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INTERACTION OF QUATERNARY SALTS OF 1,2-AMINOALCOHOL WITH CONCENTRATED AQUEOUS LIQUIDS OF ALKALI

Abstract: One of the topical subjects of modern organic chemistry is to identify ways to regulate the reactions of multifunctional organic compounds in order to obtain, as well as increase the number and volume of production of high-quality synthetic substances, products of small-scale chemistry. These substances include quaternary ammonium salts containing a 1,2-amino alcohol fragment. In this work, the laws of the interaction of some quaternary salts of 1,2-aminoalcohols with bases are investigated and ways of regulating the reactions of functional centers are revealed. As the result of our researches, it was discovered that in concentrated aqueous liquids of alkali, quaternary derivatives of 1,2-amino alcohol show new specifications, constituting formation of active, reactive bipolar compounds – 2-trialkylammonio ethoxides. Mechanism and process pattern was studied.

Key words: quaternary salts, 1,2 – aminoalcohol, alkali, ammonioethoxides, ammonium salt, trialkylammonio ethoxides, three-phase systems, tertiary amines.

Language: English

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Introduction

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In the present work, it was noted that there is no information in the literature regarding the interaction of the quaternary salts of 1,2-aminoalcohols with concentrated aqueous solutions of alkalis. Therefore, in order to search for new ways of directed synthesis of multifunctional organic compounds, we have studied and identified the process pattern.

It was defined that solid alkalis or their concentrated solutions at 15–20°C interact with aqueous solutions of quaternary salts of 1,2-aminoalcohols, and cause salting-out of light-yellow viscous organics from the solution. The salting-out process starts while the alkali concentration reaches 25-30%. The salting-out process finishes when the

alkali concentration is up to 50-55%. In this case, a hetero three-phase system is obtained.

It should be noted that the similar occurrences are observed in the interaction of solutions of tetraalkylammonium halogenides $R_4N^+X^-$ with concentrated aqueous solutions of alkalis [1]. It was defined that the organic phase that was liberated by the process contains a mixture of the initial halogenide $R_4N^+X^-$ and its OH form, i.e. the exchange reaction is not fully completed and the yield of $R^4N^+OH^-$ does not exceed 30%. However, in the interaction of quaternary salts solutions of 1,2-aminoalcohols with alkali solutions, the conversion nature is significantly different from the conversion nature of tetraalkylammonium halogenides in the same solutions. So, in an analogical treatment of quaternary salts of 1,2-aminoalcohols, the phases of the obtained system contain the following reaction components:

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the lower solid phase mainly consists of an alkali metal halogenide; besides water and alkali, the liquid middle phase contains an alkali metal halogenide; the upper phase correspond to a viscous organic, sometimes crystallizing at 5-10°C. The analysis results of this phase showed the following:

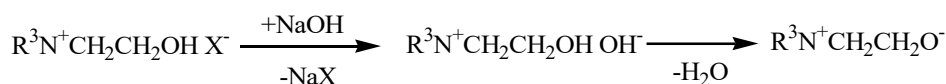
- it does not contain a tertiary 1,2-aminoalcohol, a tertiary amine or organic compounds of different composition, i.e. the destruction of the ammonium cation of the initial quaternary salt does not happen;

- halogenide absence; by gravimetry method with implementation of AgNO₃ it was defined that the halogen quantity which corresponds to initial quaternary salt, remains in two phases;

- during titration, halogen hydrogen acid is consumed, the amount of which stoichiometrically corresponds to the amount of halogen contained in the initial quaternary salt;

- in the phase ultimate composition there is no stoichiometric amount of water elements corresponding to the OH form of the initial quaternary salt of 1,2-aminoalcohol;

These data allow us to come to the conclusion that at the result of de-protonization of hydroxyl in the 2-hydroxyethyl group in the intermediate OH-form of the initial quaternary salt of 1,2-aminoalcohol, bipolar 2-trialkylammonioethoxides are generated and formed.



The direction of this reaction is determined by the properties of aqueous solutions of alkalis. It was determined that in 48% aqueous solution of NaOH there are 71% of free hydroxyl ions [2]. And hydroxyl ions have a huge proton affinity ($RA_{\text{OH}^-} = 16.6$ eV), being second only to the amide ion ($RA_{\text{NH}_2^-} = 18.17$ eV) among monovalent ions [3,4]. Beside it, their activity in concentrated alkali solutions is higher, and the activity of water is much lower [5]. In such solutions, water in its free form does not exist, but is in a specific interaction with alkali, since a 50% aqueous solution of NaOH has a rather high drying property and the vapor pressure of water above the solution at 20°C is only 3.9 kPa (2 mmHg) [6]. In the H-NMR spectra, a water signal appears at 4.95-5.01 ppm. when alkalis are dissolved in it, up to 50-55% is shifted to a weaker field and is detected at 6.04-6.23 ppm. As is seen from these data, despite of 50% water presence, such alkali solutions behave similarly to anhydrous, highly dehydrated systems. Therefore, in these solutions, the de-protonization of the 2-hydroxyethyl group in the quaternary salts of 1,2-amino- alcohols under the action of strong proton acceptors - OH ions easily occur, and in the ammonium cation the electron-withdrawing trialkyl ammonium group (R_3N^+) causes shift of average positions of the electrons forming the bonds along atoms' chains, increases the dissociation of the proton

of $\text{N}^+\text{CH}_2\text{CH}_2\text{OH}$ group and stabilizes the formed ethoxide - ion.

Analogical to ionic compounds, the low solubility of 2-trialkylammonioethoxides in inert apolar solvents (CDCl_3 , CCl_4 , C_6H_6) and their sensitivity to proton solvents limited the wide implementation of H-NMR spectroscopy for recording these compounds.

The removal of the H-NMR spectra of 2-trialkylammonioethoxides in alkali solutions in D₂O is also complicated by the transition of the system to heterophase by increasing concentration of these solutions.

The H-NMR spectral data of some quaternary salts of 1,2-amino alcohols in D₂O solutions at various alkali concentrations are shown below:

In the H-NMR spectra of (2-hydroxyethyl)triethylammonium chloride in D₂O (Fig. 1a) and in the presence of NaOH in D₂O (Fig. 1b), the multiplet in the area of 4.32 ppm. (signal center), caused by protons of -CH₂O group in the presence of 27% alkali, appears as sharp triplet at 4.34 ppm. The multiplet of protons of methylene groups and directly bonded with quaternary nitrogen atom at 3.73 ppm in the presence of NaOH strongly transform its shape and appears as a triplet. Signal of methyl protons at 1.63 ppm shift towards weak fields and appears at 1.73 ppm. It should be mentioned that the sharpness of the triplet is disturbed and looks like an overlay of triplets.

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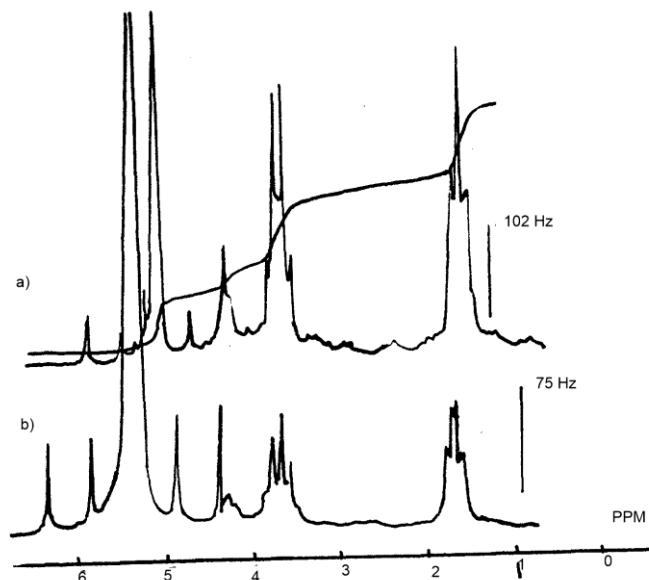
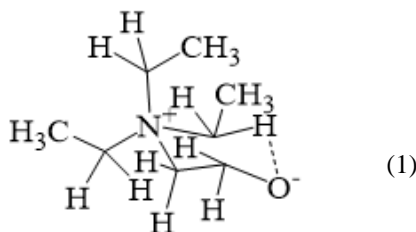


Fig. 1. H-NMR spectrum of (2-hydroxyethyl)triethylammonium chloride in a solution of D₂O (a) and in a 27% solution of NaOH in D₂O (b)

Probably, it is connected with the formation of 2-triethylammonioethoxide (1) in the solution. While an anionic oxygen of the 2-ethoxide group, having a high proton affinity, interacts on H-bond type with the

most protonated hydrogen atom of the methylene group of one of the ethyl radicals. At the result of de-screening, the CH₃ group of this radical appears to be varied from the rest.



Analogical effect is clearly detected in compounds spectra, in which the protonation of the hydrogen of the methylene group at the nitrogen atom is strengthened by the action of the neighboring aromatic system. For example, in the H-NMR spectrum of benzyl(2-hydroxyethyl) diisopentylammonium chloride - (*i*-C₅H₁₄)₂N⁺(CH₂CH₂C₆H₅)(CH₂CHOH)Cl⁻ taken in a mixture of 50% D₂O and 50% (CD₃)₂CO in the presence of KOH, the splitting of protons signal occur in the -C₆H₅

group, which is not observed in the spectra of the same compound in the indicated solvent mixture (Fig. 2b) and in D₂O (Fig. 2a) in the absence of alkali, which proves the contribution of the intramolecular association of the formed bipolar ion (2). As a result of the delocalization of electrons in the aromatic ring, respectively in the regions of 7.68 ppm. o-H and p-H are revealed, and at 7.48 ppm m-H. In this case, the proton signal of the N⁺-CH₂-Ar group is shifted for 8 Hz to the side of weak fields.

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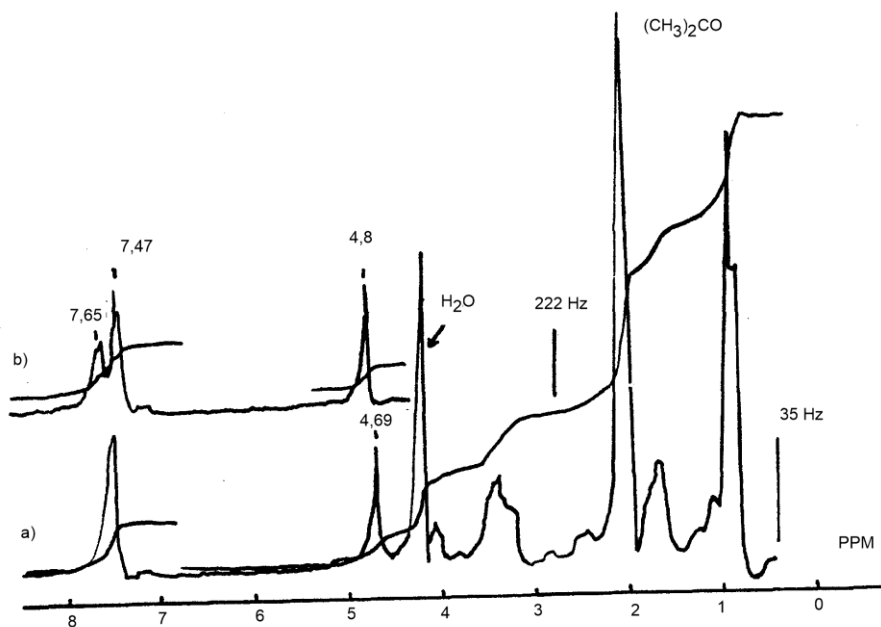
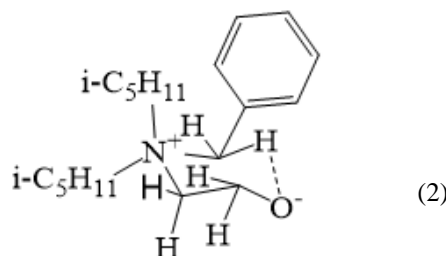


Fig. 2. H-NMR spectrum of benzyl(2-hydroxyethyl)diisopentylammonium ($i\text{-C}_5\text{H}_{11}$)₂N⁺(CH₂CH₂C₆H₅)(CH₂CHOH) Cl⁻ in solution of D₂O + (CD₃)₂CO (a) and in solution of D₂O + (CD₃)₂CO + KOH (b)



The confirmation of the above mentioned - is the fact that in the H-NMR spectrum of benzyltriethylammonium chloride (C_2H_5)₃N⁺CH₂C₆H₅ Cl⁻ containing besides 2-hydroxyethyl, the same groups as the compound (2-hydroxyethyl) triethylammonium chloride and benzyl (2 -hydroxyethyl) diisopentylammonium chloride

under the same conditions, due to the impossibility of the formation of a bipolar compound, no splitting of the proton signal of the aromatic ring is observed, and at the same time a sharp triplet of methyl groups existing in ethyl radicals is observed (Fig. 3).

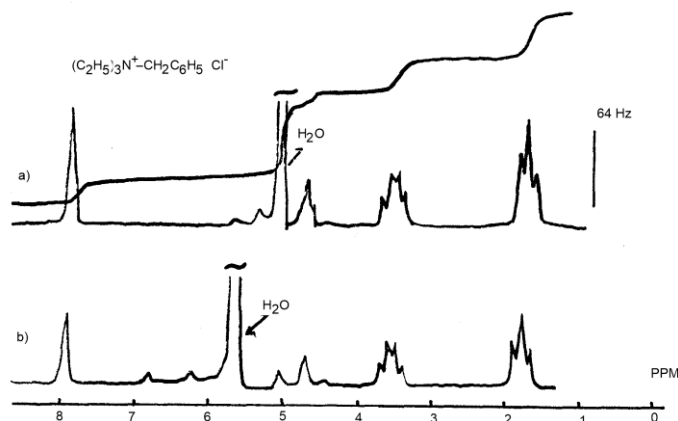


Fig. 3. H-NMR spectrum of benzyltriethylammonium chloride (C_2H_5)₃N⁺CH₂C₆H₅Cl⁻ in of D₂O (a) solution and in D₂O + (CD₃)₃CO + KOH solution

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In the H-NMR spectra of 4-benzyl-4-(2-hydroxyethyl)morpholinium chloride, proton signals in C₆H₅, ArCH₂, CCH₂O, CCH₂N⁺ and solvent groups appearing in ranges of 7.83, 5.05, 4.38, 3.84 and 5.01 ppm, in the presence of 50% KOH are detected at 8.01, 7.89, 5.09, 4.46, 3.79 and 6.04 ppm, respectively (Fig. 4). Herewith, the splitting of proton signals in the

aromatic ring of 8.01 and 7.89 ppm and a shift in the weak ranges of the signal centers of the CCH₂Ar and CCH₂O groups also occur. In this case, the signal of the CCH₂O group relative to the signal of the CCH₂N group is shifted for 11.2 Hz (0.14 ppm) towards weak fields, which proves a strong change in the electronic environment of the oxygen atom

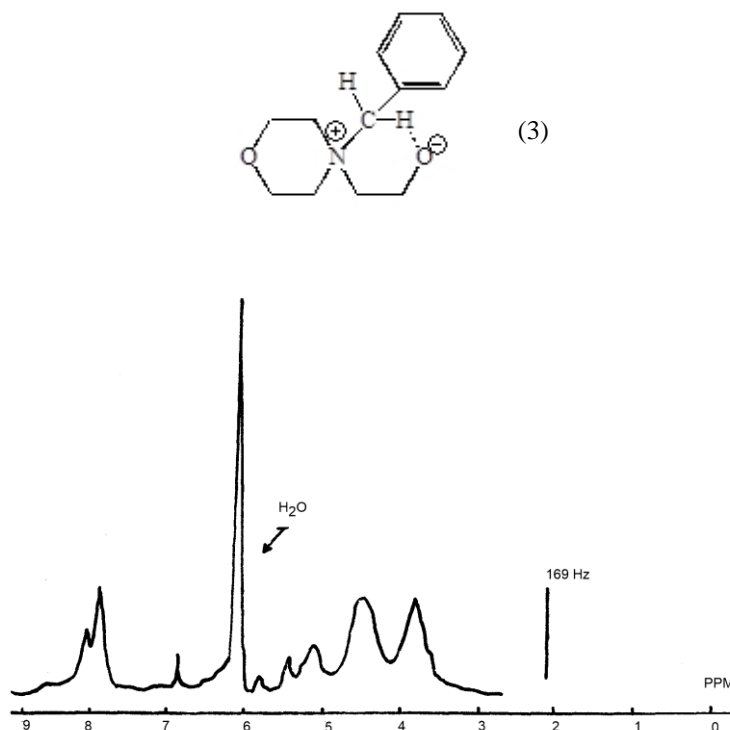


Fig. 4. H-NMR spectrum of 4-benzyl-4-(2-hydroxyethyl)morpholinium chloride O(CH₂CH₂)N⁺(CH₂CH₂OH)Cl⁻ in H₂O + D₂O + KOH (50 %) solution

We also studied the H-NMR spectrum of 1-(2-hydroxyethyl)pyridinium chloride in solutions of

KOH in D₂O with a concentration of 0 (a), 27 (b), 50% (c), respectively (Fig. 5)

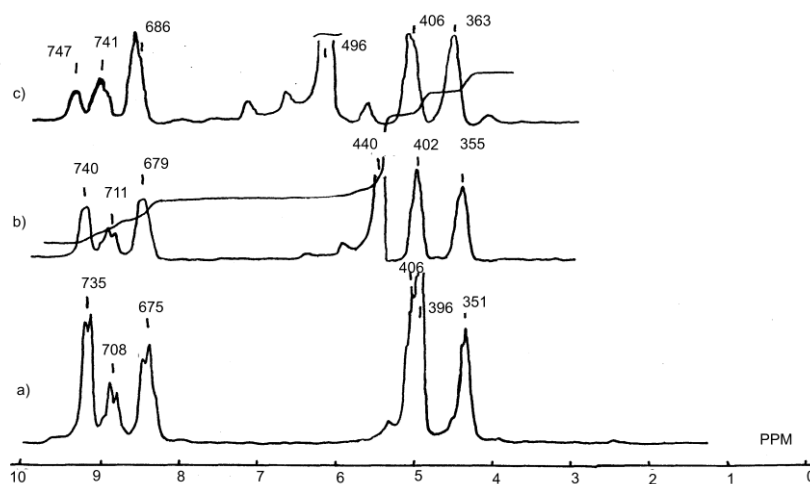


Fig.5. H-NMR spectrum of 1-(2-hydroxyethyl)pyridinium chloride in solutions of KOH in D₂O with a concentration of 0 (a), 27 (b), 50% (c), respectively

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As is seen from the H-NMR spectral data shown in Table 1, with an increase of KOH concentration in D₂O-H₂O solutions of 1-(2-hydroxyethyl)pyridinium chloride, a dislocation occurs at 11-13 Hz signals of various nuclear of H, existing in the heterocycle and CCH₂O group. Herewith, $\delta_{H_2O} = 6.23$ ppm. (498 Hz).

As it should be expected, the proton signal of the methylene group directly born by the nitrogen atom practically does not react to changes in KOH concentration and appears at 5.1 ppm. (406Hz). However, the intensity of the proton signal, in α -C atom of heterocycle decreases, and the proton signal at the β -C atom increases twice, i.e. the signal of one of the protons at α -C atom in the heterocycle appears at the same range, where the proton signal at the β -C

atom emerge, which indicates the interaction of hydrogen at α -C atom of heterocycle with electron-negative oxygen in the 2-ethoxide group. Herewith, the signal dislocation to a stronger field is 61 Hz. Additionally, the mismatch of chemical dislocations values of corresponding protons in free pyridine ring and in analyzed compound proves that the nitrogen atom in the latter is in a positively charged state. It should be noted that, similarly to ionic compounds, the chestnut-brown organic layer, released during the interaction of 1-(2-hydroxyethyl)pyridinium chloride under the action of 50% aqueous KOH does not dissolve in aprotic apolar compounds, and also practically does not contain halogen in its structure. Upon adding HCl acid, the original salt is restored, and the color of the solution disappears

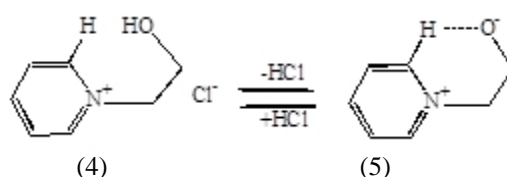
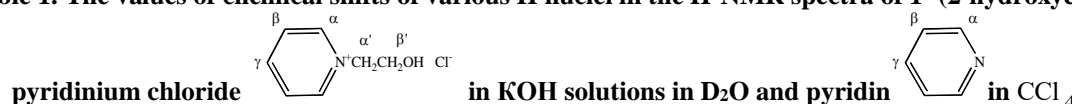


Table 1. The values of chemical shifts of various H nuclei in the H-NMR spectra of 1- (2-hydroxyethyl)



Compounds	KOH concentration in D ₂ O, in %	Values of chemical shifts of H, in Hz					
		α	β	γ	α'	β'	D ₂ O \rightleftharpoons H ₂ O
1-(2-hydroxyethyl) - pyridinium chloride	0	735	657	708	406	351	396
	27	740	679	711	402	355	440
	50	747	686	721	406	363	448
Signal shift, in Hz							
Pyridine		12	11	13	0	12	102
	-	680	564,8	596,8	-	-	-

As was shown above, the formation of 2-trialkylammonioethoxides from the quaternary salts of 1,2-aminoalcohols is associated with a change of series of physicochemical properties of water in alkali solutions.

With a decrease in alkali concentration in an aqueous solution, this reaction is suppressed, and the nature of the transformation complies with thermodynamic control. For example, (2-

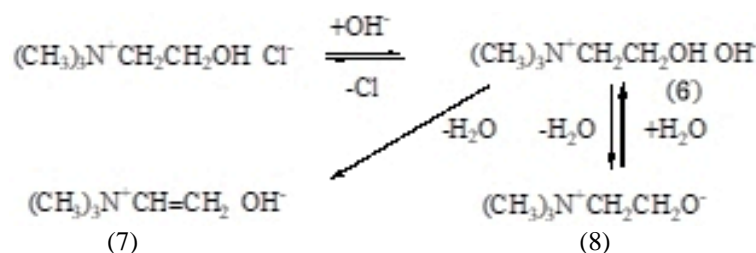
hydroxyethyl) trimethylammonium chloride or its OH form (6) obtained by exchanging the Cl⁻ ion for OH⁻ ion with wet Ag₂O in a 5-10% aqueous KOH solution, at 15-20°C stable, at 100°C for 20 hours forms neurine – vinyltrimethyl ammonium hydroxide (7), and in a 50-55% aqueous solution of KOH at 15-20 ° C for 30 minutes forms 2-trimethylammonioethoxide (8) according to scheme:

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It shows that the formation of two possible products depends on the reaction conditions, i.e. in the absence of a solvent of hydroxyl nature and in the presence of a strong base (OH⁻-ions), the reaction product is 2-trimethylammonioethoxide (8), a product of kinetic control. In the presence of water and alkali in neurine (7), the double bond does not hydrate and therefore it is more stable than 2-trimethylammonioethoxide (8). Consequently, it is thermodynamic control product and when equilibrium is established between (2-hydroxyethyl)trimethylammonium hydroxide (6) and 2-trimethylammonioethoxide (8), neurine is the dominant reaction product.

Thus, while studying the reaction of some quaternary salts of 1,2-amino alcohols with concentrated aqueous solutions of alkalis, we found out their important and common new feature, which is the formation of a new type of compounds, namely, bipolar 2-trialkylammonioethoxides.

Methods of compounds preparations in the reactions of 1,2-aminoalcohols and their quaternary derivatives proceeding with the participation of a hydroxyl group (Experimental part)

The interaction of quaternary salts of 1,2-aminoalcohols with aqueous solutions of alkalis

1. Generic methodology of the preparation of 2-trialkylammonioethoxides.

a) In 100 ml weighing bottle, 0.1 mol of the quaternary salt of 1,2-aminoalcohol in 8-10 ml of water was added to 50 g of a 50-55% aqueous solution of potassium hydroxide (or sodium hydroxide) at 5-10 °C. The reaction mixture is stirred for 20-30 minutes. Afterwards, stopping mixing, the weighing bottle is closed with a lid. When left to stand, the reaction mixture demixes and forms a three-phase system. Herewith, the upper phase consists of viscous organics. The middle phase contains water; alkali and alkali metal halogenide. The lower solid phase consists of alkali metal chloride. In a parallel experiment using gravimetric methods with implementation of silver nitrate, it was found that the amount of halogenide in the middle aqueous and third solid phases corresponds to the quantity of used initial quaternary salt of 1,2-aminoalcohol. A solid alkali is carefully loaded into the middle phase through a glass

tube until water is completely absorbed in the middle phase. Then the viscous organic part is separated and transferred to a flask and washed with benzene. Residues of benzene are removed at 8-10°C under reduced pressure. The residue consists of 2-trialkylammonioethoxide, which is practically obtained in quantitative yield. In Table 2 the physical indicators and analytical data of some 2-trialkylammonioethoxides obtained by the above procedure are presented.

b) In a flask equipped with a stirrer, 0.5 mol of silver oxide is added to 0.1 mol of quaternary salt of 1,2-amino alcohol and 25 ml of water at 5-10°C while stirring. Then the precipitated residuum of silver halogenide is separated. The aqueous solution of the quaternary hydroxide of the 1,2-aminoalcohol is transferred to a 100 ml bottle containing 50 ml of 50% aqueous alkali at 5-10°C. Then the reaction mixture is left off for foliation. Herewith, the separated organic phase is treated and separated as described in method 1.

2. Obtaining benzyltriethylammonium chloride.

Benzyltriethylammonium chloride was obtained on the basis of triethylamine and benzyl chloride in benzol upon the application of heat.

3. H-NMR spectral studies of D₂O solutions of quaternary salts 1,2-aminoalcohols, resulting in the formation of the corresponding 2-trialkylammonioethoxides (1, 2, 3, 5, 8) in the presence of various concentrations of alkalis were carried out on a device BS 4870-80, made by Tesla firm.

4. Preparation of vinyl trimethylammonium hydroxide (7) on the basis of (2-hydroxyethyl) trimethylammonium chloride. In a three necked flask, 35 ml of a 15% aqueous potassium hydroxide solution are added to 13.95 g (0.1 mol) of (2-hydroxyethyl)trimethylammonium chloride. Then the reaction mixture is boiled for 15 hours. At the end of the reaction, water is removed by evaporation until a viscous mass is formed. The residue remaining in the flask is dissolved in 20 ml of propyl alcohol. The solution is filtered. Then propyl alcohol is distilled off under reduced pressure. The residue is a colorless syrupy liquid, which forms salts with hydrochloric and hydrobromic acids at melting points: 211-213 ° C and 192 ° C, respectively. Yield 8 g (77.7%). Found, in% C 58.49, H 12.84, N 13.41. C₅H₁₃NO. Calculated in%: C 58.25, H 12.62, N 13.59.

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5. Preparation of vinyl trimethylammonium hydroxide (7) based on (2-hydroxyethyl)trimethylammonium chloride hydroxide (6). In a beaker, to a mixture of 50 ml of water and 6.975 g (0.05 mol) (2-hydroxyethyl) trimethylammonium chloride, 5.79 g (0.025 mol) of silver semi oxide are added with stirring. Then, a quantitatively formed precipitate of silver chloride is

separated by filtration. An aqueous solution of (2-hydroxyethyl) trimethylammonium hydroxide is transferred to a flask equipped with a stirrer and 10 g of potassium hydroxide are dissolved in it. The reaction mixture is boiled for 15 hours. At the end, the reaction mixture is treated as described in method 4. The compound (7) is obtained with a yield of 82.6%.

Table 2. The results of the interaction of quaternary derivatives of 1,2-aminoalcohols with a concentrated alkali solution

№	Compound name Chemical formula	Yield, в %	T., °C	Elemental analyses, in %			The content of water elements in the com- pound com- posi- tion, in %
				found/ C	calculated H	N	
1	2- Triethyl ammonio ethoxide C ₈ H ₁₉ ON	94,7	55-60	66,49 66,21	13,37 13,10	9,93 9,66	2,5
2	2- Benzyl diizopentyl ammonio ethoxide C ₁₉ H ₁₃ ON	96,8	105-107	78,20 78,35	11,57 11,34	4,63 4,81	2,1
3	2- (N-Benzylmorpholinio) ethoxide	97,0	107-110	70,35 70,59	8,88 8,60	6,20 6,33	1,5
5	C ₁₃ H ₁₉ O ₂ N	89,5	45-47	6,56 6,56	7,60 7,32	11,12 11,38	2,7
8	2- Pyridinioethoxide C ₇ H ₉ ON	92,4	67-68	58,40 58,25	12,43 12,62	13,70 13,59	2,2
	2- Trimethylammonioethoxide C ₅ H ₁₃ ON						

CONCLUSION

For the first time, a new direction has been discovered for the reaction of quaternary salts of 1,2-aminoalcohols with concentrated aqueous solutions of alkalis, which constitutes in the formation of bipolar 2-trialkylammonioethoxides at 0-20°C. The reaction

complies with kinetic control, includes the intermediate formation of the OH form of trialkyl(2-hydroxyethyl)ammonium and the subsequent deprotonization of hydroxyl in the 2-hydroxyethyl group with OH⁻ ions

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SIS (USA) = 0.912
ПИИИ (Russia) = 3.939
ESJI (KZ) = 9.035
SJIF (Morocco) = 7.184

ICV (Poland) = 6.630
PIF (India) = 1.940
IBI (India) = 4.260
OAJI (USA) = 0.350

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