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**ABSTRACT**

of the dissertation for the degree of Doctor of Science

**DEVELOPMENT OF DEAROMATIZATION AND  
DESULFURIZATION PROCESSES FOR DISTILLATES OF  
OIL AND FUELS BY IONIC-LIQUID SOLVENTS**

Speciality: 3303.01 - Chemical Technology and Engineering

Field of science: Technical sciences

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**Baku - 2024**

The work was performed in the laboratory of “Polifunctional monomers and oligomers” of the Institute of Petrochemical Processes named after Y.H.Mammadaliyev of Ministry of Science and Education Republic of Azerbaijan

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## GENERAL DESCRIPTION OF THE WORK

**Relevance of the topic and the degree of elaboration.** Given the environmental problems that have arisen at the moment, there is increasing interest in research aimed at developing chemical processes that meet the principles of green chemistry, that is, based on the use of reagents that do not pollute the environment and are harmless to living organisms and produce waste products that can be disposed of.<sup>1</sup>

In this regard, the identification of eco-friendly ionic liquids that can be used on as a solvent medium, extractant, catalytic system or component of a catalytic system in many processes an industrial scale is very urgent problem.<sup>2</sup>

Currently, one of the stages used in the production of high-quality oils and fuels for various purposes is the process of selective purification of the distillate from unnecessary components, including aromatic hydrocarbons, sulfur and resinous compounds, and for this purpose, along with the processes of hydrotreating and hydrogenation in industrial the method of extraction with various organic solvents is also widely used.

Among the listed methods, in comparison with the hydrotreating method, which requires the use of large amounts of hydrogen, expensive catalysts and the process under conditions of high temperatures and pressure, the use of an extraction purification process, which is carried out under mild conditions and is based on simple technology, is of greater interest. However, at present, it is important to replace used as solvents in the extraction process corrosive, toxic and characterized by low selectivity solvents - phenol, furfural, morpholine, N-methylpyrrolidone, sulfolane and other solvents with easily regenerated ionic liquids, characterized by high solubility and selectivity and allowing to create more efficient, eco-friendly, and is the focus of attention of specialists, scientists and engineers working in this field.

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<sup>1</sup> <https://bigenc.ru/c/zelionaia-khimia-91655e>

<sup>2</sup> G.M.J. Al Kaisy, M.I.A.Mutalib, M.A.Bustam, J.M.Leveque, N.Mohammad. Liquid-Liquid extraction of aromatics and sulfur compounds from base oil using ionic liquids // Journal of Environmental Chemical Engineering, vol. 4, issue 4, part. A, 2016, p.4786-4793

Recent literature has widely covered research on the synthesis of ionic liquids, various fields of application, including solvent media, and their use as selective solvents in extraction processes, and review articles and books published in this area confirm this.

Considering the above, research carried out in the field of synthesis of ionic-liquid salts suitable for use in oil refining processes, selective purification of oils and fuel fractions from unnecessary components at the last stage, identification of promising compositions as extractants are relevant and has theoretical and practical significance.

This interest in ionic liquids is due to their complex of unique properties. Thus, ionic-liquid compositions have high solubility of various classes of compounds, low vapor pressure, high thermal and chemical stability, as well as other properties.

**The object and subject of the research.** The object of the ongoing research is the removal of unnecessary components from the composition of oil and fuel fractions for various purposes using the method of ion-liquid extraction. The subject of the study is to determine the conditions for the selective purification of oil and fuel distillates using ionic liquids that differ in cationic and anionic fragments and synthesized on the basis of formic and acetic acids as extractants.

**Goals and objectives of the study.** The purpose of the dissertation work is determined by the development of selective purification conditions for removing unnecessary components from the composition of oil and fuel fractions for various purposes, meeting modern and future requirements, through a simple, economically and environmentally effective extraction method. For this purpose, based on a comparative study of the physicochemical parameters of the raffinate obtained during the purification of petroleum fractions, it is necessary to conduct research in the field of determining effective ionic liquids differing in anionic or cationic fragment as extractants, synthesized using morpholine, N-methylpyrrolidone, mono-, di-, triethylamine and butylamine as the amine component and formic and acetic acids.

To achieve this goal, the following problems were solved:

- synthesis of various ionic liquids based on formic and acetic acid, study of their composition and structure by IR and NMR spectral analysis;
- determination of the effective composition of the ionic liquid in the selective purification of Naftalan oil using the method of ion-liquid extraction;
- determination of the effective composition of the ionic liquid and optimal conditions for the selective purification of transformer oil distillate by the method of ionic-liquid extraction;
- selective purification of petroleum distillates with different viscosity index at a temperature of 100°C by ion-liquid extraction;
  - purification of kerosene fractions, differing in boiling point and the amount of aromatic hydrocarbons in the composition, by the method of ionic liquid extraction;
  - purification of a mixture of diesel distillates, differing in the amount of aromatic and sulfurous compounds - straight diesel fraction, hydrotreated diesel distillate, a mixture of straight diesel fraction with products of secondary oil processing - light gas oil catalytic cracking and coking in their different ratios by the method of liquid extraction;
  - studying the possibilities of regeneration and reuse of compositions of ionic liquids used as extractants.

**Research methods:** The research carried out in the dissertation work is based on the use of IR,  $^1\text{H}$  and  $^{13}\text{C}$  NMR, UV spectroscopy methods, determining the indicators of distillates taken as raw materials, as well as raffinates and extracts obtained as a result of selective purification using the extraction method using standard methods. The analyzes were carried out in accordance with the requirements of the relevant GOCT.

#### **The main provisions of the defense.**

- for the first time, the selective purification of kerosene fractions differing in the content of aromatic hydrocarbons with various ionic liquids was studied, the effective composition of the ionic liquid and optimal extraction conditions were determined;
- the influence of various factors, including the composition of the ionic liquid, on the process of selective purification of transformer

oil distillate and the quality indicators of the resulting raffinate was studied, and the advantages of this method were determined in comparison with the process of the acid-contact purification method;

- in the process of Naftalan oil distillate extraction, the effectiveness of synthesized ionic liquids of various compositions was determined, the conditions for almost complete dearomatization were established when using ionic liquids as extractants- N-methylpyrrolidoneacetate and diethylamino-formate;
- selective purification of hydrotreated diesel distillates with a boiling point of 185-364°C and straight-run diesel distillates with a boiling point of 191-350°C was studied and the conditions for almost complete dearomatization were determined;
- the conditions for the extraction of mixtures of diesel distillates obtained as a result of compounding secondary oil products - light catalytic cracking and coking gas oil with ionic liquids synthesized on the basis of formic and acetic acid;
- the conditions for the purification of petroleum distillates with a viscosity index of 7.8 or 18 mm<sup>2</sup>/s at a temperature of 100°C, as well as mixed distillates obtained by compounding them using the ion-liquid extraction method, were determined;
- the effectiveness of selective purification of oil and fuel fractions by the method of ionic-liquid extraction was studied and demonstrated in comparison with the process of extracting them with an organic solvent N-methylpyrrolidone.

#### **Scientific novelty of the research:**

- based on the research carried out, the processes of selective purification of oil and fuel fractions for various purposes using ionic-liquid compositions were studied and effective purification methods were developed, which differ from existing purification methods in terms of environmental safety and waste-free nature.
- in comparison with the existing acid-contact method of purification of transformer oil distillate, purification conditions have been developed using an environmentally effective, waste-free ionic-liquid extraction method;

- the process of selective purification of Naftalan oil fraction boiling in the range of 200-450°C with ionic liquids - N-methylpyrrolidoneacetate and diethylaminoformate - has been studied, and a practically high degree of dearomatization of the distillate has been established;
- the prospect of ionic-liquid purification of straight-run and hydrotreated diesel distillates has been identified and the effective ionic-liquid composition has been determined;
- the conditions for the regeneration of each ionic liquid used as a selective solvent are disclosed, as well as the possibility of its reuse with the same effect;
- the cost of diesel fuel was determined as 331.01 AZN, obtained in the process of extraction purification of diesel distillate - a mixture of straight-run diesel fraction and secondary oil products - light catalytic cracking and coking gas oil at their mass ratio of 70:15:15, respectively.

#### **Theoretical and practical significance of the research.**

- study of selective purification of oils and fuels taken as raw materials, differing in the number of components that need to be extracted, using the method of ion-liquid extraction;
- study of the influence of various factors - extraction time and temperature, the ratio of components - distillate and extractant - on the process of selective purification of distillates for various purposes studied as raw materials;
- study of the influence of anion-cation combination in an ionic-liquid extractant on the efficiency of the extraction process;
- determination of the effective composition of the ionic liquid in the process of selective purification of various petroleum distillates;
- determination of the effective composition of the ionic liquid in the process of selective purification of various fuel distillates by extraction method;
- calculation of the economic efficiency of the process of ionic-liquid extraction purification of mixed diesel distillate obtained by compounding straight-run diesel fraction and products of secondary origin - light catalytic cracking and coking gas oil at

their mass ratio of 70:15:15%, respectively.

**Approbation of application.** Materials related to the topic of the dissertation are reflected in 39 scientific works, including 21 articles and 18 theses materials. 7 of them were published in foreign and domestic journals with a high impact factor, included in the «Web of Science» and «Scopus» databases. The results of the research work carried out were presented at the following republican and international conferences and symposiums and published in conference proceedings:

"Naghiyev Readings" International Conference dedicated to Academician Murtuza Naghiyev's 110th anniversary (Baku, 2018); 12th International Multidisciplinary Scientific and Technical Conference "Advances in Science and Technology" (Moscow, 2018); International Scientific Conference "Current problems of modern natural sciences" (Azerbaijan, Ganja, 2018); International Scientific and Practical Conference dedicated to the 100th anniversary of Academician Vahab Aliyev (Baku, 2018); International Scientific Conference "Actual problems of modern chemistry" dedicated to the 90th anniversary of academician Y.H. Mammadaliyev Institute of Petrochemical Processes (Baku, 2019); Baku State University, the XIII International scientific conference "Actual Problems of Chemistry" dedicated to the 96th anniversary of the birth of national leader Heydar Aliyev (Baku, 2019); Dedicated to the 96<sup>th</sup> Anniversary of the National leader Azerbaijan Heydar Aliyev "III International scientific conference of young researchers" (Baku, 2019); Институт Ядерной Физики им. Б.П. Константинова (Rusiya, Sankt-Peterburq, 2020); V - Международной научно-практической конференции (Rusiya, Ufa, 2021); The 17th International Conference on "Technical and Physical Problems of Engineering" (Türkiyə, İstanbul, 2021); International Scientific Conference "Current problems of modern natural sciences" dedicated to the 99th anniversary of the birth of national leader Heydar Aliyev (Azerbaijan, Ganja, 2022); International conference "Modern problems of theoretical and experimental chemistry", dedicated to the 90th anniversary of the birth of academician Rafiqa Aliyeva (BSU, 2022); 87th Scientific and Technical Conference of faculty, researchers and



graduate students (Minsk, 2023); Scientific conference “Petrochemistry, synthesis of polyfunctional monomers, oligomers and polymers” dedicated to the 90th anniversary of the birth of professor Sahib Aliyev (Baku, 2023); Republican scientific conference "Chemistry and Chemical Technology" dedicated to the 100th anniversary of the birth of national leader Heydar Aliyev (Baku, 2023);; Scientific conference “Metal complex and organometallic catalysis, synthesis and research of (co)oligomers and (co)polymers” dedicated to the 80th anniversary of academician Akif Azizov (Baku, 2023).

**Author's personal participation.** The applicant was the responsible executor of all stages of work related to setting problems, conducting experimental studies, analyzing the results obtained, as well as preparing theses, articles and dissertations.

**The organization where the dissertation work was performed.** The submitted dissertation reflects the researches carried out according to the scientific research work plan of IPCP of ANAS (State registration No. 0106 A2 0017, 2016-2023 years).

**The scope and structure of the work.** Dissertation work is 273 pages of computer printout, consists of introduction, 4 chapters, conclusions and bibliography including 306 references. Dissertation is 328212 characters long, excluding 46 pictures, 72 tables and literature list.

The introduction substantiates the relevance of the research, reflects the purpose of the work, the main provisions being defended, and based on the results obtained, the scientific novelty and practical significance of the research is formulated.

**In the first chapter,** research work was carried out, methods for improving the quality indicators of oil and fuel fractions for various purposes, including well-known scientific works published in the literature on purification using the liquid-liquid extraction method, and also analyzed the advantages of the extraction method, in particular the ion-ion method liquid extraction. This literature review covers 92941 characters.

**The second chapter** presents the physicochemical parameters of the used in the research samples of oil and fuel distillates for various purposes, the rules for purification of other reagents used, information

on the synthesis and composition of ionic liquids used as extractants, covering 19486 characters.

**The third chapter** reflects research carried out in the field of purification of undesirable components - aromatic hydrocarbons, sulfur and resinous compounds by the method of ionic-liquid extraction from petroleum distillates for various purposes, in particular Naftalan oil distillate, transformer oil distillate and oil distillates with a viscosity index of 7.8 and 18 mm<sup>2</sup>/s at a temperature of 100°C and mixtures obtained by compounding them in various proportions, covering 83071 characters.

**The fourth chapter** reflects the results of selective purification of various diesel distillates, in particular hydrotreated diesel distillate, straight-run diesel distillate and mixed diesel distillates obtained by compounding this distillate with oil refining by-products - light catalytic cracking and coking gas oil, the effective ionic liquid and optimal extraction conditions were determined. Based on economic calculations, the prospects of the process of ionic-liquid extraction purification of mixed diesel distillate are shown in comparison with the purification process with N-methylpyrrolidone, and the general technological flow diagram of the process is also shown. This chapter covers 112040 characters

**At the end** of the dissertation, the scientific results obtained as a result of the research work performed, a list of cited literature and a list of abbreviated terms are presented.

## MAIN CONTENT OF THE WORK

The rapid development of technology and the introduction of new technologies are constantly tightening the requirements for the quality indicators and performance properties of the base oils and fuels used. Taking this into account, in order to protect the environment, the development of new, simple, cost-effective and green chemical technologies for the purification of oil and fuel fractions for various purposes is required.

Among the existing traditional methods for purifying petroleum fractions, the liquid-liquid extraction method is easy to perform at low temperature, and the solvent used as an extractant can be recovered

and reused.

In order to improve the quality indicators of oils and fuels, the development of processing methods is aimed at improving existing technologies, identifying new highly selective environmentally friendly solvents that ensure the production of base oils and fuels with high yield and quality.

One of the promising solutions to this problem is the development of an extraction method based on the use of environmentally friendly ionic liquids that comply with the principles of “green chemistry” as a selective solvent in the process of purifying oil and fuel fractions from undesirable components. In the conducted studies, when purifying petroleum and fuel distillates taken as raw materials, the method of ionic-liquid extraction used ionic liquids synthesized on the basis of formic and acetic acids, characterized by a combination of cationic and anionic fragments, including morphoinformate (MF), N-methylpyrrolidoneformate (NMPF), diethylamineformate (DEAF), dibutylamineformate (DBAF), morpholineacetate (MA), N-methylpyrrolidoneacetate (NMPA).

The synthesis of ionic liquids was carried out by the reaction of acid and amine components at a temperature of 50-60°C and a contact time of 2-3 hours; their structure was confirmed by IR and NMR spectral analysis.

Selective purification of petroleum distillates for various purposes has been carried out, in particular Naftalan oil distillate with a boiling point of 200-450°C, transformer oil distillate with a viscosity index of 7.5 and 18 mm<sup>2</sup>/s at 100°C and their mixtures in various ratios, optimal extraction conditions and effective ionic-liquid composition were determined.

### **Selective purification of Naftalan oil distillate using ionic-liquid extraction**

It is known that Naftalan oil (NO) is the natural wealth of Azerbaijan, which has no analogues in the world, and has been used in the treatment of many diseases since ancient times. Previously, this oil in its natural form was used for medicinal purposes, but later, from the 60s of the 20th century, research began in the field of removing carcinogenic compounds from its composition in order to improve its

medicinal properties and expand its areas of application.

In studies of the purification of naftalan oil distillate by extraction method, dearomatization of the distillate with a boiling point of 200-450°C, the amount of aromatic hydrocarbons was 17.0% % mass (ГОСТ 6994-74), the amount of sulfur compounds was 423 ppm, and the density at 20°C was 914. 6 kg/m<sup>3</sup>, refractive index 1.4994, color index 8.0, was carried out using NMPA and DEAF as extractants.

In order to determine the optimal conditions for the process of selective purification of Naftalan oil, the influence of various factors on the yield and degree of dearomatization of the raffinate, including the ratio of ionic liquid and naphthalan oil distillate, extraction temperature, contact time of components and the number of extraction stages, was studied. The results obtained are shown in Table 1.

It has been established that under similar conditions, with an increase in the mass ratio of the ionic liquid to the raw material from 1:1 to 4:1, the degree of dearomatization of naphthalan oil increases from 41.2% to 76.5%, while the yield decreases from 89.2% mass to 68.3% mass. With a sixfold excess of ionic liquid relative to the distillate of Naftalan oil, the degree of dearomatization of the raffinate is 82.3 wt%.

**Table 1**

**Effect of the the ratio of components on degree of dearomatization of Naftalan oil (extraction temperature -40°C, extraction time -1s.)**

İL:distillate ratio, % mass	Yield, mas. %		Indicators of raffinate		
	raffinate	extractant	Amount of aromatic hydrocarbons, % mass	Removal rate of aromatic h/c, % mass	$n_D^{20}$
1:1	89,2	9,2	10,0	41,2	1,4875
2:1	88,0	9,6	6,0	64,7	1,4866
3:1	77,0	20,9	5,0	70,6	1,4756
4:1	73,67	25,4	4,0	76,5	1,4611
6:1	68,3	30,7	3,0	82,3	1,4562

The influence of extraction temperature on the yield of raffinate and the degree of removal of aromatic hydrocarbons in a threefold excess of ionic liquid relative to the distillate, with a contact time of

components of 1 hour, was studied and it was determined that the residual amount of aromatic hydrocarbons in samples of raffinates obtained at relatively high extraction temperatures (60°C and 80°C), almost the same (8.0-8.5% mass), the degree of dearomatization is 50.0%, 52.9% mass, respectively.

At the next stage of the study, the process of step-by-step purification of the naftalan oil fraction of NMPA was studied at different temperature conditions and different ratios of components and, based on the research carried out, the conditions for complete dearomatization of the oil fraction were determined (Table 2).

**Table 2**

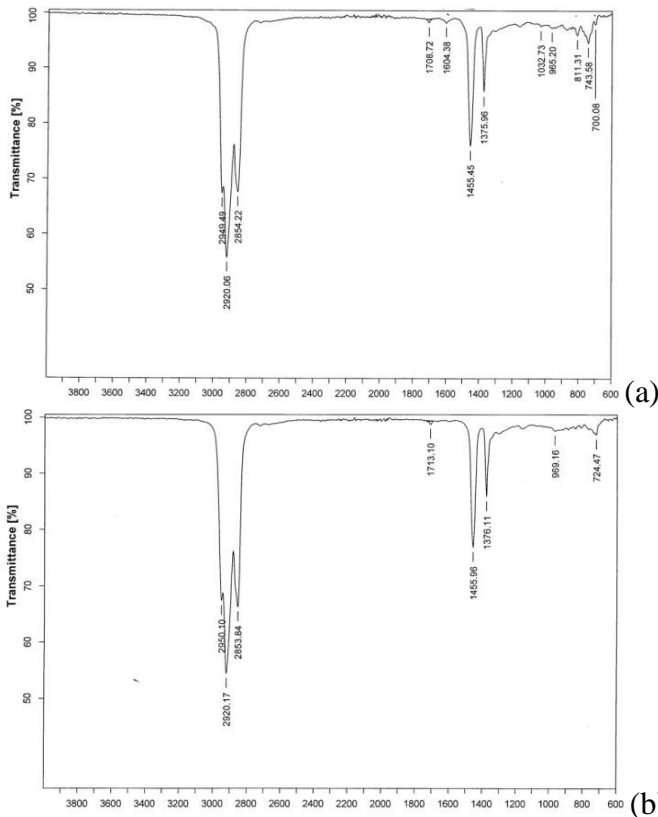
**The influence of various factors on the process of stepwise selective purification of Naftalan oil using the ion-liquid extraction method**

step extraction	Extraction conditions			yield of extract, %	Indicators of raffinate			
	IL:distillate ratio, % mass	T, °C	Extraction time, h		yield, mas.%	Amount of aromatic hydrocarbons, %	Removal rate of aromatic h/c, % mass	$n_D^{20}$
In two stages								
I	1:1	40	1,0	10,0	89,2	10,0	41,2	1,4875
II	1:1	40	1,0	13,2	86,8	8,0	52,9	1,4825
I	4:1	40	2,0	27,0	72,2	4,0	76,6	1,4618
II	2:1	40	2,0	41,9	57,23	2,0	88,2	1,4410
I	4:1	40	2,0	27,0	72,2	4,0	76,6	1,4622
II	3:1	40	2,0	30,0	69,3	0,6	96,5	1,4482
I	1:1	60	1,0	13,2	86,1	11,0	35,3	1,4330
II	1:1	60	1,0	15,1	85,4	8,0	52,9	1,4825
In four stages								
I	2:1	40	1,0	9,2	88,0	6,0	64,7	1,4786
II	2:1	40	1,0	19,8	76,3	4,0	76,6	1,4620
III	2:1	40	1,0	25,1	73,5	2,0	88,2	1,4493
IV	2:1	40	1,0	35,2	62,9	0,0	100	1,4416

Thus, when carrying out the extraction process at the first stage with a 4-fold, and at the second stage with a 3-fold excess amount of

ionic liquid relative to the raw material (with a contact time of 1 hour at each stage), the residual amount of aromatic hydrocarbons in the raffinate is 0.6% mass, the degree of dearomatization of the resulting raffinate is 96.5% mass. When carrying out the selective purification process at each stage with a double amount of extractant, almost complete removal of carcinogenic aromatic hydrocarbons from the composition of the Naftalan oil fraction is achieved at the fourth stage of extraction.

The composition of the feedstock and raffinate obtained by two-stage extraction purification was studied using IR-, UV- and NMR-spectral analysis methods and the experimental results were confirmed.



**Fig. 1. IR spectrum of naftalan oil (a) and raffinate (b), obtained as a result of its purification by ion-liquid extraction.**

Thus, the IR spectra of the raffinate sample obtained by a two-stage ion-liquid extraction method and the distillate are characterized by identical absorption bands. It is important to note that, in contrast to the IR spectrum of the raw material, absorption bands (700, 811, 1604  $\text{cm}^{-1}$ ) characteristic of aromatic hydrocarbons are not observed in the spectrum of the raffinate.

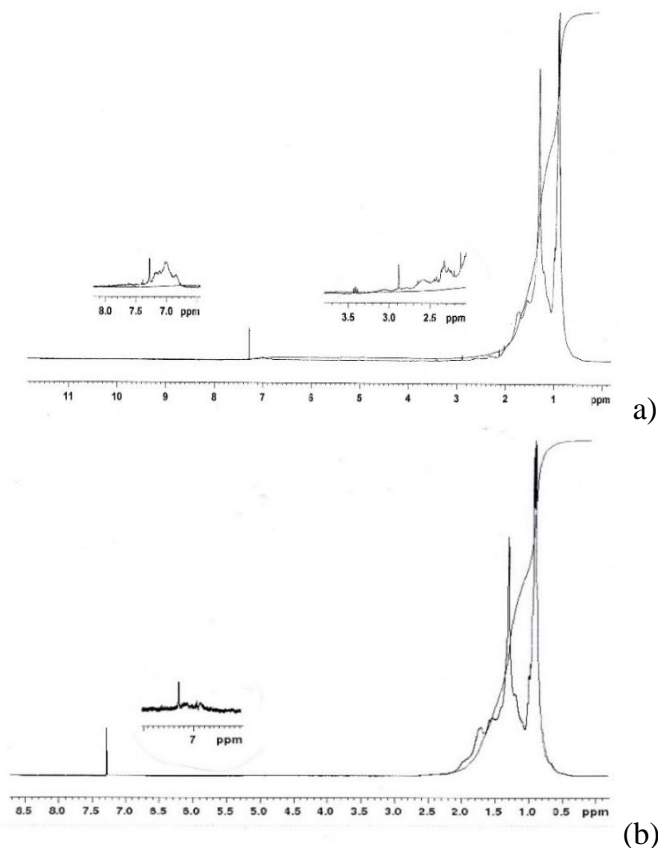
According to the results of the UV-spectral analysis (Table 3), the residual amount of aromatic hydrocarbons in the raffinate, obtained during the one-stage purification of Naftalan oil with an ionic liquid, is 3.2% mass, and during the two-stage purification - 0.44% mass, including the amount of derivatives benzene - 0.12% mass, naphthalene - 0.11% mass, phenanthrene - 0.21% mass.

**Table 3**

**Structural and group composition of Naftalan oil purified by NMPA**

Sample	Molecular mass	Amount of aromatic hydrocarbons in raffinate, % mas.				
		Benzene derivatives	Naphthalene derivatives	Phenanthrene derivatives	Anthracene derivatives	Total
Naftalan oil, raw materials	289	7,2	6,1	2,4	1,4	17,1
I stage, IL:NO 4:1 mass ratio	252	1,7	1,01	0,5	traces	3,21
II stage, IL:NO 3:1 mass ratio	266	0,12	0,11	0,21	-	0,44

Spectral analysis of  $^{13}\text{C}$  NMR of the naphthalan oil fraction and a raffinate sample obtained by purification by ion-liquid extraction in two stages showed that the signals reflecting the protons of the aromatic ring in the region are very weakly noticeable, only with increasing spectrum intensity  $\sigma = 7.4$  m. h. (Figure 2).



**Fig. 2.  $^{13}\text{C}$  NMR spectra of naftalan oil (a) and raffinate (b) obtained by two-stage ion-liquid extraction purification.**

Thus, the possibility of the process of deep dearomatization of naphthalan oil using NMPA ionic liquid as a selective solvent has been established, and the optimal conditions for the resulting raffinate with a yield of 69.3% by weight have been determined:

- mass ratio of ionic liquid and naftalan oil fraction at the 1st stage – 4:1 by mass;
- at the 2nd stage – 3:1 by mass;
- Extraction temperature - 40°C;
- Extraction time – 1 hour at each stage.

The extraction process of the naphthalan oil fraction was also carried out using DEAF as a selective solvent, the influence of various



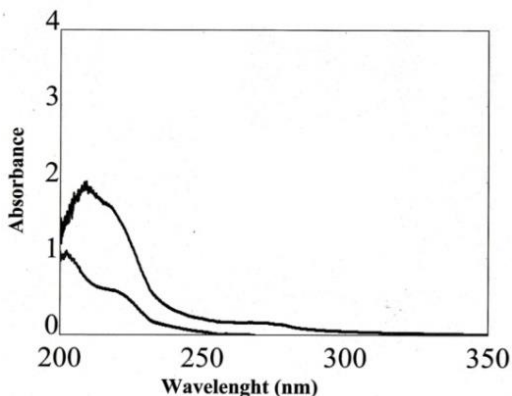
factors was studied and the conditions for complete dearomatization were determined, carrying out the process at room temperature with a twofold excess of ionic liquid. (Table 4).

**Table 4**

**Influence of various factors on the process of dearomatization of naphthalan oil using DEAF ionic liquid**

iL:distillate ratio, % mass	T, °C	Extraction time, min	Yield of raffinate, %	yield of extract, %	Amount of aromatic hydrocarbons, % mass	$n_D^{20}$
2:1	60	60	61	39	5,0	1,4849
3:1	60	120	66,8	23,2	0,0	1,4725
3:1	40	120	69,8	30,8	0,0	1,4656
2:1	25-30	60	88,0	22,0	0,0	1,4637

It has been shown that the use of DEAF-containing ionic liquid as an extractant in the process of purifying naftalan oil from aromatic hydrocarbons and resinous compounds is more effective and makes it possible to develop an environmentally friendly technology. The results obtained were also confirmed by UV spectral analysis (Fig. 3).



**Fig. 3. UV spectrum of naftalan oil purified in two stages with diethylaminoformate**

It has been established that the residual amount of aromatic hydrocarbons in the raffinate obtained by extraction with DEAP ionic

liquid is 0.5%, and derivatives of naphthalene, phenanthrene and anthracene are completely removed from the composition during selective purification.

Thus, it has been established that it is possible to almost completely remove aromatic hydrocarbons from the distillate of naftalan oil when using NMPA and DEAF ionic liquids as extractants.

In the process of purifying petroleum distillates of various viscosities using the ion-liquid extraction method, along with obtaining base oils with improved performance properties in high yields, the identification of effective selective solvents is of great importance.

In this regard, given modern environmental problems, replacing organic solvents used as extractants with environmentally friendly ionic liquids is of interest for the development of new effective technologies.

The process of purification of petroleum distillate isolated from low-paraffin oils and having a viscosity index of 311.63 mm<sup>2</sup>/s at 40°C, 18.0 mm<sup>2</sup>/s at 100°C and a viscosity index of 46.65 using DEAF ionic liquid as a selective solvent has been studied. The influence of various factors - the ratio of components, temperature and duration of extraction on the extraction process was studied and with an equal mass ratio of components, an extraction time of 1 hour and a temperature of 60°C, a raffinate was obtained with a yield of 89.6% mass, characterized by kinematic viscosity of 103 and 10.0 mm<sup>2</sup>/s at temperatures of 40°C and 100°C, respectively, and the viscosity index is 22.25 points higher than that of the feedstock (68.9 mm<sup>2</sup>/s).

At a temperature of 80°C, when using a twofold excess of extractant, the raffinate yield is 87.2% mass, and with a threefold amount of ionic liquid extractant relative to the distillate, 85% mass. The viscosity index of the obtained raffinate samples does not differ significantly and is 69.3 and 70.0, respectively.

At the next stage of research, selective purification of oil distillate obtained from low-paraffin oils with a viscosity index of 7.5 mm<sup>2</sup>/s at a temperature of 100°C was carried out, using DEAF ionic liquid as an extractant at an equal mass ratio of components, extraction temperature 60°C. C and a contact time of 1 hour, and it was shown

that the resulting oil with a yield of 61.8 mass at temperatures of 40°C and 100°C has a kinematic viscosity of 64.4 mm<sup>2</sup>/s and 7.4 mm<sup>2</sup>/s, respectively and viscosity index 65.

In order to expand the raw material base of AK-15 oil, selective purification of mixtures obtained by compounding petroleum distillates with a viscosity index of 7.5 and 18.0 mm<sup>2</sup>/s at a temperature of 100°C in a mass ratio of 1:2, 1:1 and 2:1 was studied, respectively, with DEAF ionic liquid and it was shown that the best result is achieved with a mass ratio of components in the distillate mixture of 1:2. The resulting raffinate with a yield of 92.8% by weight is characterized by a characteristic viscosity index of 110.5 mm<sup>2</sup>/s at a temperature of 40°C, 10.5 mm<sup>2</sup>/s at a temperature of 100°C, a viscosity index of 69.9 and a relatively high thermal stability.

**Table 6**

**Physico-chemical parameters of raffinate samples obtained from the purification of mixed petroleum distillates using the ion-liquid extraction method**

Properties	Mass ratio of oil distillates in a mixture with a viscosity index of 7.5 and 18 mm <sup>2</sup> /s at 100°C		
	1:2	1:1	2:1
Yield, % mass			
raffinate	92,8	96,4	90,0
extract	7,2	3,6	10,0
Raffinate indicators:			
kinematic viscosity, mm <sup>2</sup> /s			
at 40°C	110,5	98,0	68,6
at 100°C	10,5	9,6	7,76
Viscosity index	69,9	66,4	68,4
Freezing temperature, °C	23	20	21
Flash temperature, °C	220	217	210
Density, 20°C, g/cm <sup>3</sup>	0,903	0,901	0,902
After oxidation:			
Kinematic viscosity, at 100°C mm <sup>2</sup> /s	11,8	10,4	11,1
Viscosity increase, %	1,3	1,0	1,1
Ash content, wt. %	there is not	0,01	0,01
Amount of coke, mass. %	1,14	-	1,11
Amount of sediment, mass. %	0,8	1,4	0,96

The conditions for obtaining purified oils with viscosity index 73-76.4 in the process of selective purification of oil distillate with a kinematic viscosity index of 7.6-8.0 mm<sup>2</sup>/s at a temperature of 100°C using ionic liquids - MF, NMPF and NMPA - as extractants were determined.

It is shown that a relatively high yield is achieved when using the MF ionic liquid extractant. The raffinate obtained with a yield of 96.4% is characterized by a viscosity index of 67.0.

A relatively high degree of desulfurization is achieved when using NMPA as an extractant. In this case, the residual amount of sulfur in the raffinate obtained with a yield of 93% is 0.0623%.

The raffinate obtained through a step-by-step extraction process with a yield of 95.5% of the mass was characterized by: kinematic viscosity at 40°C - 68.61 mm<sup>2</sup>/sec, at 100°C - 7.75 mm<sup>2</sup>/sec, viscosity index - 68,7.

### **Ionic-liquid extraction purification of transformer oil distillate**

To ensure stable operation in transformer systems, it is recommended to use high performance oils. However, it should be taken into account that the hydrocarbon group composition, physicochemical indicators, and complex operational properties of oils obtained from oils extracted from different fields differ, and these indicators depend on the selective purification method used in production.

At the «Azerneftiyag» refinery, the base product of transformer oil is obtained by selective purification of fractions of low-paraffin oils boiling in the temperature range of 300-400°C, followed by hydrotreating, or by an acid-contact purification method based on the use of 92-97% sulfuric acid as the main reagent.

This method is based on multi-stage processing of raw materials with sulfuric acid and neutralization of the resulting distillate with alkali, washing with water and drying using white clay as an adsorbent. However, this method is characterized, along with the production of waste and wastewater, by a decrease in oil yield as a result of the partial transition of the necessary components into the acid resin.

Recent environmental problems and the constant tightening of requirements for the performance properties of insulating oils, like other oils, have led to the development of environmentally friendly, promising extraction methods using readily available, thermally and chemically stable reagents as extractants.

In our studies, transformer oil distillate was purified using DBAA ionic liquid as a selective solvent and in order to determine the conditions for obtaining base oil with improved quality indicators, the extractant was mixed with raw materials in various mass ratios (1-3:1), temperature conditions were studied (60 , 70, 80°C) and different contact times of the components (1, 2 and 3 hours).

An increase in the selectivity of the extraction process with a threefold amount of ionic liquid was established by increasing the temperature from 40°C to 80°C and it was shown that under optimal temperature conditions, at 60°C, the residual amount of aromatic hydrocarbons in the raffinate obtained with a yield of 90.5% of the mass is 9.8% (Table 7).

**Table 7**

**Dependence of the degree of purification when using DBAA ionic liquid on the extraction temperature**

Extraction temperature, °C	Yield of raffinate, %	Content of aromatic hydrocarbons % mass	Degree of dearomatization, % mass	Raffinate		
				Acid number, mg KOH/g	$d_{20}^4$ kq/m <sup>3</sup>	$n_D^{20}$
40	96,0	13,0	13,34	0,5	885,0	1,4800
60	90,5	9,8	32,41	0,02	882,5	1,4760
80	78,06	11,0	24,14	2,1	883,8	1,4780

The transparency of the color of the distillate indicates that resinous compounds that have a negative effect on its quality indicators have also been removed from the composition. On the basis of the obtained results, in the process of cleaning transformer oil with DBAA ionic liquid, it is recommended to under the following conditions extraction:

- mass ratio of extractant to distillate - 3:1;
- extraction temperature – 60°C;
- extraction time - 3 hours.

The results of IR and UV spectral analysis of purified transformer oil under the specified conditions once again confirmed the results obtained. In the IR spectrum of the raffinate obtained by extraction purification of oil distillate, the intensity of absorption bands reflecting bending vibrations of the C-H bond of the benzene ring noticeably decreases (Table 8)..

**Table 8**

**Results of UV spectral analysis of transformer oil distillate and raffinate purified by DBAA ionic liquid**

Substance name	Molecular mass	The concentration of aromatic hydrocarbons, % mass				
		benzene derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
Transformer oil distillate	267,5	6,4	8,2	5,2	1,05	20,85
Raffinate	150,5	2,2	4,1	2,9	traces	9,2

The results of UV spectral analysis indicate that during the extraction process, the amount of aromatic hydrocarbons in the distillate decreased by ~2.27 times, and the residual amount of aromatic hydrocarbons in the resulting raffinate was 9.2% mass.

Some physicochemical parameters of the raffinate sample obtained from the extraction of transformer oil distillate DBAA with ionic liquid were determined and it was established that it complies with the basic requirements of ГOCT 982-80 (Table 9).

**Table 9**

**Physico-chemical parameters of transformer oil before and after purification by ion-liquid extraction**

Properties of transformer oil	Before purification	Selectively purified		ГOCT 982-80
		IL-MF	IL-DBAA	
Density, 20°C, kg/m <sup>3</sup>	885,6	884,1	882,6	Should not be exceed 885.0
kinematic viscosity, at 50°C mm <sup>2</sup> /s	7,75	6,78	6,7	Should not be exceed 8.0

## continuation of Table 9

Acid number, mg KOH/g	-	neutral	neutral	Should not be exceed 0,01
Color using the UHT method	4,0	1,0	1,0	Should not be exceed 1,5
Flash temperature in a closed crucible, °C	145	138	140	should not be less 135
Freezing temperature, °C	-53	-53	-53	-45
$n_D^{20}$	1,4880	1,4840	1,4760	-

At the next stage of research, the process of selective purification of distillate of transformer oil DEAF with ionic liquid was carried out. The influence of the extraction temperature on the process of purification with a three-fold excess of the extractant and a contact time of 2 h was investigated, and it was established that at a temperature of 60°C, the obtained raffinate with a yield of 96.5% , characterized by the residual amount of aromatic hydrocarbons - 9.4% mass against 14.5% mass in the original raw material (table 10).

**Table 10**

### Dependence of the extraction process on temperature

Extraction temperature, °C	Yield of raffinate, %	Content of aromatic hydrocarbons % mass	Degree of dearomatization, % mass	Acid number, mg KOH/g	$d_{20}^4$ kg/m <sup>3</sup>	$n_D^{20}$
40	96,4	12,0	14,6	0,43	885,0	1,4800
60	91,5	9,4	35,0	0,01	882,6	1,4760
80	79,1	10,0	31,3	1,8	883,0	1,4780

When the extraction temperature increased to 80°C, the solubility of the contained ionic liquid increased, the raffinate yield decreased to 79.1% mass, and it was shown that the purified transformer oil does not meet the requirements of GOST 982-80 for a number of qualitative indicators. This indicates that in the process of high-temperature ionic-liquid extraction from the distillate, along with unnecessary components, necessary components are also removed.

When using a fourfold excess of the extractant relative to the distillate, a decrease in the selectivity of the ionic-liquid extraction process and the yield of the raffinate is observed (82.4%), and the residual amount of aromatic hydrocarbons in the resulting raffinate is relatively large and amounts to 10.2% mass (Table 11).

**Table 11**

**Dependence of the extraction process on the ratio of components**

IL:distillate ratio, % mass	Yield of raffinate, %	Content of aromatic hydrocarbons % mass	Degree of dearomatization, % mass	$d_{20}^4$ kg/m <sup>3</sup>	$n_D^{20}$
1:1	94,0	12,0	17,2	884,5	1,4800
2:1	92,3	10,9	24,83	882,6	1,4780
3:1	91,5	9,4	35,82	882,6	1,4760
4:1	82,4	10,2	29,6	-	-

When the contact time of the components increases from 1 hour to 3 hours, the raffinate yield partially decreases and is 91.5% mass versus 95.2% mass.

Thus, based on research carried out in the field of purification of transformer oil distillate by the method of ionic-liquid extraction, when using DEAF as an extractant, it is proposed to carry out the process: mass ratio of ionic-liquid extractant and distillate - 3: 1, extraction temperature - 60°C and contact time of components is 3 hours.

Transformer oil purified under the specified conditions meets the requirements of ГOCT 982-80 and has a kinematic viscosity of 6.7 mm<sup>2</sup>/s at a temperature of 50°C, an flash temperature of 140°C and a freezing point of minus 48°C.

Optimal conditions for the selective purification of transformer oil distillate with an ionic liquid containing NMPA have been established: mass ratio IM:TOD - 2:1, extraction temperature - 60°C; contact time of components is 2.0-2.5 hours. The physico-chemical parameters of the transformer base oil obtained with a yield of 81.15% were determined, and it was found that it complies with ГOCT requirements, at a temperature of 90°C it is characterized by a dielectric loss tangent angle of 0.41 after 1 hour of treatment with 5%



mass with clay at a temperature of 120°C (Table 12).

**Table 12**

**Physico-chemical parameters of samples of transformer oil  
distillates purified by ion-liquid extraction**

Properties	Purificated transformer oil			Transformer oil distillate	ГОСТ 982-80
	NMPA	DBAA	DEAF		
Density, 20°C, kg/m <sup>3</sup>	878,0	882,6	885,0	885,9	Should not be exceed 885
kinematic viscosity, mm <sup>2</sup> /s					
at 50°C-d <sub>50</sub>	6,81	6,7	6,7	7,17	Should not be exceed 8,0
at 30°C-d <sub>30</sub>	9,95	-	-	7,75	Should not be exceed 11
$n_D^{20}$	1,4780	1,4750	1,4760	1,4880	
Acid number, mg KOH/g	0,0	0,0	0,01	3,8	Should not be exceed 0,01
Flash temperature in a closed crucible, °C	137	140	140	145	should not be less 135
Freezing temperature, °C	-52	-53	-48	-53	-45
Color using the IQHT method	1,0	1,0	1,0	4,0	Should not be exceed 1,5
Amount of low molecular weight volatile acids, mg/KOH per 1 g of oil	0,02	-	-	-	0,04
Clarity at 50°C	transparent	-	-	-	transparent
Dielectric loss tangent, at 90°C	0,41	-	-	-	Should not be exceed 0,5
Amount of mechanical impurities, %	there is not	-	-	there is not	olmamalı
Natrium sample, score	0,19	-	-	-	0,4
Amount of water,%	0,005	-	-	-	0,005
Sediment, %	there is not	-	-	-	there is not

The results obtained show that raffinate samples obtained as a result of selective purification of transformer oil distillate using ionic liquids as extractants meet the basic requirements of ГОСТ 982-80.

A comparative study of the group hydrocarbon composition of

the raffinate obtained by purification by the ion-liquid extraction method with the group hydrocarbon composition of the raffinate obtained by the acid-contact purification method showed that transformer oil purified by the ion-liquid extraction method contains 93.8% mass naphthene-paraffin hydrocarbons and 6.2% mass of aromatic hydrocarbons (Table 13).

**Table 13**

**Group hydrocarbon composition of transformer oil purified by ion-liquid extraction**

Group hydrocarbon composition	Transformer oil distillate		Purified by acid contact method		Purified by ionic liquid extraction	
	yield, %	$n_D^{20}$	yield, %	$n_D^{20}$	yield, %	$n_D^{20}$
Naphten-paraffin hydrocarbon	88,25	1,4795	92,5	1,4868	93,8	1,4731
Aromatic hydrocarbons:						
I group	4,54	1,5000	4,1	1,5240	0,3	1,4894
II group	5,04	1,5835	3,4	1,5320	5,9	1,4985
III group	0,26	1,5900	-	-	-	-
Resinous compounds	>1,5900	1,91	-	-	-	-
Total:	100	-	100	-	100	-

Thus, the results obtained when purifying transformer oil distillate by extraction using various ionic-liquid compositions indicate that this method is effective compared to the acid-contact method and has a number of advantages:

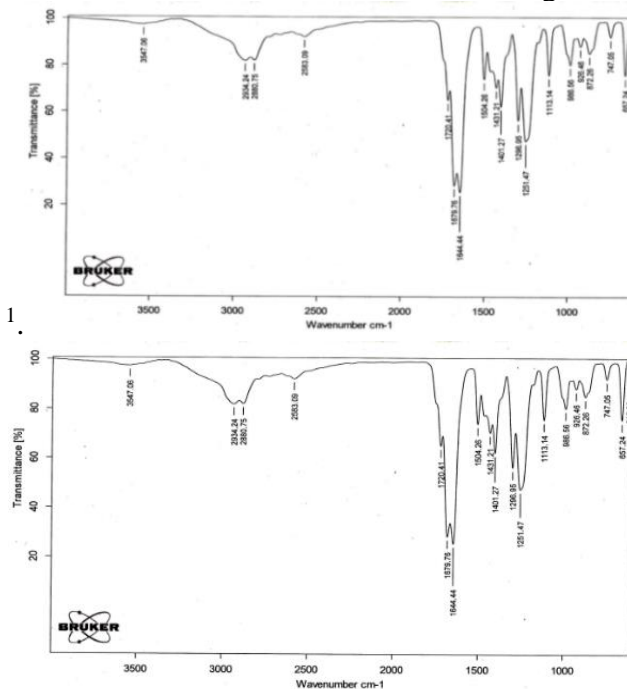
- the raffinate yield with the ionic-liquid extraction method (81.2-85% by weight) is significantly higher than with the acid-contact method (76.7% by weight);
- the amount of unnecessary components (aromatic hydrocarbons) extracted from the distillate is higher (44.68-47.23%) compared to the acid-contact method (36.17% of the mass), that is, the degree of purification of the distillate increases;
- ionic liquids used in the extraction method can be easily regenerated and reused;
- in the process of ionic-liquid extraction, waste (acidic resins and wastewater) that pollute the environment is not generated;
- the equipment used in the extraction process is not subject to corrosion, unlike the acid-contact method.

The regeneration of ionic liquid extractants used in the process of selective purification was carried out, and the possibility of their regeneration and reuse with the same effect was established.

An IR- and NMR- spectral analysis of the ionic liquid studied by NMPA was carried out before and after regeneration and it was found that this ionic liquid practically did not undergo any changes. Since the IR spectrum of both samples is characterized by identical absorption bands:

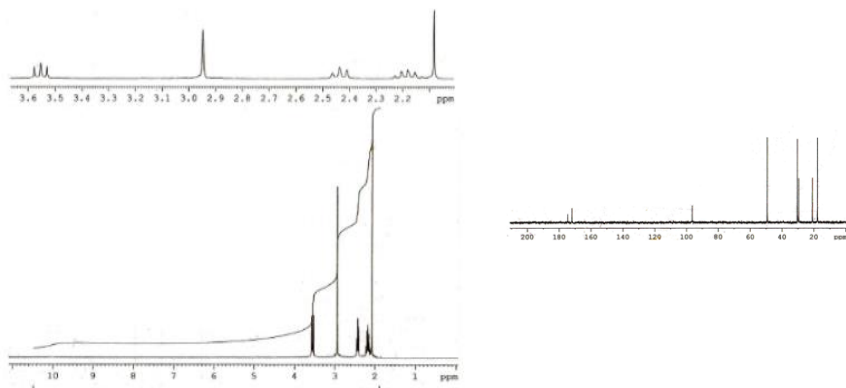
- absorption band characteristic of the C=O fragment in the ester group –  $1720\text{ cm}^{-1}$ ;
- absorption band characteristic of the C=O group in the cationic fragment –  $1679\text{ cm}^{-1}$ ;
- absorption band characteristic of the N-H bond in the cationic fragment -  $1644\text{ cm}^{-1}$ ;

absorption band characteristic of the ammonium fragment -  $2583\text{ cm}^{-1}$

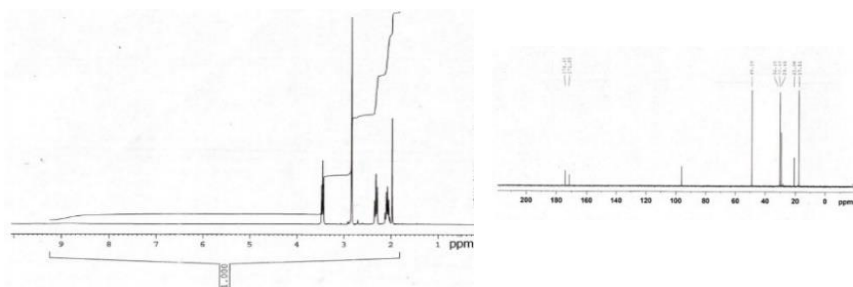


**Fig. 5. IR spectra of NMPA ionic liquid before (a) and after regeneration (b)**

Both samples are characterized by  $^1\text{H}$  NMR spectra peaks of proton-specific signals in the  $\text{CH}_3$ ,  $\text{CH}_2\text{O}$  and  $\text{N-CH}_2$  fragments, and  $^{12}\text{C}$  NMR spectra peaks corresponding to carbon-specific signals— $\text{O-C-CH}_3$  (30.479 ppm),  $\text{N-CH}_3$  (29.447 ppm), in the pyrrolidine fragment  $\text{N-CH}_2$  (49.289 ppm),  $\text{C=O}$  (171581 ppm) and in the ester group (174445 ppm) (Fig. 6).



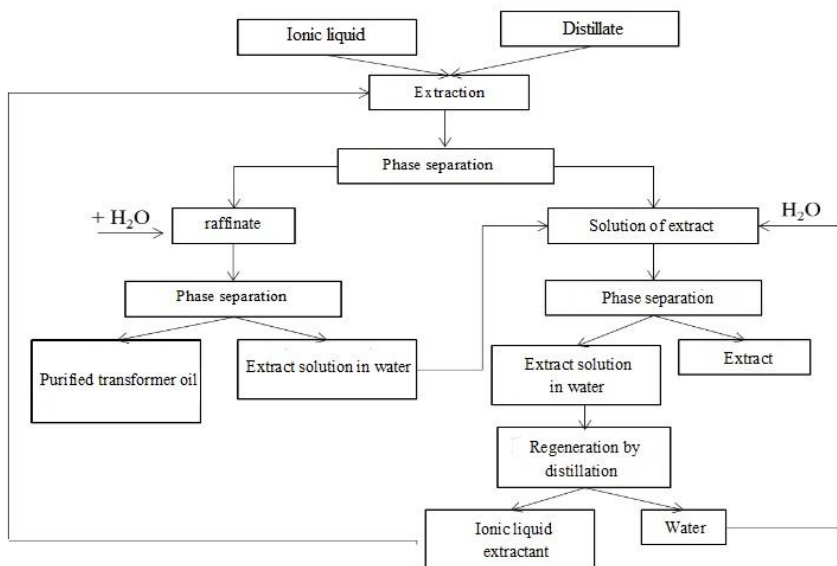
(a)



(b)

**Fig. 6. NMR spectra of N-methylpyrrolidone acetate ionic liquid before use (a) and after regeneration (b)**

Considering that selective purification of transformer oil distillate DBAA, DEAP and NMPA with ionic liquids is carried out in a similar way, the basic diagram of the extraction process with ionic liquid can be presented as follows:



**Fig.7. The principle technological scheme of the transformer oil distillate purification process using the ion-liquid extraction method.**

Based on the studies conducted, it was established that the ionic-liquid method of extraction of transformer oil distillate is safe from an environmental point of view. This, in turn, makes it possible to replace the acid-contact method used in industry for purifying transformer oil distillate with an ionic-liquid extraction method that meets the requirements of “green chemistry.”

### **Selective purification using ion-liquid extraction of various compositions of fuel**

The conducted studies examined the selective purification of samples of kerosene and diesel distillates that differ in the content of aromatic hydrocarbons and sulfur compounds using the method of ion-liquid extraction.

### **Selective purification of kerosene fractions using ion-liquid extraction**

It is known to remove aromatic hydrocarbons from the kerosene fraction by hydrotreating and the acid-contact method, as well as liquid-liquid extraction using various organic solvents (sulfolane,

phenol, morpholine, furfural, N-methylpyrrolidone, etc.).

In the research, were used as raw materials three kerosene fractions with boiling points in the range of 136-240°C (S1), 145-250°C (S2) and 185-250°C (S3), containing 12%, 16.5% and 17% by weight of aromatic hydrocarbons, respectively. The yield of raffinate obtained during a one-stage process of extraction of kerosene fractions of NMPA with ionic liquid, at room temperature (22-25°C), mass ratio of ionic liquid and raw material 2.5:1, contact time of components 2 hours is 81.2-86,4% mass, residual amount of aromatic hydrocarbons 1.0-2.0% mass.

The process of stepwise selective NMPA with an ionic liquid was carried out under similar conditions using at each stage an equal mass amount of components and a contact time of 1 hour and it was found that, depending on the number of stages, the yield of the raffinate was 81.0-87.3% mass, the residual amount of aromatic hydrocarbons is 1-4% mass (Table 14).

**Table 14**

**Dependence of the degree of dearomatization of the kerosene fraction of NMPA by ionic liquid on extraction conditions**

Kerosene fraction	IL:distillate ratio, % mass	Number of stages	Extraction time, h	Yield of raffinate, %	Indicators of raffinate		
					$n_D^{20}$	$d_4^{20}$	residual amount of arom/hydroc.
Sample 1	2,5:1	I	2,0	86,4	1,4350	0,772	1,0
	1:1	II	1,0	87,3	1,4355	0,7715	-
	1:1		1,0	85,0	1,4348	0,7709	1,0
Sample 2	2,5:1	I	2,0	82,1	1,4363	0,7730	2,0
	1:1	II	1,0	85,0	1,4365	0,7718	4,0
	1:1		1,0	81,0	1,4353	0,7709	2,0
	1:1	III	1,0	84,3	1,4361	0,7720	2,0
	1:1		1,0	81,0	1,4350	0,7700	-
Sample 3	2,5:1	I	2,0	81,2	1,4364	0,7755	2,0
	1:1	II	1,0	85,3	1,4358	0,7771	6,0
	1:1		1,0	82,1	1,4340	0,7760	4,0

The process of selective purification with a kerosene fraction was carried out at different mass ratios of components in one stage or in stages, and it was shown that with a fourfold excess of ionic liquid, the raffinate obtained with 85.0% mass yield contains 2.0% mass of aromatic compounds (degree of dearomatization 83.33% mass). When the selective purification process is carried out step by step at the first stage - with an equal mass ratio of ionic liquid and kerosene, at the second and third stages - with a double amount of ionic liquid, and a contact time of 1 hour at each extraction stage, the residual amount of aromatic hydrocarbons in the raffinate obtained with the yield 86.16% mass, is 1.5% mass, the degree of dearomatization - 87.5% mass.

The process of extraction purification of kerosene fractions was carried out at a temperature of 25°C in one stage and in stages with MF, MA and DEAA ionic liquids in comparison with the industrially used extractant N-methylpyrrolidone (Table 15).

**Table 15**

**Dependence of the degree of dearomatization of the kerosene fraction with a boiling point of 136-240°C on extraction conditions**

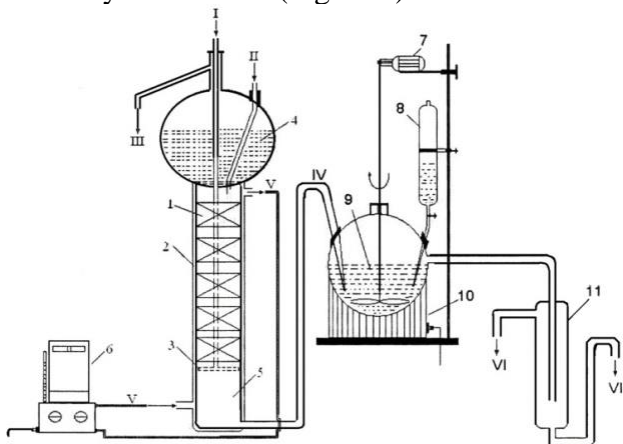
Ionic liquid	İL:distillate ratio, % mass	Extraction time, h	Yield of raffinate, %	Indicators of raffinate		
				$n_D^{20}$	$d_4^{20}$	residual amount of arom/hydroc.
MF	1:1	1,0	95,3	1,4421	-	-
	1:1	1,0	91,7	1,4395	0,7980	2,0
	1:1	1,0	88,67	1,4380	0,7970	0,0
MA	1:1	1,0	94,0	-	-	-
	1:1	1,0	91,5	1,4382	0,7980	2,0
	1:1	1,0	88,1	1,4378	0,7970	0,0
	3:1	1,0	89,4	1,4390	0,7995	3,0
	3:1	2,0	88,1	1,4380	0,7980	2,0
DEAA	1:1	1,0	96,6	1,4420	-	-
	1:1	1,0	92,37	1,4395	0,8010	4,0
	1:1	1,0	88,92	1,4385	0,7980	2,0

It has been established that in the process of extraction of the kerosene fraction using MF as a selective solvent, when carrying out the process in one stage, with a threefold amount of ionic liquid in

relation to the raw material, the contact time of the components is 1 hour, the residual amount of aromatic hydrocarbons in the raffinate obtained with a yield of 89, 4% mass is 3.0% mass, and with a contact time of the components of 2 hours it was 2% mass, the yield is 88.1% mass.

Based on the research carried out, the prospects for selective purification of the kerosene fraction using the ionic-liquid extraction method have been established and it has been determined that with stepwise extraction using an MF or MA ionic liquid as an extractant, aromatic hydrocarbons are practically completely removed from the raw material and the yield is 88.67% and 88. 1% mass, respectively.

Based on these results, the process of continuous selective purification of the kerosene fraction (S1) with a boiling point of 136-240°C using MA ionic liquid as an extractant was studied on a flow-through laboratory installation (Figure 8).



**Fig. 8. Continuously operating laboratory installation for selective purification of oil fractions**

- 1 – reactor filled with Rasig rings;
- 2 – heating;
- 3 – distribution collector;
- 4 – raffinate collection area;
- 5 – Extract solution precipitation zone;
- 6 – thermostat

Streams:

- I – kerosene fraction
- II – ionic liquid extractant
- III – the resulting raffinate
- IV – the resulting extract solution
- V – system heating fluid



When carrying out the extraction process in a multi-flow laboratory installation, the ionic liquid is supplied to the extractor from the upper part, filled with Razig rings, and the kerosene fraction taken as a raw material is supplied from the lower part. The extractant supplied from the upper part of the reactor passes through a glass pipe and enters the lower part of the reactor, mixes with the kerosene fraction taken for purification, and this mixed mixture enters the working area of the reactor and begins to rise through the extractor. This ensures uninterrupted contact between the components and selective purification of the kerosene fraction with ionic liquid.

The rate of supply of the ionic liquid and kerosene fraction to the extractor is adjusted so that the components in the working area are in contact for 2.5 hours. These conditions correspond to the optimal conditions determined on the basis of the research: mass ratio of the ionic liquid extractant to the kerosene fraction - 3:1, extraction temperature 25°C, contact time of components – 2 hours.

After the completion of the extraction process, that is, when the mixture of components reaches the upper part of the extractor, the separation of the purified kerosene fraction from the extract solution is observed. Thus, since the specific weight of the MA ionic liquid used as an extractant (1.135 g/cm<sup>3</sup>) is higher than the specific weight of the kerosene fraction taken as a raw material (0.796 g/cm<sup>3</sup>), the obtained raffinate is collected on the upper layer of the extract, consisting of components removed from the kerosene fraction are merged into the extractor and collected in the lower layer. As a result, during the entire period of extraction, the contact of the extractant - ionic liquid, fed from the top of the reactor, and the kerosene fraction, fed from the bottom of the reactor and moving upwards.

The influence of the ratio of components on the degree of dearomatization of the raffinate obtained in a flow system was studied, and with a threefold excess of the extractant, almost complete dearomatization of the kerosene fraction was achieved (Table 16). The results of UV spectral analysis of kerosene fractions studied as raw materials show that the kerosene fraction, boiling in the temperature range 145-250°C, compared with other kerosene fractions, is richest in aromatic hydrocarbons, including monocyclic (9.41% mass) and

bicyclic (6.13% mass) aromatic hydrocarbons. It was found that a sample of kerosene with a boiling point in the range of 136-240°C was characterized by a low content of anthracenes (0.02% mass) (Table 17).

**Table 16**

**Influence of the amount of ionic-liquid extractant on the process of aromatization of the kerosene fraction in a flow system**

IL:distillate ratio, % mass	Yield of raffinate, %	Yield of extract, %	Content of aromatic hydrocarbons % mass	Degree of dearomatization, % mass	Indicators of raffinate	
					$d_4^{20}$	$n_D^{20}$
2:1	96,5	4,5	6,5	45,9	0,790	1,4423
2,5:1	92,1	7,9	3,0	75,0	0,783	1,4390
3:1	88,1	11,9	0,65	94,8	0,783	1,4380
4:1	87,3	12,7	0,60	95,0	0,780	1,4380

**Table 17**

**Group hydrocarbon composition of the used kerosene fractions and raffinates obtained as a result of their selective purification by ion-liquid extraction**

Kerosene samples obtained by selective purification	Molecular mass	The concentration of aromatic hydrocarbons, % mass				
		benzene derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
Sample 1	153,0	6,13	5,7	1,2	0,02	13,22
Purified at I stage	147,0	1,1	0,42	0,15	-	1,67
Purified at II stage	144,0	0,41	0,17	0,04	-	0,62
Sample 2	134,4	9,41	6,13	2,62	1,04	19,2
Purified at I stage	134,0	0,44	0,31	0,1	-	0,76
Purified at II stage	133,5	0,8	0,9	0,1	-	1,5
Purified at III stage	133,5	0,5	0,4	-	-	1,0
Sample 3	139,0	8,33	5,76	2,1	1,12	17,31
Purified at II stage	138,7	1,0	0,05	0,11	-	1,17

The raffinate obtained as a result of two-stage selective purification of the kerosene fraction with a boiling point of 136-240°C with NMPA is characterized by a low residual amount of aromatic hydrocarbons (0.62% mass).

The raffinates obtained during the purification process with a kerosene fraction (S2) with a boiling point in the range of 145-250°C in one stage with a double amount of ionic liquid and in three stages with an equal amount of ionic liquid and raw materials, do not contain hydrocarbons of the anthracene and phenanthrene series.

The results of UV spectral analysis of the raffinate obtained as a result of three-stage purification of the kerosene fraction with a boiling point of 185-250°C using the ion-liquid extraction method show that the residual amount of aromatic hydrocarbons is 1.17% mass, and three-ring aromatic hydrocarbons - anthracenes - are completely removed from the composition.

Thus, the determination by UV- spectral analysis of the group hydrocarbon composition of raffinates obtained in the process of selective purification of kerosene fractions by extraction with NMPA ionic liquid analysis in comparison with kerosene fractions taken as raw materials showed a decrease in the amount of aromatic hydrocarbons from 13.2-17.3% mass to 0.62-1.17% mass.

At the next stage of research, the results of selective purification of the kerosene fraction (S1) with a boiling point range of 136-240°C by extraction method using MF, DEAA and NMPF ionic liquids as extractants were once again confirmed by UV spectral analysis of the group hydrocarbon composition of raffinates (table 18).

According to the results of UV spectral analysis, the residual amount of aromatic hydrocarbons in the raffinate obtained using MF and DEAA ionic liquids as extractants is 0.76% and 0.99% of the mass, respectively, and the degree of dearomatization is 94.25% and 92.51 % mass. The raffinate obtained by purification with a threefold excess of NMPA ionic liquid contains 3.51% mass of aromatic hydrocarbons and practically consists of monocyclic aromatic hydrocarbons.

Table 18

**Group hydrocarbon composition of raffinates obtained as a result of selective purification of MF, DEAA and NMPP with ionic liquids**

Kerosin nümunələri	Molecular mass	The concentration of aromatic hydrocarbons, % mass				
		benzene derivatives	naphthalenes	Phenanthrenes	Anthracenes	Total
Kerosene fraction	159,0	6,3	5,7	1,2	0,02	13,22
with MF at III stage	156,5	0,55	0,21	0,002	-	0,76
with DEAA at I stage	-	2,25	1,52	0,93	-	4,7
at III stage	155,4	0,65	0,33	0,01	-	0,99
with NMPP at I stage	156,9	3,51	-	-	-	3,51
at III stage	157,1	4,1	0,01	-	-	4,11

IR- spectral analysis of raffinates obtained by selective purification indicates a decrease in the intensity of absorption bands ( $1607\text{ cm}^{-1}$ ), reflecting the C-H bond of the benzene ring.

The calculated spectral coefficients of raffinates obtained as a result of selective purification of the studied kerosene fractions MF and DEAA using ionic liquids using the extraction method are shown in Table 19.

Table 19

**Group hydrocarbon spectral coefficients of the kerosene fraction and raffinates obtained by purification using the ion-liquid extraction method**

Samples	P%	N%	A%
Kerosene fraction	47,83	39,13	13,04
Raffinate obtained as a result of two-stage purification of DEAA	60,61	33,33	6,06
Raffinate obtained as a result of three-stage purification of DEAA	65,62	31,32	3,13
Raffinate obtained as a result of two-stage purification of MF	62,5	31,25	6,25
Raffinate obtained as a result of three-stage purification of MF	67,46	29,32	3,22

here: P% - the amount of paraffin hydrocarbons

N% - amount of naphthenic hydrocarbons

A% - amount of aromatic hydrocarbons

A study of the group hydrocarbon kerosene fractions obtained as a result of selective purification with DEAA and MF ionic liquids based on the results of IR- spectral analysis shows a relative increase in the amount of paraffin hydrocarbons.

Thus, the results obtained during the selective purification of the studied kerosene fractions by the method of ionic-liquid extraction show that the use of ionic liquids synthesized on the basis of formic and acetic acids as a selective solvent is effective.

The advantage of the ionic-liquid extraction method is determined by the simplicity of the process, the possibility of regenerating the used ionic liquid and reusing it as an extractant with the same effect. In addition, the ionic-liquid extraction method for the purification of kerosene fractions eliminates the environmental problems that arise using on an industrial scale the acid-contact method of purification, in particular non-recycled waste.

### **Selective purification of diesel distillates of various compositions using ion-liquid extraction**

The development of the oil refining industry is determined by an increase in the depth of oil refining, as well as an increase in the volumes and quality indicators of commercial oil products, oils and fuels for various purposes.

Currently, motor fuels, including diesel fuel, occupy a special place among the petroleum products produced. Thus, the rapid growth of vehicle fleets (agricultural machinery, trucks, railway transport, etc.) has led to an increase in the production of diesel fuel.

In order to improve the quality indicators of diesel fuel, it is necessary to strictly control the amount of aromatic hydrocarbons in the fuel, especially polycyclic aromatic hydrocarbons, sulfur and tar compounds, since the combustion products of these compounds in diesel engines are extremely toxic and lead to environmental pollution.

In our studies, we used hydrotreated, straight-run diesel distillates, as well as mixed diesel distillates obtained by compounding straight-run diesel distillate with products of secondary oil refining - light gas oil from catalytic cracking and coking at their mass ratio of

70:30 and mixing all three compositions of distillates at their ratio of 70 :15:15, accordingly, the processes of distillate purification by the method of ion-liquid extraction were systematically studied, and the conditions for obtaining diesel fuel of improved quality were determined.

### **Purification of hydrotreated diesel distillate using ion-liquid extraction**

Recently, to obtain diesel fuel of improved quality, preference is given to a combined purification method, that is, purification of hydrotreated diesel distillate by extraction.

The experiments investigated the extraction purification of hydrotreated diesel distillate with a boiling point range of 208-342°C, containing 16% aromatic hydrocarbons, determined by the sulfonation method, 479 ppm of sulfur compounds, with DBAA ionic liquid, at room temperature (20-25°C), contact time 1 hour, at different mass ratios of components in one stage or in stages.

**Table 20**

**Dependence of purification of hydrotreated diesel distillate DBAA using ionic liquid by extraction method on the ratio of components**

IL:distillate ratio, %	Yield of raffinate, %	Yield of extract, %	The residual con-tent of aromatic hydrocarbons in the raffinate, % mass	The residual sulfur content in the raffinate, ppm	Degree of dearomatization, % mass	Degree of desulfurization, % mass	Properties of raffinate			
							$d_4^{20}$	$n_D^{20}$	Kinematic viscosity, at 20 °C, mm <sup>2</sup> / s	Cetane number
1:1	93,35	5,5	7,0	356	56,25	25,68	831	1,4639	5,84	50
2:1	8,0	12,0	6,0	304	62,5	36,53	830	1,4638	5,50	50
3:1	80,0	20,0	5,0	221	68,7	53,86	832	1,4636	5,34	52
4:1	70,0	30,0	4,8	192	70,0	60,0	833	1,4632	5,40	51
I	93,35	5,5	7,0	-	56,25	-	-	-	-	-
II	85,3	3,0	5,0	-	68,75	-	-	-	-	-
III	82,75	3,0	0,9	174,8	94,37	63,5	-	1,4564	5,40	52

When the mass ratio of the ionic liquid extractant to the distillate increases from 1:1 to 4:1, the efficiency of the extraction process increases relatively and the raffinate obtained with a threefold excess

of the extractant with a yield of 70% mass contains 5.0% aromatic hydrocarbons (degree of dearomatization 68.7% mass), 211 ppm sulfur compounds (degree of desulfurization 53.86% mass).

It has been established that when carrying out stepwise extraction using at each stage an equal amount of ionic liquid and raw material, a contact time of one hour, the amount of aromatic hydrocarbons and sulfur compounds in the raffinate is 0.9% mass and 174 ppm, and the degree of dearomatization and desulfurization is 94.37% and 63.5% mass, respectively.

The results obtained were further confirmed by IR- and UV-spectral analysis of the raffinates.

According to the results of UV spectral analysis, the residual amount of aromatic hydrocarbons in the raffinate (1.8% mass) obtained by purifying the HDD, compared to the diesel distillate taken (17.1% mass) is reduced by 9.5 times, and the degree of dearomatization is 89.47% mass.

**Table 21**

**Results of UV spectral analysis of the raffinate obtained from the extraction of hydrotreated diesel distillate with an DBAA ionic liquid**

Samples	Molecular mass	benzene derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
HDD	210	8,2	6,4	2,5	0,01	17,1
Distillate purified by ionic liquid extraction	182	1,6	0,2	traces	traces	1,8

As can be seen from the table, 80.49% of benzene derivatives, 96.87% of naphthalene derivatives are removed from the distillate taken in the process of ionic-liquid extraction, and phenanthrene and anthracene derivatives are almost completely removed.

Many physicochemical indicators of diesel distillate purified by DBAA with ionic liquid, in particular the residual amount of aromatic hydrocarbons, cetane number, kinematic viscosity, etc., comply with Euro-5 requirements.

During the purification process using ionic liquid extraction,

resinous compounds are removed from the distillate, and the resulting raffinate is transparent and characterized by a low color index of -1.0 and a refractive index of 1.4564.

**Table 22**

**Indicators of hydrotreated diesel distillate and raffinate obtained by its purification with DBAA**

Properties	diesel distillate	
	Before purification	After purification
Kinematic viscosity, at 20 °C, mm <sup>2</sup> /s	5,39	5,4
Cetane number	47	52
Content of aromatic hydrocarbons % mass	16,0	0,9
$n_D^{20}$	1,4698	1,4564
$d_4^{20}$ , kg/m <sup>3</sup>	845,7	831
Content of sulfur components, ppm	479	174,8

### **Ionic-liquid extraction purification of a mixture of straight-run diesel distillate with secondary oil products**

In order to expand the raw material base of diesel fuel and obtain diesel fuel with improved quality indicators, at the next stage of the study, ionic-liquid extraction purification of mixed diesel distillate obtained by compounding straight-run diesel fraction and light gas oil coking at their mass ratio of 70:30%, containing 25% mass of aromatic hydrocarbons and 1629 ppm of sulfur compounds.

The extraction process was also carried out using NMPA, MF, DBAA and DPAA ionic liquids as extractants. The effect of the ratio of components on the extraction process was studied at a temperature of 30-32°C and a contact time of 1 hour and it was found that, depending on the ionic liquid taken, the degree of dearomatization of the MDD was 40.0-68.0% mass (Table 23).

It has been established that in the process of three-stage purification of a mixed diesel distillate with MF, using at each stage an equal mass ratio of components, a contact time of components of 1 hour and at a temperature of 30-32°C, the raffinate yield is 83.0% mass, the degree of dearomatization is 60% mass, the degree desulfurization 54.2% mass (Table 24).



**Table 23**

**Purification of a mixture of SDD and LGC using ion-liquid extraction**

İL:distillate ratio, % mass	Yield of raffinate, %	Yield of extract, %	Content of aromatic hydrocarbons % mass	Degree of dearomatization, % mass	$n_D^{20}$	
					raffinate	extract
DBAA:MDD						
1,5:1,0	86,75	13,2	12,0	52,0	1,4700	-
2,0:1,0	74,3	25,0	11,0	56,0	1,4685	-
4,0:1,0	70,0	29,3	8,0	68,0	1,4635	-
DPAA:MDD						
2:1	72,0	27,0	15	40,0	1,4723	1,5030
NMPA:MDD						
2:1	72,0	28,0	10	60,0	1,4673	1,5165
MF:MDD						
2:1	73,6	25,1	15	40,0	1,4696	1,5160

**Table 24**

**Step-by-step purification of mixed diesel distillate**

Staged extraction	Yield, %		in raffinate		of raffinate		$n_D^{20}$	
	raffinate	extract	Content of aromatic hydrocarbons % mass	Content of sulfur components, ppm	Degree of dearomatization, % mass	Degree of dearomatization, % mass	raffinate	extract
Extraction with morphoinformate ionic liquid								
I stage	90,0	7,7	-	-	-	-	1,4722	-
II stage	85,25	4,3	-	-	-	-	1,4672	1,5477
III stage	83,0	3,75	10	746	60	54,2	1,4647	1,5318
Extraction with N-methylpyrrolidoneacetate ionic liquid								
I stage	87,5	11,95	-	-	-	-	1,4692	-
II stage	81,0	5,65	-	-	-	-	1,4647	1,5282
III stage	77,75	3,25	6	580	76	64,4	1,4622	1,4994

When carrying out the process under similar conditions using NMPA ionic liquid as an extractant, a raffinate was obtained with a relatively low yield (77.75% mass) with a degree of dearomatization

of 76% mass and a degree of desulfurization of 64.4% mass.

A study of the group hydrocarbon composition of raffinates obtained during extraction with MF and NMPA using UV spectral analysis revealed that the residual amount of aromatic hydrocarbons in the raffinate was 11.8% mass and 7.7% mass, respectively (Table 25).

**Table 25**

**Results of UV spectral analysis of raffinate samples obtained as a result of stepwise purification of a mixture of SDD and LGC**

Samples	The concentration of aromatic hydrocarbons, % mass					
	benzene derivatives	Naphthalenes	Phenanthrenes	Anthracenes	four ring aromatic	Total
MDD	10,3	8,5	3,6	2,8	1,2	26,4
purified NMPA	5,1	2,6	-	-	-	7,7
purified MF	7,3	3,2	1,3	-	-	11,8

At the next stage, the process of extraction purification of mixed diesel distillate based on straight-run diesel fraction (70% mass) and light catalytic cracking gas oil (30% mass), containing 32% mass of aromatic and 1768 ppm sulfur compounds using NMPA, MF and DBAA ionic liquid at a temperature of 30-32°C, extraction time 1 hour in one stage and in stages. It has been established that when using MF and DBAA ionic liquid as an extractant in the process of three-stage extraction purification of mixed diesel distillate, a similar result is observed, obtained with yields of 63.2 and 65.5% mass of the raffinate, characterized by a degree of dearomatization of 87.5% and 84,37% mass respectively (Table 26). The results of UV spectral analysis showed that the amount of aromatic hydrocarbons in the resulting raffinates, compared to the taken mixture of diesel distillate, is reduced by 79.6%, 72.72% and 80.0% mass, respectively, and the amount of residual aromatic compounds in the raffinates is 9.33%, 6.98%, 5.3% mass, which indicates dearomatization of the mixed diesel distillate by ion-liquid extraction (Table 27).

**Table 26**

**Stepwise purification of mixed diesel distillate using  
ion-liquid extraction**

Number of stages	Yield of, %		Residual amount in raffinate		Degree of dearomatization, %	Degree of desulfurization, % mass	raffinate	
	raffinate	extract	Aromatic hydrocarbons % mass	sulfur components, %			$n_D^{20}$	$d_{20}^4$
Extraction with morphoinformate ionic liquid								
I	82,8	15,3	22	1161	31,25	34,37	1,4779	-
II	69,1	13,1	10	754	68,75	37,38	1,4672	-
III	63,2	7,0	4,0	583	87,5	67,04	1,4647	822
Extraction with N-methylpyrrolidoneacetate ionic liquid								
I	88,2	10,1	26	1278	18,75	27,75	1,4692	-
II	73,6	14,4	13	911	59,37	48,50	1,4647	-
III	66,5	6,9	8,0	693	75,0	60,82	1,4622	830
Extraction purification with dibutylamineacetate ionic liquid								
I	85,6	13,5	24	1114	25,0	37,02	1,4796	-
II	72,1	13,2	12	803	62,5	54,60	1,4674	-
III	65,5	6,3	5,0	580	84,37	67,21	1,4614	827

**Table 27**

**Results of UV- spectral analysis of mixed diesel distillate based  
on SDD and LGCC and raffinates obtained by ion-liquid  
extraction purification**

Sample	The concentration of aromatic hydrocarbons, % mass				
	benzene derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
mixture of diesel distillate	10,1	14,1	7,31	2,7	34,21
purified MF	2,33	3,7	2,1	1,2	9,33
purified NMPA	2,01	2,05	1,9	1,02	6,98
purified DBAA	2,6	2,7	-	-	5,3

When carrying out the extraction purification process of both mixed diesel distillates under similar conditions, with the same mass ratio of components, room temperature and contact time of 1 hour, a higher degree of desulfurization and dearomatization of the raw

material is observed (Table 28).

**Table 28**

**The degree of extraction of aromatic and sulfur compounds in the process of ion-liquid extraction purification**

mixture of diesel distillate	used ionic liquid	Yield, %		Content of aromatic hydrocarbons % mass		Content of sulfur components, ppm		K <sub>a</sub>	K <sub>k</sub>
		raffinate	extract	raffinate	extract	raffinate	extract		
SDD+ LGC	MF	83,0	15,75	10,0	11,2	746	649,8	11,2	8,7
	NMPA	77,75	20,85	6,0	10,3	580	565,1	17,1	9,7
SDD+ LGCC	MF	66,5	31,4	8,0	8,9	693	419,6	11,1	6,1
	NMPA	63,2	35,4	4,0	8,7	583	397,1	21,75	6,8
	DBAA	65,5	33,0	5,0	9,0	580	421,9	18,0	7,3

The results obtained indicate that as the content of aromatic hydrocarbons and sulfur compounds in diesel distillate increases, the degree of removal of these unwanted components from the distillate increases.

The results obtained show the promise of the process of ionic-liquid extraction purification of mixed diesel distillates enriched in aromatic hydrocarbons (25 and 32% mass) and sulfur compounds (1629 and 1768 ppm).

**Purification of three-component mixed diesel distillate based on straight-run diesel distillate and secondary oil products**

The process of ion-liquid extraction purification of three-component diesel distillate based on straight-run diesel fraction, light gas oil from catalytic cracking and coking was studied at their mass ratio, respectively, 70:15:15% with a boiling point of 185-312°C, containing 29% aromatic mass and 1727 ppm sulfur compounds at a mass ratio of ionic liquid to diesel distillate equal to 2:1, temperature 20-25°C in one stage and stage by stage.

It has been established that in the process of one-stage selective purification, the yield of raffinate, regardless of the composition of the

anionic or cationic fragment of the ionic liquids used as a selective solvent, is very close and varies within the range of 72.1-74.2% mass, and a relatively high result was obtained when using NMPA ionic liquid. Thus, the degree of dearomatization of the raffinate obtained with a yield of 73.4% during the extraction process under the specified conditions is relatively high and amounts to 72.41% mass.

When carrying out the extraction process step by step with NMPA ionic liquid, the yield of the raffinate at the second stage of purification is 71.1% mass, the residual amount of aromatic compounds in the raffinate is 4.0% mass, the degree of dearomatization is 86.2% mass, when using MF ionic liquid as an extractant under similar conditions, the yield of the resulting raffinate is 71.2% mass, the degree of dearomatization is 82.76% mass. The process of selective purification of the specified composition of diesel distillate was carried out with NMPF ionic liquid in three stages with an equal mass ratio of the components and a raffinate was obtained with a yield of 68.6%, aromatic hydrocarbon content of only 2% mass, and a degree of dearomatization of 93.1% mass (Table 29).

**Table 29**

**Purification of a mixture of SDD, LGC and LGCC using ion-liquid extraction**

iL:distillate ratio, % mass	Yield, %		Content of aromatic hydrocarbons % mass	Degree of dearomatization, % mass	Content of sulfur, ppm components, ppm	Degree of desulfurization, % mass	$n_D^{20}$ , raffinate
	raffinate	extract					
NMPA:MDD							
2:1	73,4	26,0	8,0	72,41	764,0	55,76	1,4674
2:1*	71,1	28,2	4,0	86,21	616,0	64,33	1,4610
MF: MDD							
2:1	74,2	25,1	10,0	65,52	786,0	54,49	1,4684
2:1*	71,2	27,6	5,0	82,76	673,0	61,03	1,4634

**continuation of Table 29**

DBAA: MDD							
2:1	72,1	27,1	12,0	58,62	798,0	53,79	1,4707
NMPF: MDD							
1:1	86,3	13,1	18	37,93	-	-	1,4708
1:1	76,5	9,1	10	65,52	-	-	1,4653
1:1	68,6	7,6	2,0	93,10	832	51,82	1,4608

During the extraction process, sulfur compounds are also noticeably removed from the MDD content, and the greatest result is achieved in the process of selective purification of NMPA: the residual amount of sulfur compounds in the resulting raffinate is reduced to 616 ppm, and the degree of raffinate desulfurization is 64.3% mass.

When the extraction process is carried out at a temperature of 20-25°C, the mass ratio of the ionic liquid to the distillate is 3:1 and the contact time of the components is 1 hour, the amount of residual aromatic compounds in the resulting raffinate with a yield of 70.3% is 6% mass, the degree of dearomatization of the distillate is 79.3% mass, amount of residual sulfur - 604 ppm, degree of desulfurization - 65.23% mass.

**Table 30**

**Properties of the raffinate obtained by purifying a mixture of SDD, LGC and LGCC using the ion-liquid extraction method**

Properties	Three-component mixed of diesel distillate	After purification by ionic liquid
Density, 20°C, kg/m <sup>3</sup>	862,3	830,1
Kinematic viscosity, at 20 ° C, mm <sup>2</sup> / s	6,455	1,86
Amount of aromatic hydrocarbons, %	20,0	6,0
Flash temperature in a closed crucible, °C	42,8	72,4
Temperature, °C:		
cloud point	-	minus 23
Freezing	minus 28	minus 26
Fractional composition, °C:		
initial boiling point	185	190

A study of the physicochemical parameters of the raffinate obtained as a result of selective purification under optimal conditions with NMPA ionic liquid showed that the ignition temperature

increased from 42.8°C to 72.4°C, and the kinematic viscosity decreased from 6.455 mm<sup>2</sup>/s to 1.86 mm<sup>2</sup>/s (Table 30).

UV spectral analysis of the obtained raffinate samples shows that the residual amount of aromatic compounds in the raffinate with one-stage purification is 9.1% mass, with two-stage purification - 5.6% mass, and phenanthrene hydrocarbons are completely removed (Table 31).

**Table 31**

**Results of UV spectral analysis of raffinates obtained as a result of selective purification of a mixture of diesel distillates based on SDD, LGC and LGCC using the method of ion-liquid extraction**

Sample	M	The concentration of aromatic hydrocarbons, % mass				
		benzene derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
mixture of diesel distillate	249	8,2	8,9	4,1	0,5	27,7
Raffinate obtained by IL extraction:						
IL – NMPA						
I stage	207	4,5	4,1	0,5	-	9,1
II stage	197	3,2	2,4	-	-	5,6
IL – MF						
I stage	208	5,3	4,4	0,8	-	10,5
II stage	198	3,7	2,4	-	-	6,1
IL – NMPF	199	3,51	1,32	-	-	4,83
IL – DBAA	208	5,3	4,78	3,1	0,02	13,2

The conducted studies showed the promise of the method of ionic-liquid extraction purification of mixed diesel distillate, and the effectiveness of using NMPA ionic liquid as an extractant.

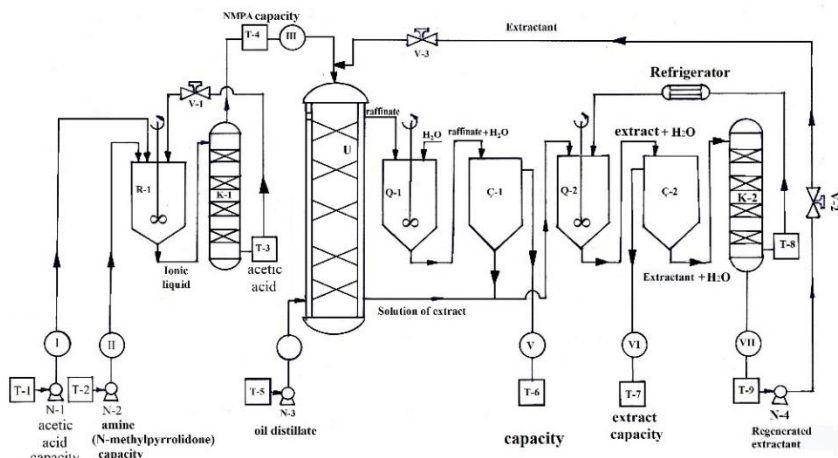
As can be seen from the results obtained, the purification of oil fractions for various purposes using the ionic-liquid extraction method is a waste-free process, and the extract obtained along with the target product (raffinate), consisting of a mixture of aromatic hydrocarbons of various compositions, can be used as plasticizing additives in concrete mixtures. At the same time, the extract obtained during the extraction purification of SDD, based on aromatic hydrocarbons (83%), was used to obtain sulfate-reducing compounds - Na and

ammonium salts of alkylarylsulfonates and it was found that at a concentration of 75 mg/l they exhibit 97% and 94.6% bactericidal effect, and at 150 mg/l they completely suppress the activity of bacteria.

A preliminary technical and economic assessment of selective purification using the MDD ion-liquid extraction method showed that the cost of diesel fuel obtained with a yield of 72.85% in the process of selective purification with NMPA ionic liquid was 331.01 AZN/t.

Thus, based on the conducted research, it has been established that the process of selective purification of oil fractions for various purposes using the ionic-liquid extraction method is based on waste-free technology and is effective from an environmental and economic point of view. The process of selective purification of oil fractions using the ionic-liquid extraction method is based on a simple, easily implemented technology, and the regeneration of the ionic-liquid extractant according to the proposed technological scheme provides an environmentally friendly approach.

The process of selective purification of oil and fuel distillates with an ionic-liquid extractant is based on the same stages. The proposed principle technological scheme is suitable for almost all compositions:



**Fig. 23. The principle technological scheme of the ionic-liquid extraction purification process**



The ionic liquid, synthesized in the R-1 reactor based on the reaction between the acid component and the amine, is distilled from unreacted acetic acid and fed into the mixer (U), where contact with the oil distillate is ensured.

The raffinate obtained during the extraction process is transferred from the top of the contactor to the mixer (Q-1) and mixed with the addition of water in an amount of 10-15% by weight of the raffinate, and then transferred to the precipitator (G-1). Phase separation occurs in the precipitator, the upper layer is the raffinate, and the lower layer is a solution of the extract in water. The resulting raffinate is collected in a tank (T-6). The aqueous solution of the extract separated from the raffinate is transferred to the mixer (Q-2) along with the extract solution obtained during the extraction process, and mixed by adding water in a mass of 20-30%, and then transferred to the precipitator (C-2). In the precipitator, the extract extracted from the distillate during the extraction process is collected in the upper layer, and the aqueous solution of the extractant - in the lower layer. After phase separation is completed, the aqueous solution of the extractant from the lower part of the precipitator enters the boiler (K-2), equipped with a heater. The water distilled from the mixture passes through the cooler and is returned to the mixing system (Ç-2) for reuse. The regenerated ionic liquid is collected from the bottom of the column into a collector intended for the extractant (T-9) and fed into the contactor for reuse.

When considering the above technological scheme, a number of advantages of the ionic-liquid extraction method compared to existing acid-contact purification methods and hydrotreating methods are noticeable.:

- performed at low temperature;
- is based on waste-free technology;
- the use of these environmentally friendly ionic-liquid compounds creates the basis for the inclusion of the extraction process in a number of green chemistry technologies

## CONCLUSIONS

1. The purification of oil and fuel distillates for various purposes using synthesized ionic-liquid compositions based on formic and acetic acids was studied and optimal conditions were developed for the production of products based on oils and fuels that meet the relevant requirements of the standards [13, 29].
2. The extraction process of transformer oil distillate was carried out using N-methylpyrrolidoneacetate, diethylamineacetate and dibutylamineformate ionic liquids as a selective solvent, and the yield compared to the yield of the acid contact method (76.7% mass) was high and amounted to 81.2-85. 0% mass, and environmentally friendly conditions for cleaning transformer oil that meet the quality standards of ГOCT 982-80 have been determined. All three ionic liquids have been shown to be easily regenerated after use and are fully recyclable [18, 24, 25, 31, 41].
3. The fraction of Naftalan oil, boiling at a temperature of 200-450°C, was purified by the extraction method using N-methylpyrrolidoneacetate and diethylaminoformate ionic liquids as extractants, and determined conditions for obtaining an almost completely dearomatized raffinate with a yield of 76.6% and 88% mass, respectively (residual amount of aromatics 0.5-0.6% mass). The results obtained were confirmed by IR-, UV- and NMR-spectral analysis methods [22, 31].
4. The process of selective purification of oil distillates of various viscosities (7.5 mm<sup>2</sup>/s and 18.0 mm<sup>2</sup>/s at 100°C) and mixed oil distillates obtained by compounding these distillates in different mass ratios (1-2:2) was studied with diethylamine informate ionic liquid based on formic acid and diethylamine, and the conditions for obtaining the raw material composition of the base oil, characterized by high thermal stability, were determined (the residue obtained during the oxidation process, determined according to ГOCT 11063-77 is 0.96% mass) [7, 20, 23, 27].
5. Selective extraction purification of kerosene fractions differing in the content of aromatic hydrocarbons and boiling point was studied using ionic liquids synthesized on the basis of formic and

acetic acids as a selective solvent, in one stage and in stages, and was established the possibility of obtaining practically dearomatized kerosene with a yield of 88,1-88.6% mass when implementing a one-stage extraction purification process with an equal mass ratio of components and a contact time of 1 hour . The obtained results were once again confirmed by IR- and UV-spectral analysis of the obtained samples of kerosene fraction and raffinate [16, 19, 21, 26, 31, 36, 37, 38].

6. It was studied the process of ionic-liquid extraction purification of diesel distillates, in particular hydrotreated and straight-run diesel fractions, as well as two- and three-component mixtures of straight-run diesel fraction with secondary oil processing products - light gas oil from catalytic cracking and coking and were determined the corresponding composition of the ionic liquid and conditions extraction [3, 14, 30, 12, 7, 17, 28, 9, 36, 10].
7. It has been shown that during the ionic-liquid extraction purification of hydrotreated diesel distillate by a selective solvent - dibutylamineacetate, with an equal mass ratio of components and a contact time of 1 hour, the residual amount of aromatic hydrocarbons in the raffinate obtained with a yield of 82.75% is 0.9% mass, the amount of sulfur compounds is 113 ppm [3, 6, 8, 14, 30, 35, 28, 12, 31, 34].
8. In order to expand the raw material base of diesel distillate, selective purification of mixed diesel distillates based on straight-run diesel fraction and products of secondary oil refining was carried out - light gas oil coking or catalytic cracking at their mass ratio of 70:30% with N-methylpyrrolidone acetate and morphoinformate ionic liquid and established that in the process of purifying a mixture of straight-run diesel fraction and light gas oil catalytic cracking, the degree of dearomatization and desulfurization is 87.5% and 67.04% mass, respectively. The results obtained were once again confirmed by IR- and UV-spectral analysis of purified raffinate samples [2 , 6, 8, 14, 30, 35, 31, 7].
9. In the process of purification of mixed diesel distillate, based on the straight-run diesel fraction of light gas oil fractions obtained

in the process of secondary oil refining at their mass ratio of 70:15:15% with N-methylpyrrolidoneacetate and morphoformate ionic liquids, it was established that when carrying out the extraction process at at 20-25C temperature with an equal amount of components at each stage, the resulting raffinate with a identical yield of 71.1 and 71.2% mass contains, respectively, 4 and 5% by weight of aromatic hydrocarbons (degree of dearomatization 86.2 and 82.76% mass), 616 ppm and 673 ppm sulfur compounds (degree of desulfurization 64.2% and 61.03% mass) [2, 8, 14, 17, 30, 35, 36, 39].

10. In the process of selective purification of athree-component mixture of diesel distillate obtained by compounding straight-run diesel fraction with light gas oil from catalytic cracking and coking with N-methylpyrrolidone acetate ionic liquid, the cost of diesel fuel obtained with a yield of 72.85% mass was determined at the level of 331.01 AZN/t and shown the effectiveness of this process compared to the purification under similar conditions of a two-component distillate based on straight-run diesel fraction and light catalytic cracking gas oil (406 AZN/t) [2, 8].

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The defense will be held on “09” april 2024 at 10<sup>00</sup> at the meeting of Dissertation Council ED 1.17 operating under academician Y.H.Mammadaliyev Institute of Petrochemical Processes of Azerbaijan National Academy of Sciences.

Address: Baku, 30 Khojaly ave., AZ 1025

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Abstract was sent to the required addresses on 07 march 2024.

Signed for print: 04.03.2024

Paper format: A5

Volume: 72800

Number of hard copies: 20