5	62,3	78,2	67,1
2	39,2	58,5	75,4	64,2
3	36,0	55,2	71,3	60,8
4	42,3	59,7	76,3	65,0
5	34,1	52,5	68,8	57,5
6	28,4	46,1	62,0	51,4
7	51,3	69,3	85,3	74,2
8	48,1	66,1	82,0	74,1
9	45,0	63,4	79,8	69,5
10	35,9	54,6	70,1	59,4
11	29,7	47,0	63,4	52,2
12	23,0	41,5	58,2	46,9

When the nucleophilic addition reaction was carried out for 90 and 120 minutes, it was determined that the catalyst did not exhibit complete catalytic activity, and the formation of intermediate calcium acetylide was insufficient. As a result, the initial reagents did not fully engage in the desired reaction and underwent partial condensation, as well as partial enolization of the ketones, leading to the formation of intermediate and additional compounds. When the reaction was conducted for a duration of 150 minutes, the selectivity of the catalyst was fully manifested. The formation of an active complex in the system, along with a decrease in the activation energy and an increase in the reaction rate, resulted in the highest yield of the desired product. However, when the reaction duration was extended to 180 minutes, the mutual interaction or influence of the initial and resulting substances in the reactor, the additional effect of the catalyst, various coordinating complex components on the coordination sphere, and the twisting of sterically hindering groups for the formation of C-C bond in the reagent and substrate, resulting in the formation of additional products due to the decrease in the quantity of the desired product were observed in the reaction mixture.

The above experiment was conducted in the temperature range of 25 °C to -40 °C for the purpose of synthesizing acetylene diols, and the results of the specific analysis were evaluated (Figure 2).

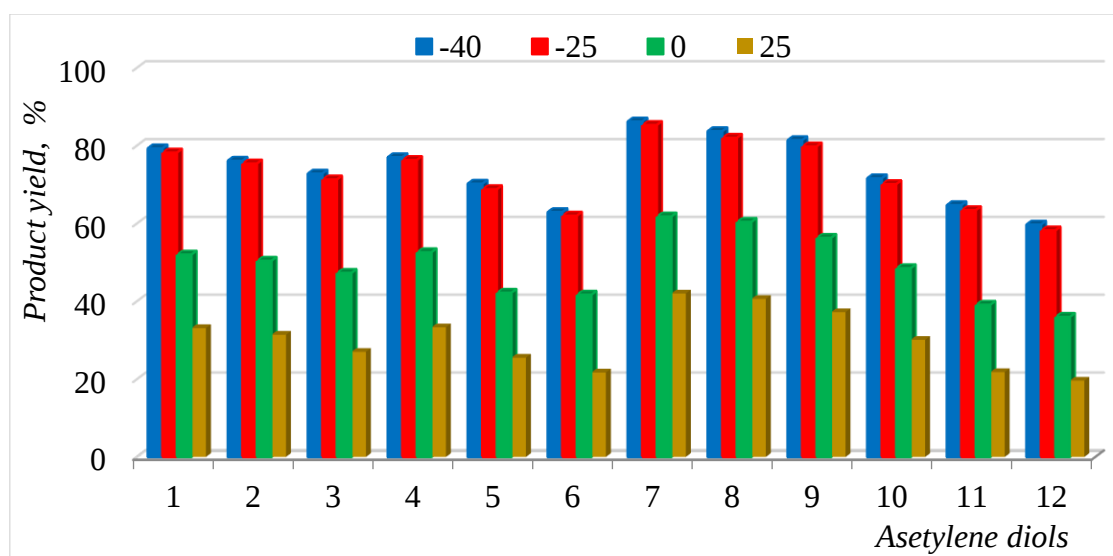


Figure 2. Effect of temperature on the yield of acetylene diols (reaction duration: 150 minutes, solvent: diethyl ether)

Based on the comparative analysis, it was determined that acetylene diols exhibited maximum yield at -25 °C temperature. In this process, the energy of colliding particles was equivalent to the activation energy, leading to the formation of new sp^3-sp^3 bonds in acetylene alcohols and ketone molecules. In other words, at -25 °C temperature, due to the substantial energy possessed by the colliding molecules and ions, the nucleophilic reagents (calcium acetylide) resulted in the efficient production of the desired product yield.

However, an increase in temperature resulted in a sharp decrease in the yield of the product. In this case, it was observed that the decrease in temperature affected both the

product yield and the direction of the reaction. Specifically, the polymerization of acetylene diols, the formation of resinous compounds, as well as the production of vinyl ethers from terminal acetylene alcohols that did not participate in the reaction, led to a decrease in product yield (1- from 78,2 to 52,0%; 2- from 75,4 to 50,4%; 3- from 71,3 to 47,3%; 4- from 76,3 to 52,6%; 5- from 68,8 to 42,2%; 6- from 62,0 to 41,7%; 7- from 85,3 to 61,8%; 8- from 82,0 to 60,4%; 9- from 79,8 to 56,3%; 10- from 70,1 to 48,5%; 11- from 63,4 to 39,1%; 12- from 58,2 to 36,0%).

Based on the obtained results, the most favorable conditions for the synthesis of acetylene diols were determined using the $\text{CaO}/\text{NH}_3/\text{Et}_2\text{O}$ complex catalytic system. According to the findings, the optimal conditions were as follows: temperature of -25°C , reaction duration of 150 minutes, initial total amount of CaO relative to the mass of starting materials at 0,025 moles, and when the starting materials were taken in an equimolar ratio, acetylene diols were synthesized with the highest yield.

The purity, composition, structure, and specific characteristics of the synthesized acetylene diols were analyzed using modern analytical techniques such as IR spectroscopy, ^1H -NMR, ^{13}C -NMR spectroscopy, mass spectrometry, chromatography (GC, HPLC), quantum chemical calculations, biological, and other physicochemical research methods. Specific constants were determined, and energetic and quantum chemical properties were evaluated. The charges of atoms in the molecule, electron densities, and optical properties were investigated using specialized software tools (Table 2).

Table 2. Acetylene diols quantum chemical results.

Acetylene diols	Formation enthalpy, kcal/mol	Van der Waals energy, kcal/mol	Coulomb energy, kcal/mol	Torsional energy, kcal/mol	Valence bond energy, kcal/mol	Bond energy, kcal/mol
1	16,0326	4,0885	0,5031	7,6384	0,5627	3,2398
2	23,6094	5,2953	2,9360	10,1677	0,9742	4,2362
3	21,2484	5,8970	0,4581	8,7364	1,5790	4,5779
4	12,7729	4,2460	1,2041	3,8602	0,8872	2,5753
5	11,0322	4,5630	0,4471	1,9544	1,2021	2,8657
6	14,2038	5,6473	1,0300	2,9177	1,8276	2,7812
7	2,5550	2,4704	2,0684	-4,6475	0,6146	2,0492
8	5,9941	3,9447	2,1165	-3,8599	0,9920	2,8009
9	5,6177	4,1200	0,9851	-4,2627	1,6282	3,1475
10	9,8741	5,2352	1,0801	1,1113	0,6728	1,7746
11	11,6829	6,4232	0,7694	1,0864	0,9928	2,3946
12	10,2917	6,8311	0,8813	2,1034	1,6896	2,9275

Based on the analysis results, the nature of radicals in terminal acetylenic alcohols and ketones, as well as their phase-specific reactivity, was determined in terms of their nucleophilic aggregation process (reactivity). According to this, the order of product yield was established as follows: $12 < 6 < 11 < 5 < 10 < 3 < 2 < 4 < 1 < 9 < 8 < 7$.

Conclusions

For the first time, a method for synthesizing aliphatic acetylenic diols was established based on the reaction of acetylenic alcohols with ketones using the $\text{CaO}/\text{NH}_3/\text{Et}_2\text{O}$ complex catalytic system, and an experimental setup was developed.

The synthesis of acetylenic diols, including the reaction method, mechanism, reaction conditions, and kinetics, was investigated. The specific characteristics of the synthesized acetylenic diols, including purity, composition, and structure, were determined using modern physicochemical research methods.

The influence of the catalytic system used in the synthesis of acetylenic diols on the product yield and the progress of the reaction was studied, focusing on the selectivity, reactivity, and stability of calcium oxide and ammonia.

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STUDY OF THE STRUCTURE OF THE NITRIDED LAYER FOR THE STRENGTH OF THE ADHESIVE BOND OF WEAR-RESISTANT COATING IN R9M4 STEEL

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Annatatsiya: Tadqiqot TiAlN qoplamani mustahkam adgezion aloqasi bilan tezkesar po‘lat asosida azotlashtirilgan qatlamini baholash ta'siriga yo'naltirilgan. Ikki bosqichli vakuum kanalida ionli azotlash va gaz plazmasi usulida azot tarkibini va gaz aralashmasini argon bilan o'zgartirib, strukturaviy qatlamni boshqarishga imkon beradi. Tadqiqotlar shuni ko'rsatdiki, azotlangan qatlamning tuzilishi qoplamaning mustahkamligi kuchiga sezilarli ta'sir qiladi. Past azot