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## SYNTHESIS OF ESTERS OF HALOGENOACETIC ACIDS

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## SYNTHESIS OF ESTERS OF HALOGENOACETIC ACIDS

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Amidoalkylating reagents containing a phthalimide group are used in the synthesis of hard-to-reach primary amines and complex heterocyclic compounds. These types of amidoalkylating compounds are suitable reagents for nucleophilic substituted reactions in acidic media due to their resistance to acids. Result of reactions of amidoalkylating reagents-N-hydroxyethylphthalimide and N-hydroxymethylphthalimide with aliphatic carbonic acids can also produce new bactericidal and fungicidal esters. In this study, halogen acids reacted with N-hydroxymethylphthalimide monochloric acetic acid, monobromic acetic acid, monoiodic acetic acid, trifluoric acetic acid and trichloroacetic acid, as well as, N-b-hydroxyethylphthalimide monohydroxy acid. As a result of the reactions, new phthalimidomethyl and phthalimidoethyl esters of haloacetic acids were synthesized. The physical properties of crystalline esters have been determined. The structure of these compounds were confirmed by the analysis of their IR and PMR spectra. The esterification reactions of halogenated acetic acids were carried out in a benzene solvent and a sulfuric acid catalyst with primary alcohols, N-hydroxymethylphthalimide and N-β-hydroxyethylphthalimide. It was found that in a benzene solvent, which increases the rate of bimolecular nucleophilic exchange in primary alcohols, phthalimidoethyl esters of halogen acid are formed in high yields, and phthalimidomethyl esters are formed in low yields. It was shown that the reaction of esterification of halogen acids with N-hydroxymethylphthalimide proceeds with monomolecular nucleophilic exchange.

**Keywords:** N-β-hydroxyethylphthalimide, N-hydroxymethylphthalimide, haloacetic acid, halogen acetic acid ester, amidoalkylation

## СИНТЕЗ ЭФИРОВ ГАЛОГЕНОУКСУСНЫХ КИСЛОТ

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Амидоалкилирующие реагенты, содержащие фталимидную группу, используются в синтезе труднодоступных первичных аминов и сложных гетероциклических соединений. Эти типы амидоалкилирующих соединений являются подходящими реагентами для реакций нуклеофильного замещения в кислой среде из-за их устойчивости к кислотам. А также, в результате реакций амидоалкилирующих реагентов - N-β-гидроксиэтилфталимида и N-гидроксиметилфталимида алифатическими карбоновыми кислотами можно получить сложные эфиры с новыми бактерицидными и фунгицидными свойствами. В этом исследовании галогеновые кислоты реагировали с N-гидроксиэтилфталимидом монохлорной уксусной кислоты, монобромистоводородной уксусной кислотой, моноиодной уксусной кислотой, трифтористоводородной уксусной кислотой и трихлоруксусной кислотой, а также с N-β-гидроксиэтилфталемидом моноиодной уксусной кислоты и трифтористоводородной кислотой. В результате реакций синтезированы новые фталимидометилловые и фталимидоэтиловые эфиры галогенуксусных кислот. Определены физические свойства кристаллических сложных эфиров. Строение этих соединений подтверждено анализом их ИК- и ПМР-спектров. Реакции этерификации галогенированных уксусных кислот первичными спиртами - N-гидроксиэтилфталимидом, N-β-гидроксиэтилфталемидом проводили в бензольном растворителе и сернокислотом катализаторе. Обнаружено, что в бензольном растворителе, увеличивающем скорость бимолекулярного нуклеофильного обмена в первичных спиртах, с высокими выходами образуются фталимидо-этиловые эфиры галогеновой кислоты, а с низкими выходами - фталимидометилловые эфиры. Было показано, что реакция этерификации галогеновых кислот с N-гидроксиэтилфталемидом протекает с мономолекулярным нуклеофильным обменом.

**Ключевые слова:** N-β-гидроксиэтилфталемид, N-гидроксиэтилфталемид, галогенуксусная кислота, сложный эфир галогенуксусной кислоты, амидоалкилирование

## GALOGENSIRKA KISLOTALARNING MURAKKAB EFIRLARI SINTEZI

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Olinishi qiyin bo'lgan birlamchi aminlar va murakkab tuzilishli geterohalqali birikmalar sintezida fталimid guruhini tutgan amidoalkilovchi reagentlardan foydalaniladi. Bu turdagi amidoalkilovchi birikmalar kislotalar ta'siriga chidamliligi sababli kislotali muxida olib boriladigan nukleofil almashinish reaksiyalari uchun qulay reagent hisoblanadi. Shuningdek, amidoalkilovchi reagentlar-N-β-gidroksietilftalimid va N-gidroksimetilftalimidlarning alifatik karbon kislotalari bilan reaksiyalari natijasida yangi bakteritsid va fungitsid xossali murakkab efirlarni olish mumkin. Ushbu ishda galogensirka kislotalari-monoxlor sirka kislotasi, monobrom sirka kislotasi, monoyod sirka kislotasi va trixlor sirka kislotalarining N-gidroksietilftalimid bilan reaksiyalari olib borildi. Reaksiyalar natijasida galogen sirka kislotalarining yangi fталimidometil va fталimidoetil efirlari sintez qilib olindi. Kristall xolidagi murakkab efirlarning fizik ko'rsatkichlari aniqlandi. Bu birikmalarining tuzilishi ularning IQ- va PMR-spektorlari taxlil orqali isbotlandi. Galogen sirka kislotalarining birlamchi spirtlari bo'lgan N-gidroksietilftalimid, N-β-gidroksietilftalimid bilan boradigan eterifikatsiya reaksiyalari benzol erituvchiligida va sulfat kislotasi katalizatorligida olib borildi. Birlamchi spirtlarda boradigan bimo- lekulyar nukleofil almashinish tezligini oshirib beruvchi benzol erituvchisida galogensirka kislotasining fталimidoetil efirlari yuqori, fталimidoetil efirlari esa past ummlar bilan hosil bo'lishi aniqlandi. Galogensirka kislotalarining N-gidroksietilftalimid bilan boradigan eteri- fikatsiya reaksiyasi monomolekulyar nukleofil almashinish bilan borishi ko'rsatib berildi.

**Kalit so'zlar:** N-β-gidroksietilftalimid, N-gidroksimetilftalimid, galogen sirka kislotasi, galogen sirka kislotasi murakkab efiri, amidoalkillash

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### Introduction

Phthalic anhydride and its imide derivatives are of great importance in the chemical and pharmaceutical industries [1-4]. Including, N-b-hydroxyethylphthalimide and N-hydroxymethylphthalimides, are important intermediates in various sectors of the economy, such as photocopying, agrochemistry, varnishes, pigments and rubber chemical industry, medicine [5, 6, 7].

In an addition to, compounds containing the phthalimide group are used as alkylating agents in organic synthesis [8].

By the amidoalkylation reaction of acids with hydroxyalkylimides, it is possible to obtain esters with different biological activity, as well as to synthesize heterocyclic compounds with complex structure and primary amines, which are difficult to synthesis [9].

Therefore, chemists are particularly interest-

ed in the synthesis of esters of halogenated acetic acids and the study of their properties.

Dey Sanjeyev K., Lightner David A., 2,5-dimethoxycetrahydrofuran was boiled in a dioxane solution and treated it with a solution of phthalimide in ethyl alcohol, resulting in 1,1-bipyrrrol.

The X-ray structure of the obtained substance was studied [10].

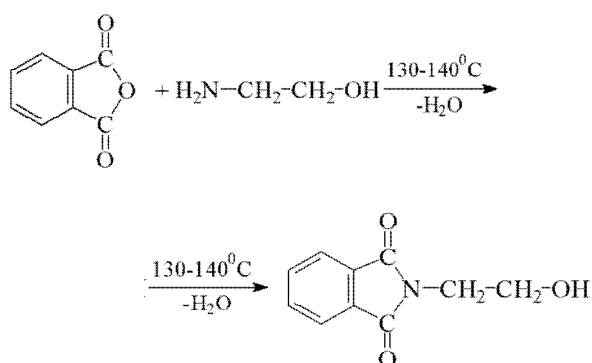
The scientists studied the bromination reactions of cis-4-cyclohexene-1,2-dicarboxylic acid N-(o-aminophenyl) and phthalic acid N-(o-aminophenyl) imide. 40% bromic acid solution and 28% hydrogen peroxide were used as oxidizers, the reaction was carried out at 0–50 °C [11].

We also carried out esterification reactions with mono chlorine, mono bromine, mono iodine acid, trifluoro-, trichloro N-(β-hydroxyethyl) phthalimide, N-methylolftalimide.

### Research methods

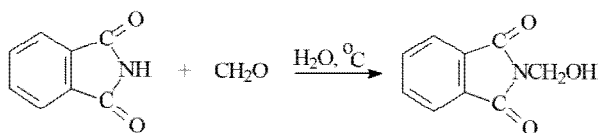
In the synthesis of esters, amidoalkylation reactions of halogen acetic acids with primary alcohols containing the phthalimide group were used. The reactions and the purity of the product were checked on thin-layer chromatography. The structure of the obtained new substances were determined using PMR-spectroscopy, IR-, chromatomass spectrometry.

For experiments, primary alcohols were synthesized by certain methods in the literature [12]. The yield of N-(β-Hydroxyethyl) phthalimide was 91%:



Melting point of N-(β-hydroxyethyl) phthalimide is  $T_{\text{melting}}=126-128\text{ }^{\circ}\text{C}$ , purity TLC (thin layer chromatography)  $R_f=0.33$  (silufol system, benzene:acetone=3:1)

The reaction equation for the production of N-methylolftalimide is as follows:



The obtained N-methylolftalimide was recrystallized in ethyl alcohol, yield was 96%.

Melting point of N-Methylolftalimide is  $T_{\text{melt}}$ .

$\text{ing}=136-138\text{ }^{\circ}\text{C}$ , purity TLC  $R_f=0.52$  (silufol, system, benzene:acetone=3:1).

We performed all the reactions in a round-bottomed tube equipped with a reverse cooler mounted on a tripod, a water collector, and a mechanical stirrer. Phthalimide N-(β-hydroxyethyl) was added to the flask (0.02mol), benzene (15ml) and catalyst sulfuric acid (2 drops) were added as a solvent for halogenic acid (0.022mol) (the amount of catalyst was reduced when trichloro- and trifluoroacetic acid were obtained). The reaction was carried out by heating at a magnetic stirring for 4–7 h (until the calculated amount of water was separated). After the water separation was complete, the reaction mixture was cooled and 20 ml of water was poured over it. The precipitate from the mixture was filtered and poured. The substance was recrystallized in acetone.

The reaction products were identified by method IR (Perkin Elmer Spektrum IR, Version 10.6.1) and NMR  $\text{H}^1$  and  $\text{C}^{13}$  (Unity 400 plus ICPSASR Uz,  $\text{Si}(\text{CH}_3)_4$  standard) Solvent  $\text{C}_6\text{D}_6$ .

The obtained product is a white crystalline product of *phthalimido ethyl ester of monoiodic acetic acid*, melting temperature  $T_{\text{melting}} = 90-91\text{ }^{\circ}\text{C}$ ,  $R_f = 0.69$  in TLC (silufol, system benzene:acetone=3:1).

Specific absorption fields of phthalimido ethyl ester of monoiodic acetic acid on IR spectrometer ( $\text{cm}^{-1}$ ): Deformation vibrations of CH bond in 1,2-disubstituted aromatic ring at 720, 790; 1425 valence vibrations of C=C bonds and 2956 C-H bonds in the aromatic ring, 1701, asymmetric vibrations of the CH group at 2956, symmetric valence vibrations at 2956; valence vibrations of the nitrogen-bound  $\text{CH}_2$  group in 1365; 1790, 1796 - Valence vibrations of the  $\text{CH}_2\text{-COOR}$  complex ester bond and 1134 - C-O-bond valence vibrations were observed.

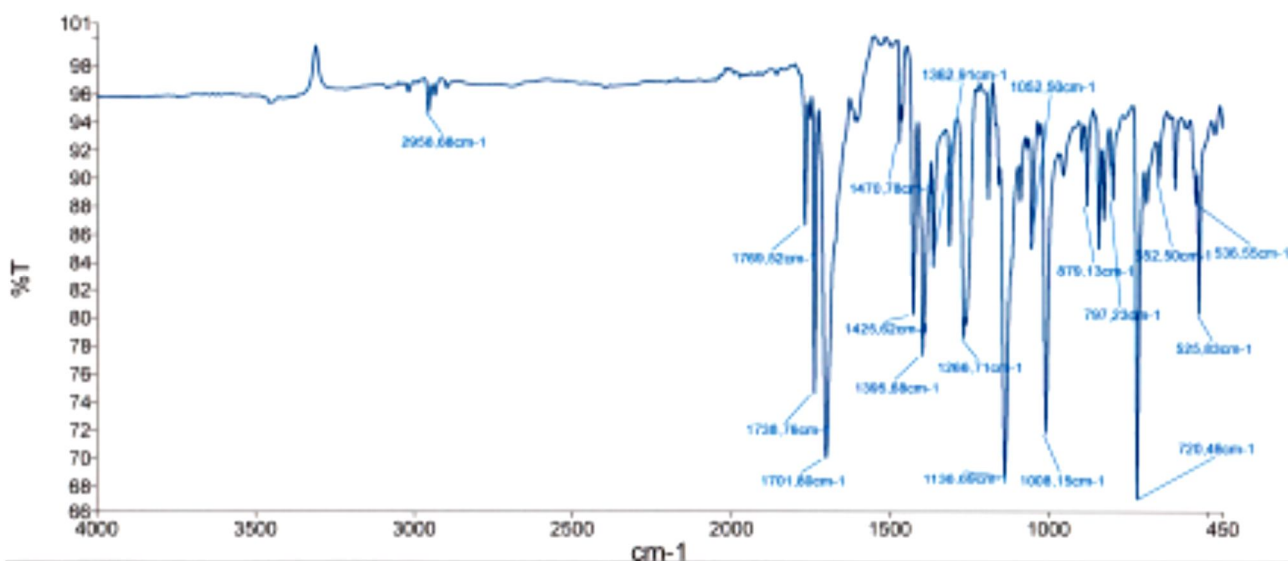


Figure 1. IR specter of phthalimido ethyl ester of monoiodic acetic acid.

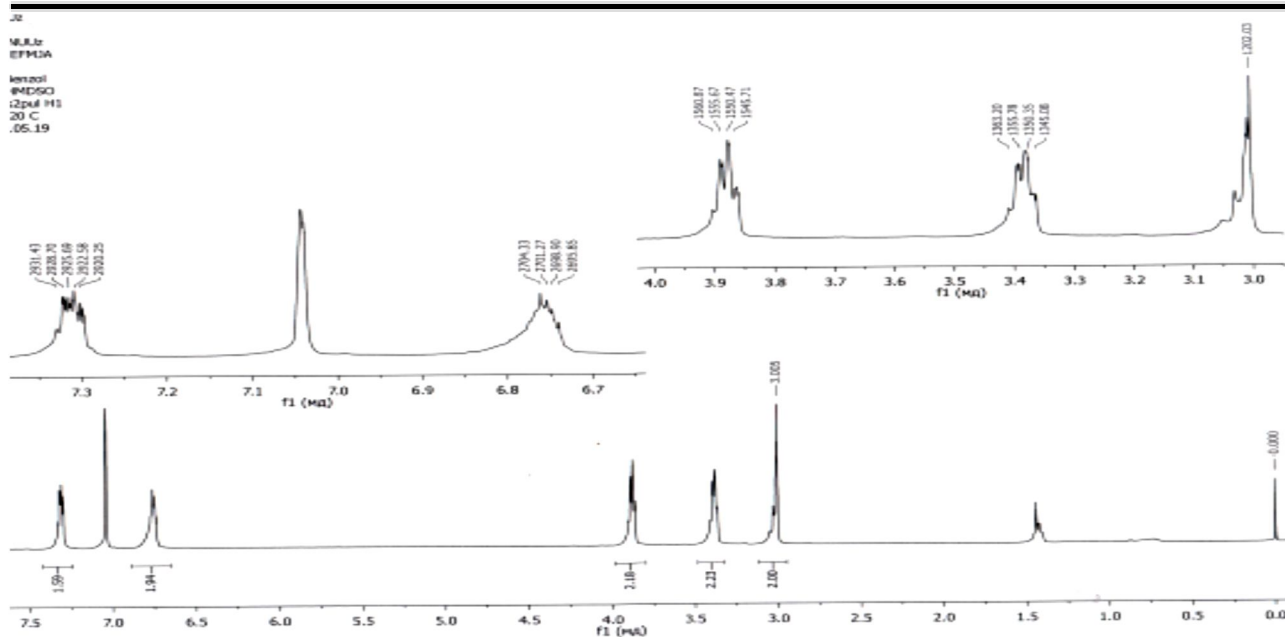


Figure 2. PMR spectrum of phthalimido ethyl ester of monoiodic acetic acid.

Analysis of the PMR spectrum: 400 MHz,  $C_6D_6$ , chemical shift of protons ( $\delta$ , ppm). triplet signal of two hydrogen protons in oxygen-bound methylene group of phthalimidoethyl ester of monohydric acid is 3.4 ppm (2H,  $-O-CH_2-CH_2$ ), triplet signal of protons of nitrogen-bound methylene group is 3.9 ppm in (2H,  $N-CH_2-CH_2-$ ), the singlet signal of the protons of the methylene group bound to iodine is 3.01 (2H,  $I-CH_2-CO-$ ), multiplet protons of the aromatic ring are 6,7 and 7,3. Ppm (4H, ArH) were observed in weak areas.

*Phthalimido ethyl ester of trifloric acetic acid* is a white crystalline substance with a yield of 83%, melting temperature  $T_{melting} = 102-103^\circ C$ . TLC  $R_f = 0,59$  in (silufol, system benzene:acetone = 3:1).

Specific absorption fields in the IR spectrum of Phthalimido ethyl ester of trifloric acetic acid ( $cm^{-1}$ ): Deformation vibrations of the CH bond in the 1,2-dialmashed aromatic ring at 714, 795; 1732 Valence vibrations of C=C bonds and 2950 C-H bonds in the aromatic ring, 1773,  $-CO-NH-$  amide; the asymmetric vibrations of the oxygen-bound  $CH_2$  group at 2950 and the valence vibrations of the nitrogen-bound  $CH_2$  group at 1342; 1773  $-CH_2-COOR$  complex valence bond and 1067,  $-C-O-$  bond valence vibrations were observed.

Analysis of the PMR spectrum: 400 MHz,  $C_6D_6$ , chemical shift of protons ( $\delta$ , ppm) the triplet signal of two hydrogen protons in the oxygen-bound methylene groups of trifluoric acid phthalimidoethyle ester at 6.85 ppm (2H,  $-O-CH_2-CH_2$ ), the triplet signal of the nitrogen-bound methylene group protons at 6.95 ppm (2H,  $-N-CH_2$ ), multiplet signal of the aromatic ring hydrogen protons in 7.25 and 7.35 ppm (4H, ArH) were observed.

*Phthalimido methyl ester of Monobromic acetic acid* is a white crystalline substance, product yield 54%, melting temperature  $T_{melting} = 78-80^\circ C$ , TLC  $R_f = 0.75$  in (silufol, system benzene:acetone = 3:1).

Specific absorption regions in the IR spectrum of phthalimido methyl ester of monobromic acetic acid ( $cm^{-1}$ ): Deformation vibrations of the CH bond in the 1,2-disubstituted aromatic ring at 721, 798; 1496 Valence vibrations of C=C bonds in the aromatic ring and 3024, 3061 C-H bonds, 1707, 3454  $-CO-NH-$  amide;  $CH_2$  group asymmetric at 2962, symmetric valence vibrations at 2939; valence vibrations of the nitrogen-bound  $CH_2$  group were observed in 1396; 1749, 1707, 1771 -Valence vibrations of the  $-CH_2-COOR$  ester bond and 1169 C-O-bond were observed.

Analysis of the PMR spectrum: 400 MHz,  $C_6D_6$ , chemical shift of protons ( $\delta$ , ppm), the singlet signal of nitrogen-bound methylene group protons at 4.47 ppm (2H,  $N-CH_2$ ), the bromine-bound methylene group proton singlet signal at 2.9 ppm (2H,  $Br-CH_2$ ), multiplet signal of aromatic ring hydrogen protons in 6.7 and 7.25 ppm (4H, ArH) were observed.

*Phthalimido methyl ester of monochloric acetic acid* is a white crystalline substance, yield 59%, melting temperature  $T_{liquid} = 87^\circ C$ ,  $R_f = 0.81$  in TLC (silufol, system benzene:acetone = 3:1)

Specific absorption regions in the IR spectrum of phthalimido methyl ester of monochloro acetic acid ( $cm^{-1}$ ): Deformation vibrations of the CH bond in the 1,2-disubstituted aromatic ring at 711, 726; 1383 C=C bonds in the aromatic ring and 1717, 3488  $-CO-NH-$  amide; valence vibrations of the nitrogen-bound  $CH_2$  group at 1405; In 1717, 1768, 1782 the valence vibrations of the  $-CH_2-COOR$  complex ester bond and in 1138 the



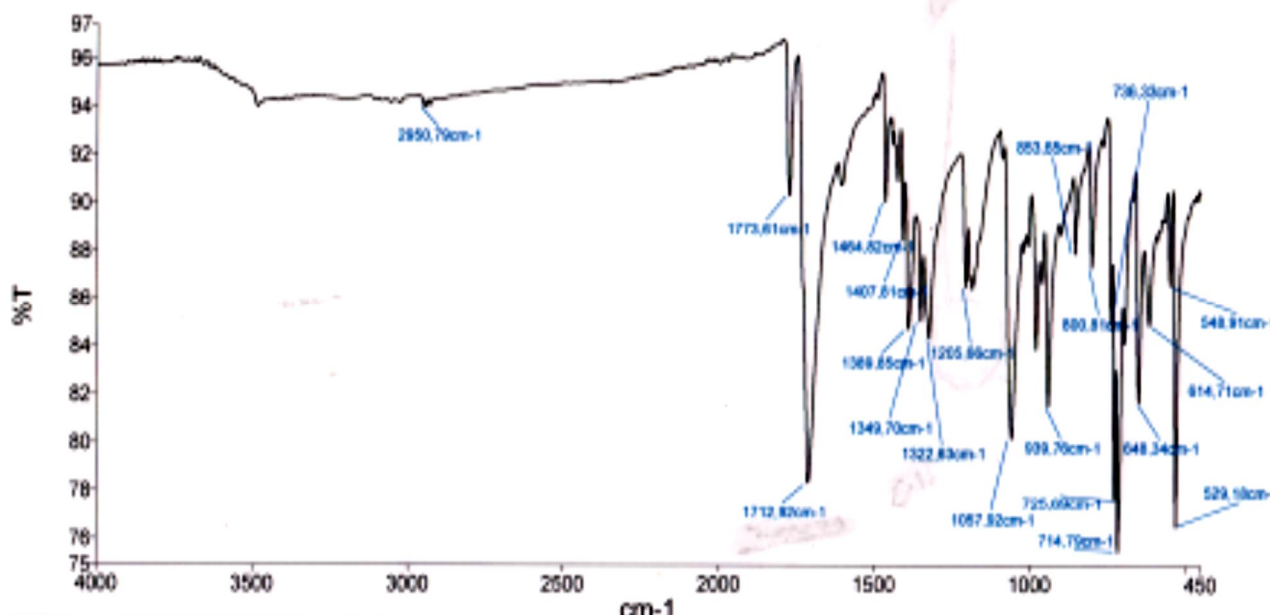


Figure 3. IR spectrum of Phthalimido ethyl ester of triftoric acetic acid.

valence vibrations of the -C-O- bond were observed.

Analysis of the PMR spectrum: 400 MHz,  $C_6D_6$ , chemical shift of protons ( $\delta$ , ppm), the singlet signal of two hydrogen protons in the nitrogen-bound methylene group at 3.2 ppm (2H, N-CH<sub>2</sub>), and the singlet signal of the chlorine-bound methylene group protons at 5.3 ppm (2H, Cl-CH<sub>2</sub>), multiplet signal of aromatic ring hydrogen protons in 6.70 and 7.25 ppm (4H, ArH) were observed.

Spectrum  $^{13}C$  (400 MHz,  $C_6D_6$ )  $\delta$ =40,485(-CH<sub>2</sub>-Cl);  $\delta$ =62,243(-N-CH<sub>2</sub>-O-);  $\delta$ =123,903-123,708(-N-C=O);  $\delta$ =128,373-134,483(ArC<sup>1</sup>-ArC<sup>6</sup>)  $\delta$ =166,437-166,289(=C=O).

*Phthalimido methyl ester of monoiodic acetic acid* is a light yellow crystalline substance, product yield 43%, melting temperature  $T_{\text{melting}} = 137$  °C,  $R_f = 0.79$  in TLC (silulfol, system benzene:acetone =3:1).

Specific absorption regions in the IR spectrum of phthalimido methyl ester of monoiodic acetic acid (cm<sup>-1</sup>): Deformation vibrations of the CH bond in the 1,2-disubstituted aromatic ring at 725, 798; Valence vibrations of C=C bonds and 2957 C-H bonds in 1390 aromatic ring, 1703, 3437 -CO-NH- amide; valence vibrations of the nitrogen-bound CH<sub>2</sub> group at 1390; In 1703, 1770 -CH<sub>2</sub>-COOR valence vibrations of the ester bond and in 1247 the -C-O-bond valence vibrations were observed.

Analysis of the PMR spectrum: 400 MHz,  $C_6D_6$ , chemical shift of protons ( $\delta$ , ppm) the singlet signal of nitrogen-bound methylene group protons at 4.47 ppm (2H, N-CH<sub>2</sub>), and the iodine-bound methylene group proton singlet signal at 5.37 ppm (2H, J-CH<sub>2</sub>), multiplet signal of aromatic ring hydrogen protons in 6.75 and 7.30 ppm (4H, ArH) were observed.

Spectrum  $^{13}C$  (400 MHz,  $C_6D_6$ )  $\delta$ =41,963(-CH<sub>2</sub>-J);  $\delta$ =61,651(-N-CH<sub>2</sub>-O-);  $\delta$ =123,429-123,936(-N-C=O);  $\delta$ =128,300-134,197(ArC<sup>1</sup>-ArC<sup>6</sup>)  $\delta$ =166,578-168,132(=C=O).

*Phthalimido methyl ester of trichloro acetic acid* is a white crystalline substance, product yield 49%, melting temperature  $T_{\text{melting}} = 178$  °C,  $R_f = 0.68$  in TLC (silulfol, system benzene:acetone =3:1).

Specific absorption regions in the IR spectrum of phthalimido methyl ester of trichloro acetic acid (cm<sup>-1</sup>): Deformation vibrations of the CH bond in the 1,2-disubstituted aromatic ring at 734,845; 1667 valence vibrations of C=C bonds in the aromatic ring and 2963 C-H bonds, 1704, 3467 -CO-NH-amide; valence vibrations of the nitrogen-bound CH<sub>2</sub> group in 1349; 1772 -CH<sub>2</sub>-COOR valence vibrations of the ester bond and 1165, -C-O-bond valence vibrations were observed.

Analysis of the PMR spectrum: 400 MHz,  $C_6D_6$ , chemical shift of protons ( $\delta$ , ppm) the singlet signal of nitrogen-bound methylene group protons is at 5.01 ppm. (2H, N-CH<sub>2</sub>), the multiplet signal of aromatic ring hydrogen protons in 6.7 and 7.3 ppm. (4H, ArH) were observed.

Spectrum  $^{13}C$  (400 MHz,  $C_6D_6$ )  $\delta$ =30,409(-CCl<sub>3</sub>);  $\delta$ =61,643-67,027(-N-CH<sub>2</sub>-O-);  $\delta$ =123,331-123,687(-N-C=O);  $\delta$ =132,660-133,734(ArC<sup>1</sup>-ArC<sup>6</sup>)  $\delta$ =167,595(=C=O).

*Phthalimido methyl ester of triftoric acetic acid* is a white crystalline substance, yield 50%, melting temperature  $T_{\text{liquid}} = 92-93$  °C,  $R_f = 0.84$  in TLC (silulfol, system benzene:acetone =3:1).

Specific absorption regions in the IR spectrum of Phthalimido methyl ester of triftoric acetic acid (cm<sup>-1</sup>): Deformation vibrations of the CH bond in the 1,2-substituted aromatic ring at 712,; 1706 C=C bonds in the aromatic ring 1720, 3485

-CO-NH- amide; valence vibrations of the nitrogen-bound CH<sub>2</sub> group were observed in 1304; valence of the ester complex -CH<sub>2</sub>-COOR ester bond in 1720 and valence vibrations of the C-O-bond in 1146,1091.

Analysis of the PMR spectrum: 400 MHz, C<sub>6</sub>D<sub>6</sub>, chemical shift of protons (δ,ppm), the singlet signal of nitrogen-bound methylene group protons is at 4.47 ppm (2H, N-CH<sub>2</sub>), the multiplet signal of aromatic ring hydrogen protons is in 6.7 and 7.25 ppm. (4H, ArH) were observed.

Spectrum <sup>13</sup>C(400 MHz, C<sub>6</sub>D<sub>6</sub>) δ=29,886-30,451(-CF<sub>3</sub>); δ=61,741-67,060(-N-CH<sub>2</sub>-O-); δ=123,364-123,719(-N-C=O); δ=132,684-137,586 (ArC<sup>1</sup>-ArC<sup>6</sup>) δ=167,629-167,999(=C=O).

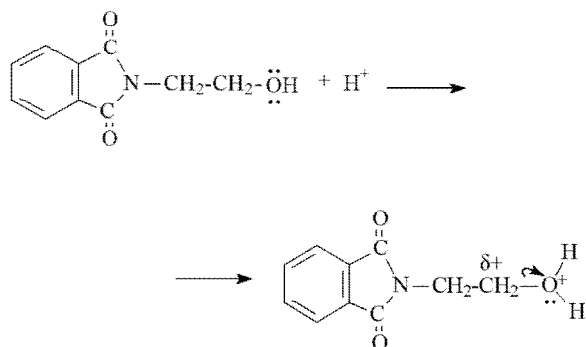
### Results and discussion

The nature of the substituent in carbonic acid is important in the production of carbonic esters. If the substituent has an electron donor property, the positive charge value on the carbon atom of the carboxyl group decreases and O-nucleophilic exchange becomes difficult [13]. We know that monochloroacetic acid reacts more easily than acetic acid. This is due to the increase in the value and acidity of the positive charge of carbon in the carboxyl group when a single hydrogen in acetic acid is exchanged for chlorine [14]:

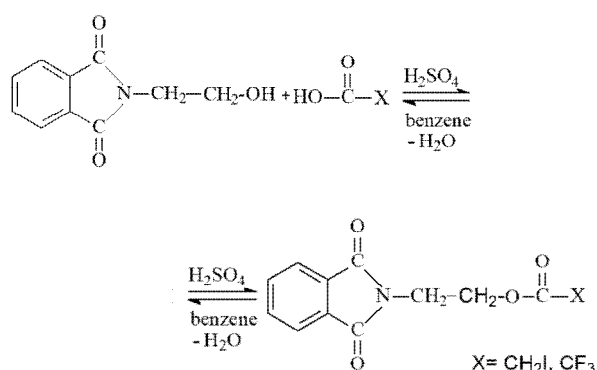
acetic pKa = 4.76, propion pKa = 4.88, caprylic pKa = 4.88, adipine 1) pKa = 4.42, 2) pKa = 5.28, chloroacetic pKa = 2.86, bromoacetic pKa = 2.86, dichloroacetic pKa = 1.29, trichloroacetic pKa = 0.65, trifluoroacetic pKa = 0.23, tribromoacetic pKa = 0.66.

As a result, the penetration of the amidoalkylation reagent is facilitated [15]. This means that acetic acid, which is held by strong acceptor atoms, is easier to exchange O-nucleophilic than other homologues, and the yield is higher [16]. As the ester formation is reversible, the release of water from the reaction medium is also important as the equilibrium shifts to the right.

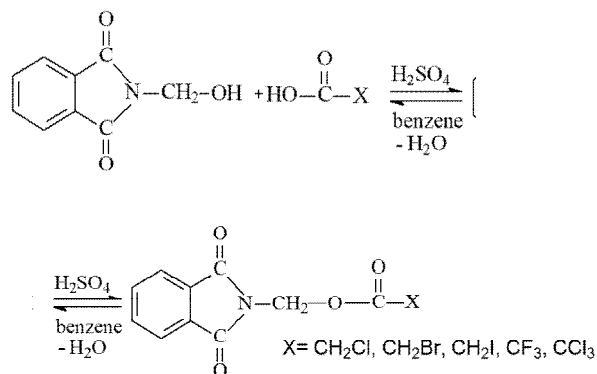
The amidoalkylation reagent N-β-hydroxyethylphthalimide can also form oxone complex ions and decrease nucleophilic properties under the influence of large amounts of catalysts or proton solvents, such as primary alcohols:



Hence, aprotic solvents or solvents that remove water in the form of an azeotropic mixture can be used for the nucleophilic substitution reaction. The use of benzene as a solvent in amidoalkylation reactions with N-β-hydroxyethylphthalimide gives good results. Synthesis of phthalimidoethyl ester of monochloroacetic acid is carried out under the conditions of re-acetoxyethylphthalimide synthesis reaction, the ester is formed with 84% yield [17]. It was found that if the resulting water is removed from the reaction medium using benzene, the product is formed with a yield of 98.5% [18]. The ester yield of the complex obtained by the production of monoiodic acetic acid for the reaction is 82%, and the yield of phthalimidoethyl ester of trifluoroacetic acid is 83%:



There is also a slight decrease in the amount of esters formed by the reaction of halogenated acetic acids with N-methylolftalimide (59%, 54%, 43%, 50%, 49%):



As a result of the electron effect of the methylene group N-methylolftalimide to the two carbonyl groups, the carbon-oxygen bond is weakened and the O-nucleophilic properties are reduced [19]. As a result, it can be assumed that N-methylphthalimide reacts not as a primary alcohol but as a tertiary alcohol [20]. Therefore, the amidoalkylation reaction of halogen acetic acids with N-methylolftalimide under the conditions selected for synchronous bimolecular substitution was somewhat lower. The formation of esters can be considered as independent of the acidity of acetic acid products.

### Conclusion

As a result of amidoalkylation reactions of halogen acetic acids with N- $\beta$ -hydroxyethylphthalimide and N-hydroxymethylphthalimides, phthalimidoalkyl esters of monochlorine, monobromine and monoiodic acetic acid and phthalimidoalkyl esters of trifluoric trichloroacetic acid were obtained. It was shown that the yield of the synthesized esters was formed according to the nature of the sol-

vent obtained for the esterification reaction. Since the selected benzene solvent is a suitable solvent for bimolecular nucleophilic substitution reactions in primary alcohols, it was found that phthalimidoethyl esters of halogen acetic acids are formed with high yield. It has also been shown that although N-hydroxymethylphthalimide is a primary alcohol, it reacts like a tertiary alcohol in reactions with halogenic acid in a benzene solvent.

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