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## ABOUT TECHNICALLY USEFUL PROPERTIES OF ESTERS ON THE BASE OF ALKYLAROMATIC HYDROCARBONS

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## О ТЕХНИЧЕСКИ ПОЛЕЗНЫХ СВОЙСТВАХ СЛОЖНОГО ЭФИРА НА ОСНОВЕ АЛКИЛАРОМАТИЧЕСКИХ УГЛЕВОДОРОДОВ

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*Abstract.* Method obtaining of ecologically pure dielectric liquid of acetoxymethyl–secondary hexylo–xylol has been developed. Its electro–physical properties allow considering it as perspective substitute of natural castor oil. Efficiency of acetoxylation stage of chlorine methylation products without prelaminar division has been shown. Carried out distillations at chlorinemethylation stage lead to the formation of significant amount of diarylalkanes and yield dielectric liquid is 72.27%. By involving of diarylalkanes into the acetoxylation stage, decomposition of chlorine methylate is prevented under the influence of high temperatures and yield of dielectric liquid increases up to 87.7%. It has been determined that the reaction chlorine methyl–secondary–hexyl–o–xylol of interaction with sodium acetate in presence of triethylphenmethyl chloride proceeds in conditions of heterogenous interphase catalysis. The yield of the product obtained under heterogeneous catalysis conditions is 98% compared to the yield in the product medium in acetic acid 89.5%. The solidification temperature of the synthesized complex product was –42 °C, which greatly exceeds the castor oil solidification temperature.

*Аннотация.* Разработан метод получения экологической чистой диэлектрической жидкости ацетоксиметил–вт.гексил–о–ксилола, электрофизические свойства которого позволяют рассматривать его как перспективный заменитель натурального касторового масла. Показана эффективность стадии ацетоксилирования продуктов хлорметилирования без предварительного разделения. При этом перегонка на стадии хлорметилирования приводят к образованию значительного количества диарилалканов и выход диэлектрической жидкости составляет 72,27%. При вовлечении диарилалканов в стадию ацетоксилирования предотвращается разложение хлорметилата под воздействием высоких температур и выход диэлектрической жидкости повышается до 87,7%. Установлено, что реакция взаимодействия хлорметил–вт.гексил–о–ксилола с ацетатом натрия в о–ксилоле в присутствии триэтилбензилхлорида протекает в условиях гетерогенного межфазного катализа. Выход полученного продукта в условиях гетерогенного катализа составляет 98% по сравнению с выходом в среде продукта в среде уксусной кислоты 89,5%. Температура застывания

синтезированного сложного продукта составила  $-42\text{ }^{\circ}\text{C}$ , что значительно превосходит температуру застывания касторового масла.

*Keywords:* alkylation, chlorine methylation, acetoxylation, dielectric liquid, castor oil.

*Ключевые слова:* алкилирование, хлорметилирование, ацетоксилирование, диэлектрическая жидкость, касторовое масло.

One of the problems of modern electrical engineering is to ensure the reliability of the operation of capacitors with electric field strengths exceeding 150-200MB.

In severe pulse regimes, castor oil (CO) is used as liquid impregnation. However, the CO does not allow providing a resource of 104 charge–discharge pulses and reliability at the level of 0.95–0.99, because the current state standards do not regulate the dielectric parameters. Development of a substitute for natural castor oil, which is a universal impregnating liquid in capacitors, is an urgent problem of the electrical engineering industry. Castor oil–ricinoleic acid triglyceride, despite the difficulty of purification and achieving the necessary thermal stability, is used in low voltage AC film capacitors in the USA, Japan, Italy and other countries [1–3]. By chemical composition, CM is a high molecular organic substance, obtained from castor seeds, cultivated in the southern parts of Asia, America and Europe. The main way to extract oil from seeds is hot and cold pressing, as well as extraction. The composition of the oil includes is following: ricinoleic acid glycerides — 80–95%, oleic 3–9%, linoleic 2–3%, and other unsaturated acids — 3–8%. The main share of glycerides of ricinoleic acid is its mono-, di-, and triglycerides, the quantitative content of which depends on the degree of maturation of the seeds [4]. Comparison of Russian and foreign CMs shows the advantage of imported CM as dielectric liquids. Taking into consideration CO is of vegetable origin, it becomes necessary to replace it with synthetic liquid. CO is used in power capacitors used in electric power industry, industrial electrical engineering and power electronics. In Table 1 the physical and electro-physical properties of the most promising esters used as dielectrics, in particular as impregnants for small capacitors [1–2].

Table 1.

PHYSICAL AND ELECTRO–PHYSICAL PROPERTIES  
 OF THE MOST PERSPECTIVE ESTERS USED AS DIELECTRICS

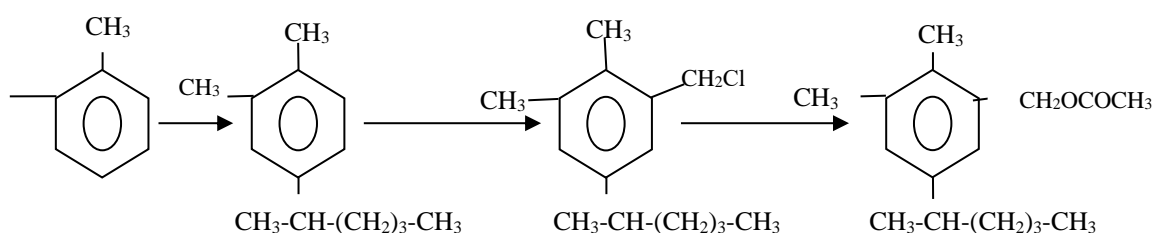
<i>Material</i>	<i>Electrical strength</i>	<i>Density, kg/l</i>	<i>Viscosity at a temperature of 20 °C, mm<sup>2</sup> / s c</i>	<i>modification point, °C</i>	<i>Boiling point, °C</i>	<i>Flashpoint, °C</i>	<i>E, with 20 °C</i>	<i>Tgδ</i>
Castor oil		0.96	1003	-15	313	285	4.65	0.002
Di-n-butyl phthalate		1.04	16.3	-35	340	171	6.30	0.0013
Di-n-heptyl phthalate		0.98	32.5	-40	360	224	5.2	0.002
Diisooctylphthalate	12	0.98	53.0	-46	229/0,6 kPa	221	5.0	0.005
Di-2-ethylhexyl phthalate	12	0.984	83.0	-55	385	218	5.3	0.0009
Diisononyl phthalate	12	—	95	-45	244–252/0.7 kPa	222	4.66	0.0005
Dihexylphthalate		—	50	-33	212/2кPa	243	4.08	0.01
Di-tridecylphthalate	12	—	230	-37	285 /0.7кPa	243	4.08	0.01

Material	Electrical strength	Density, kg/l	Viscosity at a temperature of 20 °C, mm <sup>2</sup> /s c	modification point, °C	Boiling point, °C	Flashpoint, °C	E, with 20 °C	Tgδ
Di-2-ethylheptyl-sebacynate		0.928	22.2	—	256	130	4.0	0.001
Diisodecyl phthalate		0.97	9.9	-50	370	230*	4.56	0.003

\* for Martens–Pensky.

Synthesis of esters of organic acids large of increased molecules mass, as a rule is carried out on the basis of esterification reaction, which refers to classical. At the same time, the deficit of higher alcohols and their high cost, the reversibility of the esterification reaction and the arising difficulty the target product, separation present difficulties for its practical spread. In this connection, the reaction of esters synthesis based on much more accessible halogen derivatives may be of practical interest [5]. Such method synthesis of esters may also be preferable because the carboxylic acid salt is cheaper and more accessible than the acid itself. The advantages of the method include the irreversibility, simplicity and ease the target product, separation the possibility of working on “anhydrous technology”, which becomes particularly important of separation taking into consideration the high sensitivity of esters to water and the effect of hydrolysis on deterioration of the properties of electrical insulating materials.

In connection with this, a three-stage synthesis of acetoxymethyl-sec.hexyl-o-xylene, which is of interest as a potential substitute for castor oil, was carried out. We made an attempt to synthesize the compounds with a shielded ester function based on o-xylene according to the scheme:



Acetoxymethyl-sec.hexyl-o-xylene was synthesized on the basis of successive reactions of alkylation, chloromethylation of alkyl-substituted aromatic hydrocarbons of increased molecular weight, synthesis of esters by reaction of benzyl chlorides with sodium carboxylates. Is necessary to note some results related to the production of alkylbenzenes by alkylation with C<sub>5</sub> — and higher olefins. An analysis of the above list of published sources allows us to conclude that the regularities of alkylation reaction of aromatic hydrocarbons with higher olefins in a homogeneous catalyst system are weakly illuminated.

It can be noted that in our specific case, we are talking about the synthesis of a dielectric liquid, which places high demands on the purity of the target product, since small impurities in it immediately reduce its practical value. It should be noted that in the publications reviewed, as a rule, processes for preparation of alkylaromatic hydrocarbons mixtures are described, and the problem of products synthesis for electrical purposes has not been solved. One can observe the general regularities of the chloro-methylation reaction of monoalkyl derivatives of lower

homologues of benzene with an increased molecular weight [6]. It was found that the high selectivity of  $ZnCl_2$  achieved in acetic acid solution is explained by the intermediate formation of the compound  $H_2(ZnCl_2(OCOCH_3)_2)$ , which is the true catalyst of the reaction).

The chloroalkylation products yield decreases with increasing side chain length of alkylaromatic hydrocarbons, thus requiring an increased catalyst consumption.

In the electrophilic substitution reaction, to which the chloromethylation reaction relates, the solvent plays an important role. It is established that the reaction rate increases in the series:  $C_2H_5COOH < CH_3COOH < CH_3COSH \leq HCOOH$ , which agrees with the fact that the acidity of the carbonium ion in the given acid sequence is increased. In the interphase processes of solid-liquid systems, tertiary amines, quaternary ammonium salts (HOA), crown ethers, crypts, linear polyesters, diamines; polyamines are used as catalysts [7]. A number of studies are devoted only to the reaction between benzyl chloride and potassium benzoate (or acetate).

### *Experimental part*

During the synthesis of acetoxymethyl–*o*–xylene, the reactive *o*–xylene and hexene-1 grades “chemically pure”, para–forms, acetic acid of the “chemically pure for analysis” brand was used. Sodium acetate was pre-dried at 100 °C. Hydrogen chloride was received from hydrochloric acid, followed by drying over  $H_2SO_4$ . As the catalyst for the alkylation reaction, the first stage of the synthesis of the desired product, both anhydrous aluminum chloride, its dry hydrogen chloride promoted samples and solutions in nitromethane were tested: the catalyst solvent was pre-dried over calcined  $K_2CO_3$  and distilled 92% sulfuric acid was also used. Zinc chloride, used as a catalyst for the chloromethylation reaction (second synthesis step), was pre-dried to a melted form and stored in a desiccator over sulfuric acid; before use, the catalyst pieces were quickly ground and loaded into a reactor. Reagents and solvents necessary for qualitative and quantitative determination of intermediate and final products of acetoxymethyl–hexyl–*o*–xylene–reactive synthesis have been used without additional treatment. Activated  $\gamma-Al_2O_3$  was used to dry the dielectric liquid from current-carrying impurities. The process for the preparation of acetoxymethyl–*sec*.hexyl–*o*–xylene as shown above consists of three stages: alkylation, chloromethylation and acetylation. Reactions were carried out on laboratory units with agitating devices. Alkylation of *o*–xylene with hexene-1 was carried out as follows. The calculated amount of *o*–xylene was loaded into the reaction flask and warmed to the desired temperature. The calculated amount of the catalyst under study was loaded. In the case of using the  $AlCl_3 \cdot CH_3NO_2$  catalyst, the latter was prepared separately by dissolving small portions of  $AlCl_3$  in nitromethane at room temperature. When the reaction was carried out in the presence of an  $AlCl_3 \cdot HCl$  catalyst, the contents of the flask were saturated throughout the experiment with dry hydrogen chloride. Then, the required amount of hexene-1 was fed through the dropping funnel, with constant stirring, for the selected time. After completion of the hexene-1 feed, the reaction mass was further subjected to stirring for 30 minutes. At the end of the experiment, the reaction mixture was cooled to room temperature, followed by the separation of the catalyst.

In the case of using aluminum chloride catalysts, the reaction mixture was washed with water to decompose it, and then neutralized with a 20% solution of  $Na_2CO_3$ , washed with distilled water. The separated catalyst was dried over freshly calcined calcium chloride and accelerated in distilled under vacuum. In the vacuum distillation process, fractions of unreacted reagents were selected, as well as the target of hexyl-*o*-xylene. The fractions were analyzed by gas-liquid chromatography.

Chloromethylation of *sec*.hexyl–*o*–xylene was carried out according to the procedure consisting in the following. A predetermined amount of hexyl–*o*–xylene, freshly prepared anhydrous zinc chloride, perform and glacial acetic acid was charged into the reaction flask. After

the necessary temperature was established with intensive stirring, the calculated amount of hydrogen chloride dried over H<sub>2</sub>SO<sub>4</sub> was started to flow for a predetermined time. To isolate the reaction mass from moist air, the side waste of the flask was connected to the sulfuric acid gate.

The reaction was stopped by pouring the reaction mixture into cold water. The organic layer was extracted with benzene. The benzene extract was washed with water, a weak soda solution; after drying over K<sub>2</sub>CO<sub>3</sub>, benzene was distilled off, the catalyst was distilled and subjected to analysis.

Acetoxylation of chloromethyl-sec.hexyl-o-xylene was carried out as follows. A predetermined amount of sodium acetate and acetic acid was charged into the reaction flask. The necessary temperature was set and, after dissolving CH<sub>3</sub>COONa, chloromethyl-sec.hexyl-o-xylene, dropwise taken in some deficiency with respect to CH<sub>3</sub>COONa, was started, with vigorous stirring. After feeding the entire amount of chloromethyl-sec.hexyl-o-xylene, stirring was continued for one hour. The reaction mass was then cooled, decomposed with water; after neutralization with a 20% solution of soda, the acetoxylation product over K<sub>2</sub>CO<sub>3</sub> was dried, distilled under vacuum and analyzed.

Analysis of the initial raw material and reaction products was carried out by gas-liquid column chromatography. Chromatographic analysis of alkylation products was carried out on a Chrom-5 apparatus with a thermal conductivity detector with a sensitivity threshold of at least 3.10 — 3% (volume). The length of the column is 2 m, the inner diameter is 3–4 mm. The stationary phase Apiezon-L applied on phytochrome (fraction 0.3–0.5 mm). Carrier gas is helium, internal standard-n-dodecane. The temperature of the column is 180 °C, the evaporator is 300 °C, the current is 100 mA, the hydrogen carrier gas consumption is 30 ml/min. Chromatographic analysis of the products of chloromethylation of secondary hexyl-o-xylene was carried out on a chromatograph LXM-8MD with temperature programming, a column 4 mm in diameter filled with a stationary phase of chromotone (0.1–0.125 mm fraction) with an Apisezon-L 15% liquid phase applied and a silicone elastomer E-301 (1%); carrier gas-hydrogen, hydrogen feed rate of 30 ml/min. The temperature in the column is 160 °C, the evaporator is 300 °C, the detector current is 100 mA; n-dodecane was used as an internal standard. Chromatographic analysis of the acetoxymethylation products was carried out on a Chromium-5 chromatograph with a flame ionization detector, a column length of 2 m, and an immobile Apeizon-L phase on an N-AW chromatograph. Carrier gas is hydrogen, hydrogen supply rate is 24 ml/min, air is 500 ml/min, nitrogen is 32 ml per minute; the column temperature is 200 °C.

For carrying out multiserial analyzes of chloromethyl derivatives, they were converted into ethoxymethyl-substituted boiling samples of the sample with KOH solution in ethanol. The value of the relative retention coefficient was determined by the formula:

$$Ki = \frac{m_i \cdot S_{st}}{m_{st} \cdot S_i}$$

where  $m_i$  — is the mass of the component, g;  $m_{st}$  — is the mass of the standard, g;  $S_i$  — area of the peak of the component being determined, mm<sup>2</sup>;  $S_{st}$  — peak area of the standard, mm<sup>2</sup>.

The electrophysical and physicochemical properties of synthesized acetoxymethyl-t-hexyl-o-xylene were determined by standard techniques after vacuum distillation and fine purification from current-conducting impurities by the adsorption method on aluminum oxide. The dielectric dehydration plant was a one-liter flask with a tap for sampling and a glass stopper connected to a three-way valve. A liquid was placed in the flask and a pre-prepared alumina was added to it in

an amount of 10% of the weight of the liquid to be purified. To avoid undesirable contacts of the dielectric with air oxygen, the flask was filled with nitrogen, evacuated, then nitrogen was again fed through a three-way valve. The liquid was mixed with alumina evenly. The closed flask was then disconnected from the vacuum pump and held for several hours at 90 °C.

### Results and discussion

Table 2 presents the material balances of the processes of alkylation of o-xylene with hexene-1 in the presence of various catalysts. The highest selectivity for the target product (secondary hexyl-o-xylene) is observed when the catalysts  $AlCl_3 \cdot CH_3NO_2$  are used, the conversion of the olefin is 100%.

The synthesized sec.hexyl-o-xylene had the following physicochemical parameters:  $T_{\text{boil,point}}$  104–106°C/4 mm mercury column; Elemental composition C — 88.21%, H — 11.32%. (calculated elemental composition C — 88.43%, H — 11.58%). Found: Microscopy — 188; MRfound — 61.9; MR calcul. — 61.8.

Table 2.

THE MATERIAL BALANCE OF THE PROCESS OF ALKYLATION  
 OF O-XYLENE WITH HEXENE-1 IN THE PRESENCE OF VARIOUS CATALYSTS

№	Raw materials, products, indices	Catalyst			
		$H_2SO_4$	$AlCl_3$	$AlCl_3 \cdot CH_3NO_2$	$AlCl_3 \cdot HCl$
		<i>Taken</i>			
1	o-xylene, g	318	318	318	318
2	hexene-1, g	42	42	42	42
3	aluminum chloride, g	—	10	—	6.7
4	92% $H_2SO_4$ , g	12,5	—	—	—
5	nitromethane, g	—	—	28	—
	Total, g	372.5	370	398	366.7
		<i>Received</i>			
6	o-xylene, g	258	271	275	282.8
7	hexene-1, g	—	—	—	—
8	aluminum chloride, g	—	7.2	9.1	5.2
9	92% $H_2SO_4$ , g	5.4	—	—	—
10	nitromethane, g	—	—	26	—
11	sec.hexyl-o-xylene, g	64	57	82	71.25
12	poly-alkyl-o-xylenes, g	21.3	28.8	2.4	6.2
13	losses, g	23.7	6.0	3.5	2.0
14	total, g	372.5	370	398	366.7
15	Olefin conversion, % wt.	100	100	100	100
16	Selectivity for sec.hexyl-o-xylene, % wt.	67.4	56.8	86.2	75
17	Temperature, °C	20	25	50	50
18	o-xylene: hexene-1, mol / mol	6	6	6	6
19	catalyst: hexene-1, mol / mol	0.20	0.15	0.15	0.15
20	reaction time, hour	6.0	4.0	3.0	2.0

Of various modifications of the chloromethylation reaction, we have settled on the procedure for it carrying out in a solution of acetic acid in the presence catalyst of zinc chloride as a highly effective and selective system, applied to alkylaromatic substrates of increased molecular weight. With the help of highly reactive monochloromethyl-alkyl-o-xylenes, highly branched radicals can

be introduced into other compounds, shielding adjacent functional groups, which can reduce the sensitivity of shielded functions to undesirable effects of reagents or the acidic and basic media. In this connection, the process of monochloromethylation of alkyl-o-xylene with increased molecular weight was studied. For the start of the process, the moment of introduction into the reactor at the reaction temperature of the whole portion of the paraform was taken. Chromomethylate samples taken at regular intervals were analyzed by gas-liquid chromatography. In order to obtain acetoxymethyl-sec.hexyl-o-xylene, two modifications of the acetoxylation reaction were investigated (Table 3).

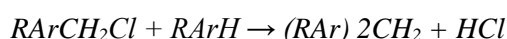
Table 3.

THE MATERIAL BALANCE OF THE PRODUCTION  
 OF ACETOXYMETHYL-SEC.HEXYL-O-XYLENE

Name of compound	In the medium acetic acid				In the conditions of interphase catalysis				Process indices			
	taken		received		taken		received		yield, %	specific productivity, mol / l, hour		
	g	mole	g	mole	g	mole	g	mole		In the medium acetic acid	In the conditions of interphase catalysis	In the medium acetic acid
C <sub>6</sub> H <sub>5</sub> (CH <sub>3</sub> ) <sub>2</sub> (iso-C <sub>6</sub> H <sub>13</sub> )CH <sub>2</sub> Cl	119.25	0.5	11.92	0.05	119.25	0.5	3.7	0.016				
CH <sub>3</sub> COONa	41	0.5	4.1	0.05	41	0.5	1.4	0.02				
CH <sub>3</sub> COOH	100	1.67	82	1.00	—	—	—	—	89.5	96	0.18	0.38
0-C <sub>6</sub> H <sub>4</sub> (CH <sub>3</sub> ) <sub>2</sub>	—	—	—	—	100	0.94	94.7	0.92				
[(C <sub>2</sub> H <sub>5</sub> ) <sub>3</sub> NCH <sub>2</sub> C <sub>6</sub> H <sub>5</sub> ]Cl <sup>-</sup>	—	—	—	—	1	0.04	0.8	0.0035				
C <sub>6</sub> H <sub>2</sub> (CH <sub>3</sub> ) <sub>2</sub> (isoC <sub>6</sub> H <sub>13</sub> )CH <sub>2</sub> OCOCH <sub>3</sub>	—	—	118	0.45	—	—	125.5	0.48				
NaCl	—	—	26.35	0.45	—	—	28.24	0.48				
Losses	—	—	18.55	—	—	—	6.91	—				
Total:	260.25		160.25	—	261.25		261.25	—				

The non-catalytic reaction of the reaction of chloromethyl-sec.hexyl-o-xylene with sodium acetate was carried out in a solution of acetic acid. Catalytic acetoxylation was carried out in o-xylene in the presence of trimethylbenzylammonium chloride — the Makoshi-TEBH catalyst.

The reaction carried out in the solution of o-xylene can be attributed to interfacial processes in the liquid-solid system, since in the temperature range studied 100–130 °C, sodium acetate is only partially soluble in o-xylene. I also wanted to mention a simple but effective method of acetoxylation, the essence of which is as follows. In the second stage, in addition to chloromethyl-sec.hexyl-o-xylene, byproducts are formed-diarylmethanes (R<sub>Ar</sub>)<sub>2</sub>CH<sub>2</sub> and hydrogen chloride is released as a result of the chloromethyl derivatives having a mobile chlorine atom undergoing a partial conversion by reaction:



Distillation is carried out to separate the chloromethylate from the diarylalkanes. At the same time, due to the influence of high temperatures, the rate of the side reaction increases, the amount of by-product increases and the amount of chloromethylate, the desired intermediate, decreases, which lead to decrease in the yield of dielectric liquid. As a result of the conducted studies, it was shown that the separation of diarylalkanes does not require the rectification after the chloromethylation stage. Diarylalkanes are involved together with the desired intermediate product into the third stage, without being converted and having no effect on the course of the acetoxylation reaction. After the third stage, the reaction mixture is distilled to separate the dielectric liquid from the diarylalkanes. Thus, the sequence of technological operations is changed, allowing to reduce the decomposition of the main intermediate product — chloromethylate and, thereby, to increase the yield of the target product (Table 4). The synthesis of the ester is carried out as follows: the first stage of alkylation of *o*-xylene with hexene-1 is carried out in the presence of a solution of aluminum chloride in nitromethane at 500 °C for two hours and a mole ratio of reactants: *o*-xylene: hexene-1: aluminum chloride: nitromethane = 5:1:0.15:0.45.

Table 4.

DEPENDENCE OF MATERIAL BALANCES OF INDIVIDUAL STAGES OF PRODUCTION  
 OF A SUBSTITUTE FOR CASTOR OIL — A SEQUENCE OF TECHNOLOGICAL OPERATIONS

Alkylation			Chloromethylation			Acetoxylation		
component name	% of mass	g	component name	% of mass	g	component name	% of mass	g
<i>With distillation in stage III</i>								
C <sub>6</sub> H <sub>4</sub> (CH <sub>3</sub> ) <sub>2</sub>	61.8	790	RArH	22.5	109	(RAr) <sub>2</sub> CH <sub>2</sub>	3.54	21
Oligomers	3.13	40	(CH <sub>2</sub> O) <sub>n</sub>	2.9	14	RArH	18.40	109
CH <sub>3</sub> NO <sub>2</sub>	5.06	68.7	R(Ar) <sub>2</sub> CH <sub>2</sub>	4.33	21	(CH <sub>2</sub> O) <sub>n</sub>	2.36	14
RArH	30	380	H <sub>2</sub> O	4.87	23.7	H <sub>2</sub> O	4.0	23.7
			RArCH <sub>2</sub> Cl	65.4	317.6	CH <sub>3</sub> COONa	2.0	12
						NaCl	10.8	64
						RArCH <sub>2</sub> OCOCH <sub>3</sub>	58.9	349
<i>Without distillation in stage II</i>								
C <sub>6</sub> H <sub>4</sub> (CH <sub>3</sub> ) <sub>2</sub>	61.8	790	RArH	22.5	91	CH <sub>3</sub> COONa	8.53	32
Oligomers	3.13	40	(CH <sub>2</sub> O) <sub>n</sub>	2.9	21	NaCl	14.67	55
CH <sub>3</sub> NO <sub>2</sub>	5.06	68.7	R(Ar) <sub>2</sub> CH <sub>2</sub>	4.33	83	RArCH <sub>2</sub> OCOCH <sub>3</sub>	76.8	288
RArH	30	380	H <sub>2</sub> O	4.87	20.12			
			RArCH <sub>2</sub> Cl	65.4	262.2			

After washing and neutralizing with 20% sodium carbonate solution, the reaction mixture is distilled to separate the alkylate from unreacted *o*-xylene and the products of the polymerization of hexene-1. Alkylate boils out at 102–104 °C / 3 mm. In the second stage, a mixture of paraform and dry gaseous hydrogen chloride in the presence of zinc chloride in glacial acetic acid for 4 hours at 600 °C is used to prepare chloromethyl–hexyl–*o*-xylene on the obtained hex–*o*-xylene.

Molar Ratio of Reactants: alkylate: paraform: chloride zinc = 1:0.6:0.22. The hydrogen chloride feed rate is 100 ml/min. After separation of the organic layer, washing and neutralization with 20% sodium hydrogen carbonate solution and drying are followed.

The third stage of acetylation of chloromethyl–*t*-hexyl–*o*-xylene is carried out with acetic acid sodium in acetic acid for 5 hours at 120 °C. The reaction mixture was washed, neutralized with 20% sodium hydrogen carbonate solution, dried, distilled in vacuum to separate from unreacted alkylate and diarylalkanes. The fraction 160–165 °C/4 mm mercury Colum was taken.



As can be seen from Table 4, the distillation in the chloromethylation stage leads to the formation of a significant amount of diarylalkanes and the yield of the dielectric liquid is 72.3%. When diarylalkanes are involved in the acetoxylation stage, the decomposition of chloromethylate is prevented under the influence of high temperatures and the yield of the dielectric liquid is increased to 87.7%. In Table 5 for comparative evaluation, material balances and dielectric liquid outlets calculated to convert alkylate during distillation in the second and third stages, respectively, are presented. In Table 6 compares the electrophysical characteristics of castor oil and acetoxymethyl-sec.hexyl-o-xylene. The traditional solvent used in this process, acetic acid serves as a homogenizer of the system, which facilitates warming, speeds up the reaction.

Table 5.

MATERIAL BALANCE OF RECEIVED CASTOR OIL SUBSTITUTE

<i>Alkylation</i>			<i>Chloromethylation</i>			<i>Acetoxylation</i>		
<i>component name</i>	<i>ta-ken, g</i>	<i>received, g</i>	<i>component name</i>	<i>ta-ken, g</i>	<i>received, g</i>	<i>component name</i>	<i>ta-ken, g</i>	<i>received, g</i>
C <sub>6</sub> H <sub>4</sub> (CH <sub>3</sub> ) <sub>2</sub>	318	262	RArH	80.75	23.35	RArH	23.35	20
C <sub>6</sub> H <sub>12</sub>	42	—	(CH <sub>2</sub> O) <sub>n</sub>	13.7	3.6	R(Ar) <sub>2</sub> CH <sub>2</sub>	4.22	3
AlCl <sub>3</sub>	20	1.65	HCl	25.12	9.81	RArCH <sub>2</sub> Cl	60.7	—
CH <sub>3</sub> NO <sub>2</sub>	27.45	25	ZnCl <sub>2</sub>	13.8	12.2	CH <sub>3</sub> COONa	28.6	6.8
dialkylbenzenes		10.24	CH <sub>3</sub> COOH	100	85	CH <sub>3</sub> COOH	100	92
RArH		80.75	H <sub>2</sub> O	—	4.85	RArCH <sub>2</sub> OCOCH <sub>3</sub>	—	69.0
losses		10.76	R(Ar) <sub>2</sub> CH <sub>2</sub>	—	4.22	NaCl		14.9
alkylate yield	%	85.0	RArCH <sub>2</sub> Cl		60.7	losses		3.8
			losses		24.3	The yield of chloromethylate, %		92.0
			The yield of chloromethylate on converted alkylbenzene, %		84.0	The yield on the taken olefin, %		51.0
						The yield of the converted olefin, %		51.0

Table 6.

COMPARATIVE CHARACTERISTICS OF THE ELECTROPHYSICAL PROPERTIES OF CASTOR OIL AND ACETOXYMETHYL-SEC.HEXYL-O-XYLENE

<i>Indices</i>	<i>Castor oil</i>	<i>Acetoxymethyl-sec.hexyl-o-xylene</i>
Boiling point, °C	313	335–340
Degree of refraction	1.475	1.5032
Sodification point, °C	–15	–42
Flashpoint, °C	285	162
Density, kg/sm <sup>3</sup>	0.960	0.965
The dielectric constant:		
by room temperature	4.65	4.5
by 90 °C, after 30 minutes		4.3
by 90 °C, after 48 hours		4.3
The loss tangent of a dielectric	0.002	0.002
by 90 °C, after 30 minutes	—	0.026
by 90 °C, after 48 hours	—	0.022

### Conclusions

Thus, it can be considered that the most expedient method for carrying out the third stage of receives the dielectric liquid of acetoxymethyl–sec.hexyl–o–xylene is acetylation in the presence of an interfacial catalyst, triethyl–benzyl ammonium chloride, a Makoshi catalyst in an o–xylene medium. Electrophysical indices of liquid acetoxymethyl–sec.hexyl–o–xylene, obtained by technology with distillation of the reaction mixture in the chloromethylation stage and by technology with distillation in the acetoxylation stage, do not change. By carrying out the reaction under the conditions of interphase catalysis, it is possible to increase the yield of the key product, eliminate acidic waste (recycle o-xylene recycles), abandon the use of sufficiently scarce acetic acid, and increase the specific productivity of the reactor. The received product obtained is not inferior to the electro physical characteristics of a standard dielectric liquid.

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