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STUDY OF THE REACTIONS OF ETHYL CHLOROHYDRIN AND MONOCHLOROACETIC ACID ETHYL ESTER WITH SOME TERTIARY AMINES

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Abstract. A quaternary ammonium salt was synthesized based on ethylene chlorohydrin. Alternative reaction conditions were studied to optimize the synthesis process. The resulting quaternary ammonium salt was subsequently reacted with chitosan. Given the inherently high biological activity of chitosan, its conversion into a quaternized, positively charged form was found to further enhance its bioactivity. The synthesized compounds were analyzed using physicochemical methods, including IR and ^{1}H NMR spectroscopy. The obtained data confirmed the successful formation of the quaternary ammonium chitosan derivative.

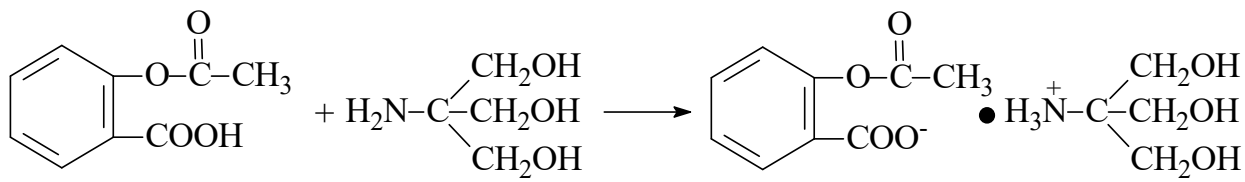
Keywords: Ethylene chlorohydrin, Chitosan, DMF, Ethanol, Choline, Trabectedin, Solifenacin, Noscapine, Quinapril, IR spectrum, ^{1}H NMR spectrum.

Introduction

Filling our country's need for biologically active substances, as well as synthesizing and implementing new import-substituting compounds, is one of the urgent tasks facing chemists. The use of new catalysts and new methods in the synthesis of organic compounds leads to further simplification of processes, development of convenient methods, increase in product yield, and acceleration of reactions. Such methods can also be used when studying the entry of tertiary amines into nucleophilic substitution reactions. To study the interaction of reagents, several groups of reagents of the same type were used, while the nature of the hydrocarbon residues in the molecules was changed. It is these factors that determine their ability to enter into the reaction. In this case, the activity of amino groups in nucleophilic substitution reactions is affected by the spatial and induction effects of substituents on the nitrogen atom.

A method for obtaining acetylsalicylate [tris(hydroxymethyl)], which has antipyretic, analgesic, antipyretic and antiplatelet activity in medicine, is presented.

This compound is well soluble in water and is characterized by low toxicity. For this, a mixture of 13.5 g of acetylsalicylic acid and 9 g of tris-(hydroxymethyl)-amino methane is poured into a system consisting of 9 ml of H2O and 9 ml of isopropanol. The mixture is stirred for 1 hour. Then the resulting precipitate is filtered and washed with 70 ml of isopropanol and then 70 ml of ether. The yield of the product is 85%.[21]



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Reaction of ethylene chlorohydrin with triethylamine

To study the reactions of obtaining choline analogues from tertiary amines, the reaction of ethylene chlorohydrin with triethylamine was studied, and the reactions were carried out in various solvents to study the effect of the solvent on the product yield. These results are given in Table 2.4.

Table 1.1

β - Effect of solvents on the yield of hydroxyethyltriethylammonium chloride

№	Ethylene chlorohydrin : triethylamine mole ratios	Solvent	Product, %
1.	1:1	Toluene	38
2.	1:1	Chloroform	63
3.	1:1	Ethyl acetate	69

The results in the table show that when the reaction was carried out in a 1:1 molar ratio of the reactants, the product yield was 69%.

$$CH_2Cl{-}CH_2OH + (C_2H_5)_3N \longrightarrow [(C_2H_5)_3N^+{-}CH_2{-}CH_2OH]Cl^-$$

Table 2.5

β - Effect of mole ratios of reagents on the yield of hydroxyethyltriethylammonium chloride

№	Mole ratios of ethylene chlorohydrin and triethylamine	Solvent	Product, %
1.	1:1	Ethyl acetate	69
2.	1:1,5	Ethyl acetate	76
3.	1:2	Ethyl acetate	79

The results of the table show that the product yield was 79% when the reaction was carried out with a molar ratio of reagents of 1:2. The reactions were carried out for a longer period of time

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in ethyl acetate solution with a molar ratio of reagents of 1:1. The results of these experiments are presented in Table 1.1.

Table 1.2

β - Effect of reaction time on yield of hydroxyethyltriethylammonium chloride (solvent ethyl acetate)

№	Mole ratios of ethylene chlorohydrin and triethylamine	reaction time, (hour)	Product, %
1.	1:1	2	69
2.	1:1	4	75
3.	1:1	6	82

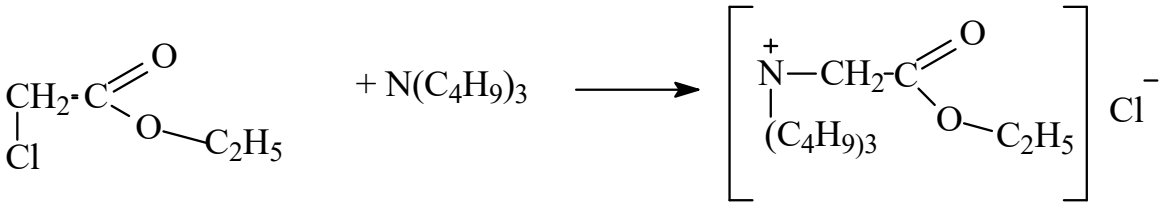
The results in Table 1.2 show that the yield of the reaction of ethylene chlorohydrin with triethylamine at room temperature in ethyl acetate solvent increases with increasing reaction time. We believe that the yield can be further increased.

When the reaction was carried out at room temperature, when we mixed the substances, the walls of the vessel heated up and white smoke was produced. When this reaction was carried out in an ice-water bath with cooling, the reaction yield increased sharply and a white salt (-hydroxyethyltriethylammonium chloride salt was isolated. The resulting salt was placed in a pre-weighed container and placed in a desiccator.

Based on the results of the experiments, it can be said that at low temperatures the solubility of the salt obtained and the volatility of the reagents decrease, allowing the preparation of (-hydroxyethyltriethylammonium chloride salt with a high yield.

Alternative conditions for the synthesis of the obtained compound were found, and β-hydroxyethyltriethylammonium chloride is a white crystalline hygroscopic substance that liquefies at 218-220°C. In order to confirm that the product is β-hydroxyethyltriethylammonium chloride, its IR spectrum was obtained and submitted for study of biological activity.

Tributyl carbethoxy methylammonium chloride salt preparation



Received for reaction:
Monochloroacetic acid ethyl ether 6.125 g, 5.28 ml, (0.05 mol)
Tributylamine 9.25g; 12 ml, (0.05 mol)

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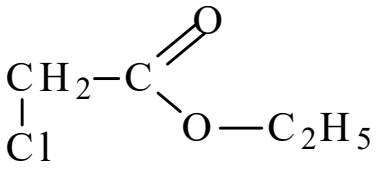

: $N(C_4H_9)_3$ Моддаларнинг моль нисбатлари

In a 1:1 mole ratio

In order to increase the yield of this reaction and determine the optimal conditions, ethyl monochloroacetic acid was prepared for the reaction by distillation at 144° C. Tributylamine was also prepared for the reaction by distillation at 243° C.
In order to further increase the yield of the reaction, 5.28 ml of ethyl monochloroacetic acid and 12 ml of tributylamine were taken for the reaction based on the information in the literature. 12 ml of a 33% solution of tributylamine in absolute alcohol was heated at a temperature of 65-70° C. The molar ratio of the reactants was 1:2 and the temperature was 65-70° C. Due to the fact that the reaction was carried out at a temperature of 65-70° C, tributylamine was absorbed into absolute alcohol in the form of a gas, taking into account the volatility of tributylamine in absolute alcohol.
At the end of the experiment, the colorless liquid in the flask was evaporated with alcohol in a water pump and placed in a desiccator to dry. It was dried in a desiccator to form colorless crystals. The dried mass was weighed to calculate the product yield. Then 10 g of product was obtained and the yield was 73%.

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