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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

DEVELOPMENT OF ENVIRONMENTALLY FRIENDLY EXTRACION PROCESSES FOR THE OIL FRACTIONS OBTAINED FROM BALAKHANY AND NEFT DASHLARY OIL FIELDS

Speciality: 3303.01 - Chemical Technology and Engineering

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GENERAL DESCRIPTION OF THE WORK

Relevance and development of the topic. Currently, one of the most important challenges of humanity that requires its solution is implementation of new, eco-friendly "green chemistry" technologies non-polluting the environment in chemical and petrochemical enterprises. One of the promising ways to eliminate the environmental problems is development of eco-friendly technologies by substituting the volatile, toxic organic solvents polluting atmosphere and environment with ionic liquids in obtaining various fuels and base oils with improved quality properties.

Recently, the "leap" growth in the number of scientific works, reviews and books published on the synthesis and multifaceted application of ionic liquids; international conferences held in Heraklion in 2000, Austria in 2005, Japan in 2007, Australia in 2009, Russia in 2017, and Bangkok in 2018 are direct confirmation of this.

This interest in ionic liquids is due to a number of useful, specific properties characteristic for them - liquidity in a wide temperature range, very low vapor pressure, thermal and chemical stability, eco-friendliness, dissolubility of a wide variety of substances, high molecular compounds, reusability, low corrosiveness, unlike salts with high melting point.

Purification of АМГ-10 (aviation hydraulic oil) hydraulic liquid distillate (HLD) from unnecessary components is currently carried out by hydropurification, hydrogenation and acid-contact method, and on the basis of systematic studies carried out at IPCP of ANAS, obtaining oil-based АМГ-10 hydraulic base oil have been developed by deparaffinization of relevant oil distillates, subsequent hydropurification, hydrogenation on industrial catalysts under mild conditions and also cleaning by acid-contact method. However, these methods are characterized by being multi-stage, loss of raw materials and obtaining large quantities of non-disposable waste products polluting the environment, rapid corrosion of devices, etc. In this regard, the development of more effective purification methods is an urgent problem facing researchers and requiring a solution from the economic and ecological point of view.

From this point of view, it is of scientific and practical importance to develop eco-friendly purification conditions from oil-based АМГ-10 HLD aromatic hydrocarbons, sulfurous and resinous compounds by ionic-liquid extraction method.

The object and subject of the research. The object of the study is АМГ-10 HLD obtained on the basis of oils produced from the Balakhany and Neft Dashlary oil fields, but the subject of the study is determination of the conditions for removing unnecessary components of these distillates by the extraction method using various ionic-liquid salts synthesized on the basis of formic acid and acetic acid as a selective solvent.

The purpose and tasks of the research. The purpose of the dissertation work is determined by the development of the conditions for obtaining АМГ-10 hydraulic liquid base product, meeting the requirements of ТУ 0253-021-45993103-2006, through selective purification of HLD developed based on oils produced from Balakhany and Neft Dashlary fields by ionic-liquid extraction method. For this purpose:

- determining the conditions for removal of unnecessary components from АМГ-10 HLD by extraction method using ionic liquids synthesized on the basis of formic and acetic acids as selective solvents;
- study of dearomatization process of HLD sample separated from the oil of Balakhany oil field by distillation, prepared by compounding the fractions differing by 10ºC of boiling temperature and accordingly IBP - 234ºC and EBP - 306ºC, freezing point – minus 75°C, kinematic viscosity – 2,31 mm^2/sec at 50°C;
- study of dearomatization process of the distillate obtained on the basis of oil extracted from Neft Dashlary oilfield by the same approach with IBP - 222ºC, EBP - 313ºC, freezing point - minus 72ºC, kinematic viscosity 2,45 mm^2/sec at 50°C;
- study of the regeneration process of N-methylpyrrolidoneacetate, which is more effective than the series of ionic liquid compositions used as a selective solvent in extraction process;

- technical-economic evaluation of the efficiency of the ionic liquid extraction method compared to the acid-contact method, which has found application on an industrial scale.

Research methods: The studies on the dissertation work were carried out using modern methods - IR, $^{1}H^{13}C$ NMR, UV, chromatomass spectrocopy and determination of the raffinate parameters obtained by HLD primary and extraction were carried out via accepted standard methods and the results were compared and confirmed.

The main provisions for defence:

- study of the effect of various factors - extraction temperature, amount of extractant, contact time of components, determination of optimal extraction conditions - on the purification process of HLD samples by ionic-liquid extraction method and on the quality properties of obtained raffinate samples;
- study of the composition of the ionic liquid extractant, that is, effect of the cation-anion combination on the efficiency of the extraction process;
- determination and justification of the effective ionic-liquid content in purification of HLD by ionic-liquid extraction method;
- a comparative study of the purification process by ionic-liquid extraction method with the acid-contact method currently applied on an industrial scale in the presence of acetic anhydride;
- justification of economic efficiency of purification process of HLD by ionic-liquid extraction method.

Scientific novelty of the research. For the first time, eco-friendly, ecologically harmless purification conditions were determined for HLD samples developed on the basis of oils extracted from Balakhany and Neft Dashlary oil fields using ionic liquids applied as selective solvents in "green chemistry" technologies;

- purification of HLD samples was carried out by extraction method by ionic liquids of various components synthesized using morpholine, N-methylpyrrolidone, di- or triethylamine as an amine component, based on formic and acetic acids as selective solvents, and the conditions were determined for obtaining АМГ-10 hydraulic liquid base product meeting the standard requirements;
- one-step or multi-step selective purification process of HLD sam-

ples obtained on the basis of Balakhany oil using morpholinformate ionic liquid composition as an extractallows determining the conditions for obtaining АМГ-10 hydraulic liquid base product with 2% of residual amount of aromatic hydrocarbons in the raffinate with a raffinate yield of 85.4%, and practically completely dearomatized with a raffinate yield of 81.6%;

- the conditions for obtaining were determined for practically completely dearomatized АМГ-10 hydraulic liquid base product with 72.5% yield meeting the requirements for HLD obtained on the basis of Neft Dashlary oil using N-methylpyrrolidoneacetate-containing ionic liquid as a selective solvent;
- the regeneration conditions were developed for the ionic liquid containing N-methylpyrrolidoneacetate used in the extraction process and shown that it may be reused with the same effect;
- the studied HLD selective purification process was comparatively investigated using organic solvent N-methylpyrrolidone as an extractand the ionic-liquid extraction method was found to be effective;
- purification process of HLD developed ionic-liquid extraction method was compared with the acid-contact method purification process in the presence of acetic anhydride, and it was determined that ionic-liquid extraction method is more appropriate from an economic and ecological point of view.
- based on the results obtained from the principle technological scheme of the selective purification process using HLD ecologically harmless, economically efficient ionic-liquid extraction method was proposed.

Theoretical and practical significance of the study:

- purification of HLD obtained on the basis of oils produced from Balakhany and Neft Dashlary oil fields from aromatic hydrocarbons by eco-friendly ionic-liquid extraction method ensures obtaining АМГ-10 hydraulic liquid base product meeting the requirements of ТУ 0253-021-46693103-2006;
- profit from HLD selective purification process using N-methylpyrrolidoneacetate-containing ionic liquid as an extractis 208,4 AZN/t by one-step process, and 143,5 AZN/t multi-step process;

– possibility for easy regeneration of ionic-liquid extractand reuse with the same effect confirms the practical importance of the selective purification process by the extraction method.

Author's personal contribution. Setting and solving issues related to the implementation of the main ideas in the dissertation, performing experiments, analyzing and systematizing the obtained results, writing papers were carried out with the participation of the applicant, each stage related to the preparation of the dissertation work was performed by the applicant personally.

Approbation of work. The main results of the studies were reported and discussed at the conferences and symposia held on the national and international scale mentioned below: International Scientific Conference "Current problems of modern natural sciences" (Azerbaijan, Ganja, 2017); Republican Scientific and Technical Conference on "Fuels, fuel components, special purpose liquids, oils and additives" dedicated to the 90th anniversary of Professor S.A. Sultanov (Baku, 2017); International Scientific and Practical Conference dedicated to the 100th anniversary of Academician Vahab Aliyev (Baku, 2018); "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 "Actual problems of modern chemistry" dedicated to the 90th anniversary of academician Y.H. Mammadaliyev Institute of Petrochemical Processes (Baku, 2019); Baku Engineering University, conference dedicated to the 96th anniversary of the birth of national leader Heydar Aliyev (Baku, 2019); International Scientific and Practical Conference "Radiation and chemical safety problems" (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); the 10th Rostock International Conference "Thermophysical Properties for Technical Thermodynamics (Rostock Germany, 2021); Baku Higher Oil School, III International Scientific Conference of Students and Young Researchers dedicated to the 99th anniversary of the birth of national leader Heydar Aliyev

(Baku, 2022).

Scientific publications. The main scientific and practical results of the dissertation work are reflected in 20 scientific works, including 9 papers and 11 theses published in international and national journals, two of which are single authored.

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. 16/2016, 16/2017, 16/2018, 14/2021).

The structure and total volume of the dissertation, noting the volume of the structural sections separately. Dissertation work is 187 pages of computer printout, consists of introduction, 4 chapters, conclusions and bibliography including 277 references. Dissertation is 209900 characters long, excluding 20 pictures, 46 tables and literature list.

The introduction (12700 characters) reflects the substantiation of the relevance of the studies, the purpose of the work, the issues raised, the scientific and practical importance of the studies.

The first chapter (63000 characters) explains the methods for improving the quality properties of oil and fuel fractions with different viscosity, including the studies on purification by liquid-liquid extraction, and the advantages of ionic-liquid extraction method.

The second chapter (17200 characters) describes the method for development of distillate samples used as raw materials in the studies, the physico-chemical parameters, purification procedure and properties of other reagents used in the studies, and synthesis of ionic liquid components used as extractants.

The third chapter (70700 characters) deals with the studies to detract unnecessary components from the distillate samples by the ionic-liquid extraction method, determining the optimal conditions and effective ionic-liquid extractant.

The fourth chapter (43800 characters) deals with comparison of selective purification processes using N-methylpyrrolidone acetate-containing ionic liquid, organic solvent N-methylpyrrolidone, and acid contact method; making an economic calculation; and proposition of a principle technological scheme for ionic-liquid extraction process based on eco-friendly technology.

The results of the carried-out studies (2500 characters) and a list of cited literature are given at the end of the dissertation.

MAIN CONTENT OF THE WORK

Low-paraffin oils, characterized by a unique property, low freezing point are used for obtaining base products of liquids used as working liquids in various mechanisms and aggregates, hydraulic systems of aircrafts.

Taking this into account, the distillates separated from the oils produced from the Balakhany and Neft Dashlary oil fields were used as raw materials in obtaining АМГ-10 hydraulic liquid base product.

The existing and increasingly severe environmental problem in oil refining processes using ionic-liquid compounds as selective solvents meeting the principles of "green chemistry" is a very relevant and promising scientific direction in the extraction of oil fractions, including hydraulic liquid distillate and is of scientific and practical importance.

Based on the results of the studies carried out on purification of various oil and fuel fractions by ionic-liquid extraction at IPCP of ANAS, the selective purification process of HLD obtained from the Balakhany oil, which was characterized firstly by the anion-cation fragment synthesized using - formic and acetic acid-based and morpholine and N-methylpyrrolidone as an amine based, was carried out by applying ionic-liquids of different components in a 1-2:1 mass ratio of ionic liquid to distillate, at a temperature of 60ºC and a contact time of the components for 2 hours.

As is seen from Tab. 1, depending on the content of the ionic liquids, the yield of the raffinate varies in the range of 85,2–95,0% mas., and using N-methylpyrrolidoneformiate (N-MPA) as a selective solvent result in relatively large mass amount 13,3–14,8% of the extract separated from the content of the taken distillate. Simultaneously, the residual amount of aromatic hydrocarbons in the raffinate under the mentioned conditions is practically close to 3,8-4,0% mas.

Table 1.

Dependence of dearomatization process of distillate by ionic-liquid extraction method on extractcomposition

Ionic liquid	IL:distillate mas. ratio	Yield, mas.%		Residual amount of aromatic hydrocarbons in raffinate, mas. %
		raffinate	extract	
Morpholineformiate	1:1	95,0	5,0	10,0
	2:1	91,4	8,6	4,0
N- methylpyrrolidoneformiate	1:1	86,7	13,3	6,0
	2:1	85,2	14,8	4,0
Morpholineacetate	2:1	91,6	8,4	4,0
N-methylpyrrolidoneacetate	2:1	92,0	8,0	3,8

Based on the preliminary results, dearomatization process of the studied raw materials by the АМГ-10 HLD extraction method was studied by each of the above-mentioned ionic liquid compositions, and the optimal conditions were determined.

Firstly, effect of various factors on the extraction process of the distillate developed based on Balakhany oil using morpholinformate (MF)-containing ionic liquid as a selective solvent was studied.

Table 2

Effect of temperature and ratio of components on ionic-liquid extraction process of distillate. Extraction time - 2 hours

MF:HLD, mas. %	Extraction temperature, °C	Yield, mas. %		Amount of aromatic hydrocarbons in raffinate , %	n_D^{20}	
		Raffinate	Extractant		raffinate	extractant
1:1	60	95,0	5,0	10,0	1,4940	1,5254
1,5:1	60	93,2	6,8	6,0	1,4685	1,5250
2:1	25-30	90,0	10,0	6,0	1,4646	1,5246
2:1	40	88,2	11,8	5,0	1,4622	1,4990
2:1	60	86,8	13,2	4,0	1,4616	1,5190
3:1	25-30	85,2	14,8	4,0	1,4610	1,5130
3:1	40	81,8	18,2	3,0	1,4608	1,5146
3:1	60	85,4	14,6	2,0	1,4610	1,5140
3:1	70	78,8	21,2	2,0	1,4609	1,5140

As is evident from the obtained results (Table 2), purification degree of the distillate from unnecessary components, that is, the degree of removal of aromatic hydrocarbons from the composition, varies depending on the amount of ionic liquid extractand temperature during the same contact time of the components.

The obtained results may be explained by the decrease in the viscosity of the distillate due to the increase in the temperature conditions of the extraction process, and the increase in the degree of solubility in the extractant, as well as the decrease in the selectivity of the ionic liquid, the difficulty in separating of the raffinate phase from the extract phase. Thus, simultaneously with a decrease in the yield of raffinate as a result of an increase in solubility index and in temperature from 60°C to 70°C in the equal mass ratio of the components, the residual amount of aromatic hydrocarbons in the obtained raffinate practically doesn't change.

Effect of the components contact time on the yield and quality properties of the obtained raffinate was studied in 2 or 3 times more mass amounts of the ionic liquid extract compared to the distillate.

It was determined that the degree of removal of aromatic hydrocarbons from the distillate reached the maximum level – 88,23% mas. (Table 3) at extraction temperature of 60ºC, in the amount of the ionic liquid taken more than the distillate for a period of 2 hours. Residual amount in the obtained raffinate was determined 2,0% mas. by sulfonation method of aromatic hydrocarbons (ГОСТ 15994-74).

Table 3

Effect of extraction time on raffinate yield and dearomatization degree. Extraction temperature – 60°C

MF:HLD, mas. %	Extraction time, h	Yield, mas.%		n_D^{20}		Amount of aromatic hydrocarbons in raffinate, %	Dearomatization degree of raffinate, mas. %
		Raffinate	Extractant	Raffinate	Extractant		
2:1	1	91,0	9,0	1,4615	1,5192	7,5	55,88
	2	86,8	13,2	1,4618	1,5190	4,0	76,47
	3	85,4	14,6	1,4610	1,5190	3,0	82,35
	4	84,2	16,8	1,4600	1,5185	2,5	85,3
3:1	1	84,8	15,2	1,4670	1,5180	6,0	64,7
	2	85,4	19,2	1,4610	1,5146	2,0	88,23
	3	79,5	20,5	1,4645	1,5140	2,0	88,23
	4	75,6	24,4	1,4640	1,5142	2,0	88,23

At the further stage of the study, selective purification process by extraction method with АМГ-10 HMD MF-containing ionic liquid was carried out in stages and conducting the ionic-liquid extraction process in three stages by different mass ratios of the extractto the distillate, the conditions for obtaining was determined for practically completely dearomatized hydraulic liquid base product with the yield of 81,6%.

Table 4

Selective purification of distillate with N-methylpyrrolidoneformiate-containing ionic liquid

Stages	IL:distillate ratio	Tem., ºC	Contact time	Yield, % mas.		n_D^{20}		Aromatic hydrocarbons amount in raffinate, % mas.
				Raffinate	Extractant	Raffinate	Extractant	
The I stage	2:1	40	3,0	77,8	15,2	1,4616	1,4890	4,0
The I stage	2,5:1	60	2,0	70,6	29,4	1,4630	1,4964	2,0
The I stage	3:1	60	2,0	89,2	14,8	1,4535	1,4990	3,0
By stages								
The I stage	1,5:1	40	1,0	87,0	11,7	1,4682	1,4990	4,0
The II stage	1,5:1	40	1,0	82,4	27,3	1,4602	1,4763	2,0
The II stage	1,25:1	50	1,5	89,53	10,5	1,4610	1,5180	3,0
The II stage	1:1	50	1,0	77,0	9,66	1,4610	1,5016	2,0
The I stage	1,5:1	60	1,0	87,25	12,75	1,4645	1,5142	3,5
The II stage	1,5:1	60	2,0	77,75	27,25	1,4615	1,4946	2,0
The I stage	1:1	60	1,0	89,10	9,53	1,4675	1,5130	6,0
The II stage	1:1	60	1,0	87,5	12,5	1,4650	1,5150	4,5
The III stage	1:1	60	1,0	89,74	10,26	1,4620	1,5130	2,0

Purification process of the distillate using N-methylpyrrolidoneformiate (N-MPF) as an extract revealed that the raffinate yield was 70,6% mas., residual amount of aromatics – 2% mas. in 2 hours contact time of the components, at 60ºC, in the amount of ionic-liquid extract 2,5 times more than the raw material. The same dearomatization degree (88,23%) was obtained by performing the process in stages at different mass ratios of ionic liquid to distillate and at different temperatures, that is, the residual amount of aromatic hydrocarbons in the raffinate is 2% mas. (Table 4).

The studies resulted in having possibility to reduce aromatic hydrocarbons amount to 2% by applying N-MPF as an extractant in selective purification process of АМГ-10 HLD.

Physico-chemical properties of raffinate samples taken from the purification of HLD by extraction method using both mentioned ionic-liquid compositions as selective solvents were determined and low freezing point (minus 75ºC), high flash point (110,5-112,4ºC) and kinematic viscosity properties (2,3-2,4 mm^2/sec at 50ºC, 200,5-190,85 mm^2/sec at minus 50ºC) wewre alsdo determined according to the corresponding technical condition for АМГ-10 hydraulic liquid base product.

At the further stage of the study, the process for purifying the distillate (12% of aromatic hydrocarbons) based on the oil produced from the Neft Dashlary oil field was studied by ionic-liquid extraction method, first of all, the conditions for complete dearomatization of the distillate were determined using N-MPF or N-MPA synthesized on the basis of N-methylpyrrolidone (N-MP) ionic liquid compositions as an extractant (Table 5).

Table 5

Dependence of the extraction process on the ratio of components. Temperature – 60°C, contact time of components – 2 hours

IL:distillate, % mas.	Yield, % mas.		n_D^{20}		Aromatic hydrocarbons amount in rafinate, % mas.
	Raffi-nate	Extrac-tant	Raffi-nate	Extractant	
N-methylpyrrolidoneformiate-containing ionic liquid					
1,5:1,0	83,3	15,4	1,4633	1,4883	6,5
2,0:1,0	80,3	19,0	1,4629	1,4968	4,0
2,5:1,0	73,6	27,0	1,4630	1,4994	2,0
3,0:1,0	70,65	28,5	1,4635	1,4845	2,0
N-methylpyrrolidoneacetate-containing ionic liquid					
1,5:1,0	78,5	21,5	1,4610	1,4885	4,5
2,0:1,0	76,4	22,0	1,4530	1,4845	2,0
2,5:1,0	72,5	25,4	1,4520	1,4850	0,0
3,0:1,0	70,5	27,0	1,4620	1,4878	0,0

It was determined that as the amount of ionic liquid extractant taken from the raw material increases, purification degree of the distillate from aromatic hydrocarbons increases, and when the extraction process is carried out using N-MPF as a selective solvent, the yield of raffinate is 2,5 times higher than IL distillate 73,6% mas., if the residual amount of aromatic hydrocarbons is 2% mas., by using N-MPA as an extractant, under similar conditions, complete dearomatization of the obtained distillate is observed, and the analysis of the raffinate obtained with a yield of 72,5% by the sulfonation method reveals that there are no aromatic hydrocarbons left in the content. Based on this result, further studies in the field of selective purification of HLD by ionic-liquid extraction method were carried out using N-MPA as an extractant and the effect of extraction temperature simultaneously with the ratio of components on the process was studied.

As is evident from the table 6, along with the increase in the amount of ionic liquid extractant containing N-MPA relative to the distillate, as the extraction temperature increases, the degree of removal of aromatic hydrocarbons from the distillate content increases, and dearomatization of the distillate is completely ensured at the extraction temperature of 60°C, 2,5 times more mass amount of the ionic liquid relative to the distillate.

Table 6

Selective purification of distillate based on oil produced from Neft Daslary oil field by N-methylpyrrolidoneacetate

IL:HLD, % mas.	Extraction		Yield, % mas.		n_D^{20}		Aromatic hydrocarbons amount in raffinate, % mas.
	Tempera-ture,°C	time, hour	raffinate	extractant	raffinate	extractant	
1:1	40	2,0	92,0	8,0	1,4636	1,4983	6,0
1,5:1	40	2,0	85,83	14,17	1,4634	1,4975	5,0
2:1	40	2,0	83,8	16,2	1,4632	1,4950	4,0
3:1	40	2,0	75,8	22,8	1,4620	1,4920	3,0
1,5:1	50	2,0	74,0	26,0	1,4634	1,4980	5,0
2:1	50	2,0	72,5	25,9	1,4531	1,4835	2,0
2,5:1	50	2,0	70,6	21,4	1,4633	1,4942	2,0
3:1	50	2,0	69,0	29,2	1,4630	1,4950	1,0
1,5:1	60	2,0	78,5	21,2	1,4610	1,4885	4,5
2,0:1	60	2,0	76,4	20,6	1,4536	1,4845	2,0
2,5:1	60	2,0	72,5	21,4	1,4540	1,4860	0,0
3:1	60	2,0	70,5	27,0	1,4620	1,4878	0,0
3:1	70	2,0	68,8	28,2	1,4615	1,4910	2,0

As is seen from the table 7, complete dearomatization is achieved in two stages by selective purification process of HLD by ionic-liquid extraction method is carried out step by step by ensuring the contact of the distillate with a new part of the ionic-liquid extractant.

Table 7

Selective purification of АМГ-10 by ionic liquid extraction method of hydraulic liquid distillate by stages

IL: HLD, % mas.	Extraction		Yield, % mas.		n_D^{20}		Aromatic h/c amount in raffinate, % mas.
	Temperature,°C	time, hour	Raffinate	Extractant	Raffinate	Extractant	
Three-stages, 1:1 at each of the stages							
The I stage	40	1,0	92,0	7,6	1,4780	1,4896	7,0
The II stage	40	1,0	82,8	16,8	1,4634	1,4884	4,0
The III stage	40	1,0	72,8	26,9	1,4630	1,4880	2,0
The I stage	60	1,0	88,9	10,4	1,4700	1,4890	4,0
The II stage	60	1,0	80,2	14,9	1,4540	1,4830	0,0
The III stage	60	1,0	71,2	26,6	1,4530	1,4845	0,0
Two-stages, 1.5:1 at each of the stages							
The I stage	50	1,0	85,7	14,3	1,4620	1,4810	5,0
The II stage	50	2,0	85,3	14,7	1,4630	1,4870	1,0
The I stage	60	1,5	84,1	17,5	1,4639	1,4890	5,0
The II stage	60	1,5	79,7	17,3	1,4634	1,4850	2,0
In 2 stages							
In the I stage 3:1	50	2,5	70,6	29,2	1,4633	1,4960	2,5
In the I stage 3:1	50	2,5	70,6	29,2	1,4633	1,4960	2,5
In the II stage 1:1	50	2,0	71,48	28,5	1,4625	1,4870	0,0

The IR spectral analysis of the raffinate samples obtained from the purification of the distillate by ionic-liquid extraction method reveals that there are practically no aromatic hydrocarbons left in the composition, and absorption bands characteristic of the aromatic fragment (699, 743, 811, 871, 1603 cm^{-1}) are not observed in the spectrum (Fig.1).

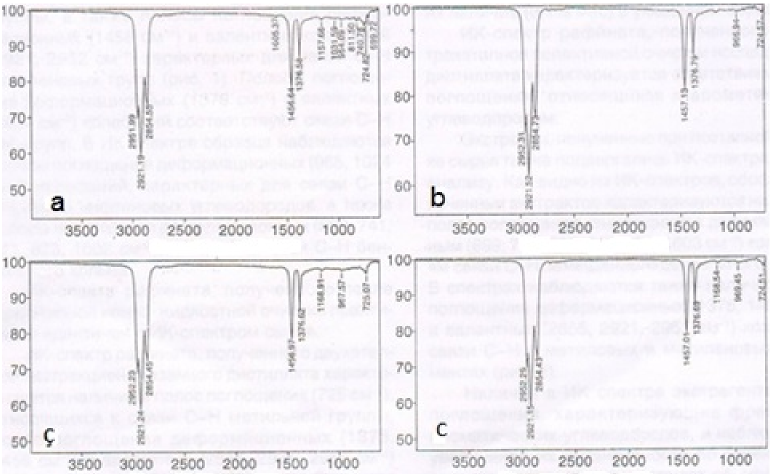

Fig. 1. IR spectra of raffinate samples obtained without purification of distillate (a) in one stage (b), two stages (ch) and three stages (c) by ionic liquid extraction method

The chromo-mass spectrometric analysis of the extracted HLD and the hydrocarbon group composition of the raffinate obtained from its purification by the ionic-liquid extraction method using "Clarus SQ-8T" mass-spectral detector manufactured by "Perkin-Elmar" "Clarus-680" company also confirms a complete dearomatization of the distillate (Fig. 2).

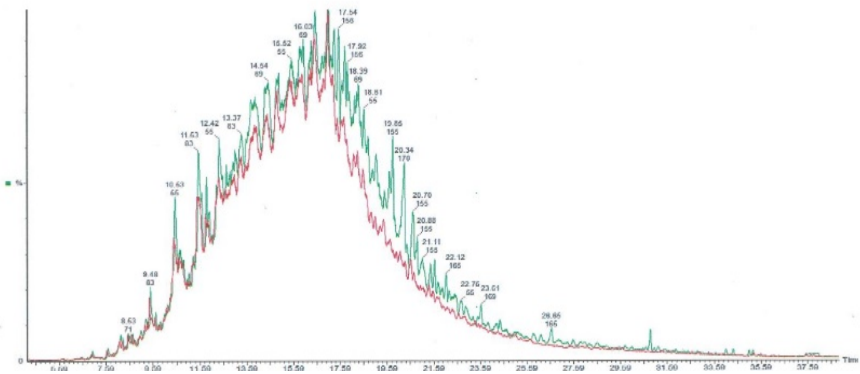

Fig. 2. Comparative chromium-mass spectra of the distillate and the raffinate obtained by ionic liquid purification

The results reveal that using N-MPA-containing ionic liquid in selective purification of AMΓ-10 HLD is purposeful.

The possibility of changing the cation and anion fragment in ionic-liquid compositions on a large scale raises the interest of carrying out studies in the direction of determining a prospective extractant useful for selective purification of oil fractions. At the next stage of studies, there were studied the possibilities for selective purification of the synthesized ionic liquids based on HLD diethyl or triethylamine and formic or acetic acid with diethylamine formate (DEAF), diethylamine acetate (DEAA) and triethylamine acetate (TEAA). For the purpose of evaluate the selectivity of DEAF, the hydrocarbon group composition of the distillate taken as raw material and the raffinate obtained in the selective purification process and the residual amount of aromatic hydrocarbons were comparatively studied by IR and UV spectral analysis methods. It was determined that absorption bands reflecting aromatic hydrocarbon fragments (669, 741, 811, 873, 1602 cm^{-1}) are practically not observed in the IR spectrum of the sample purified by ionic-liquid extraction method.

Taking into account that organic solvent - N-methylpyrrolidone (NMP) is used as an extractant on an industrial scale, selective purification of AMΓ-10 HLD was studied and it was determined that the raffinate yield obtained by selective purification process of the distillate with N-MP was much lower (82.5% mas.) than the yield of the raffinate obtained by dearomatization using N-MPA as an extractant under similar conditions totaling 65.8% mas., and the residual amount of aromatic hydrocarbons in the obtained raffinate was 3% mas. to a complete dearomatization (Table 8).

The result reveals that selectivity of N-MP is relatively low and its efficiency is low compared to the ionic liquid synthesized on its basis - N-methylpyrrolidoneacetate.

In conclusion, based on the systematic studies, it was proved efficiency of the use of AMΓ-10 HLD N-MPF and N-MPA-containing ionic liquids as an extractant developed by compounding on the basis of the fractions with a boiling point of 10°C, separated by distillation from the oils from Balakhany and Neft Dashlary oil fields.

Table 8

Comparison of АМГ-10 hydraulic liquid distillate selective purification process with N-methylpyrrolidone and N-methylpyrrolidone acetate

Extractant	Extractant:distillate, mas.	Extraction time, h	Yield, % mas.		Removal rate of aromatic h/c, % mas.	Aromatic h/c amount in raffinate, % mas.
			Raffinate	Extractant		
N-MPA	2,5:1	2,0	82,5	17,5	100,0	0,0
by stages	1:1	1,0	88,8	11,2	83,3	2,0
	1:1	1,0	80,7	19,3	100,0	0,0
NMP	3:1	2,0	65,8	34,2	75,1	3,0
by stages	1:1	1,0	78,9	21,1	76,0	3,0
	1:1	1,0	63,8	36,2	83,3	2,0
	1:1	1,0	58,7	41,3	83,3	2,0

Selective purification of АМГ-10 hydraulic liquid distillate by acid-contact method in the presence of acetic anhydride

Selective purification process of HLD obtained on the basis of oil produced from the Balakhany fields, developed at IPCP of ANAS, by the acid-contact method is known[1]. It's multi-stage process with low yield of the target product (66.7% mas.), high energy consumption, excess consumption of solid sulfuric acid and is characterized by obtaining a large amount of hard-to-utilize, environmentally polluting waste products - sour tar and wastewater, processed clay, so that can't be considered ecologically efficient.

Efficiency of the acid-contact method is determined by the concentration of sulfuric acid used, and the water released during the sulfonation process gradually reduces the concentration of the extracted sulfuric acid, as a result it becomes difficult to remove aromatic hydrocarbons from the distillate.

In order to prevent this and obtain the basic product of hydrau-

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[1] Aliyeva S.G. Obtaining prospecitve jet fuels and hydraulic liquid base oils from Azerbaijani oils / Doctoral dissertation – Baku, 2009

lic fluid with improved quality properties, purification process of the distillate by acid-contact method was carried out in the presence of a water-absorbing agent - acetic anhydride.

The advantage of the acid-contact method for purifying the extracted distillate in the presence of acetic anhydride is characterized by a high yield of raffinate - (85% mas.) and, most importantly, a very small amount of waste product (sour tar) and waste water.

For the purpose of determining the most effective approache to HLD selective purification process - ionic liquid extraction, selective purification using organic solvent N-MP as an extractant, or acid-contact purification method in the presence of acetic anhydride, raffinate and extract samples obtained by purification via these three methods were comparatively studied by IR and UV spectral analysis methods.

In the IR spectra of the obtained raffinate samples, depending on the extraction conditions and the ingredients used as extractants, the intensity of the absorption bands (742, 777, 811, 1604 cm^{-1}) reflecting the deformation vibrations of the C-H bond in the benzene ring differ, decrease or complete disappearance is observed. This is clearly noticeable when comparing the optical density properties of the absorption bands characteristic for the mentioned raffinates (Table 9).

As is evident from the table, if the optical density property of the absorption band reflecting the C-H bond in the aromatic ring (1604 cm^{-1}) in the raffinate sample obtained from the purification of the distillate by ionic-liquid extraction method decreases by 1.66 times compared to the optical density property of the corresponding absorption band in the obtained distillate, in the presence of acetic anhydride it is reduced by 2.5 times in the sample of raffinate obtained by purification via acid-contact method, and by 1.2 times in the raffinate obtained by N-MP purification.

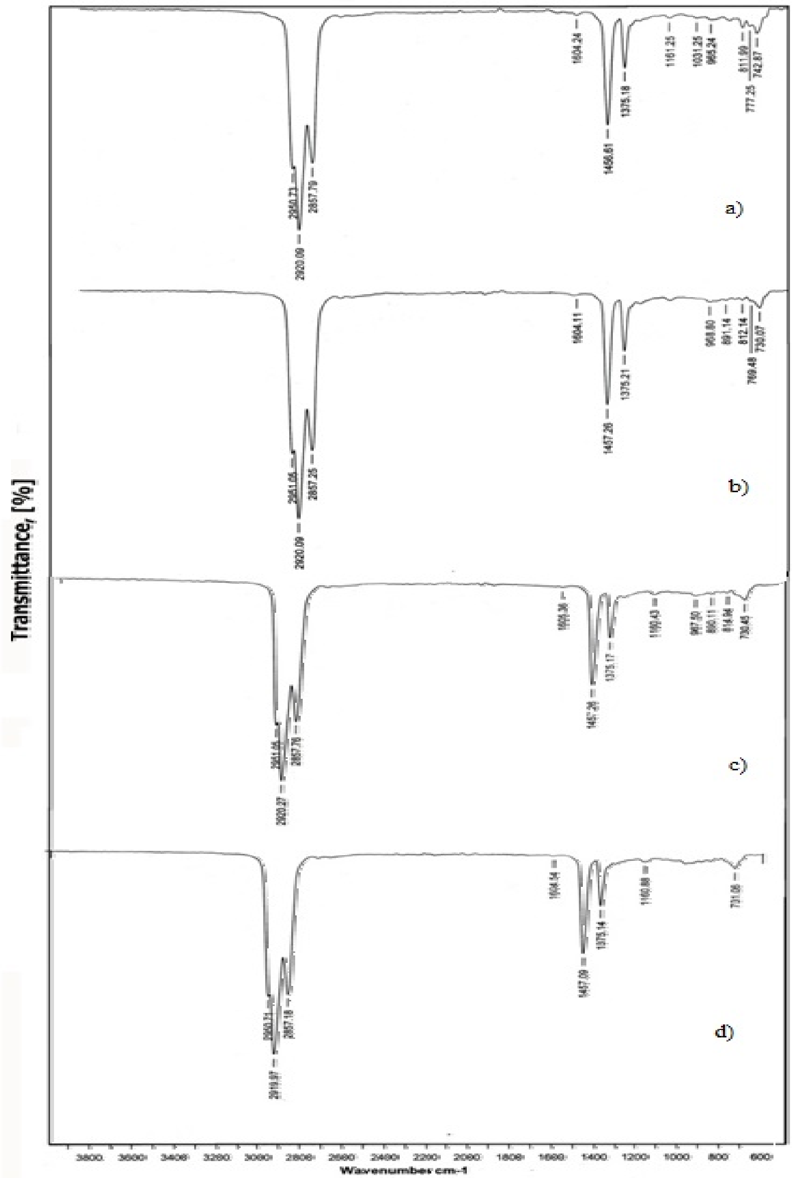


Figure 3. IR spectra of raffinate samples obtained by purification with АМГ-10 hydraulic liquid distillate (a), ionic-liquid extraction method (b), N-methylpyrrolidone (c) and acid-contact method (d)

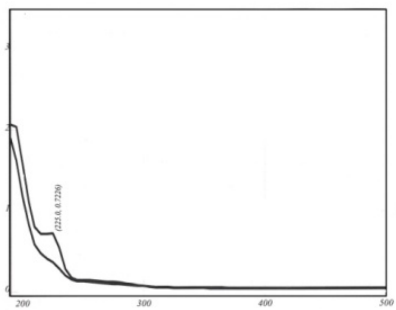
Table 9

Optical intensities of absorption bands characteristic for distilled distillate and raffinate samples obtained by selective purification

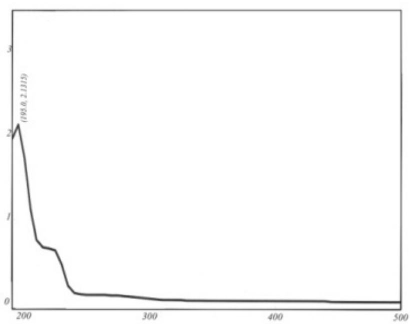
No.	Sample	D_{1375}	D_{1456}	D_{968}	D_{1031}	D_{1604}
1	Balakhany oil distillate	0,051	0,107	0,009	0,008	0,005
2	Raffinate obtained by ionic liquid extraction of distillate	0,052	0,104	0,007	-	0,003
3	Raffinate obtained by purification with organic solvent N-methylpyrrolidone	0,054	0,0106	0,008	-	0,004
4	Raffinate obtained by purification via acid-contact method	0,051	0,104	0,007	-	0,002

Besides, in the IR spectra of raffinate samples obtained by using an organic solvent - N-MP as an extractant or purification by acid-contact method, optical density property of the absorption bands specific to the C-H bond of the CH_2 fragment) in the naphthenic structure either disappear (D_{1031}) or significantly decrease (D968 cm^{-1}) in contrast to the distillate taken as raw material. It proves that naphthene-aromatic hydrocarbons are removed from the raw material during the selective purification process of the obtained distillate.

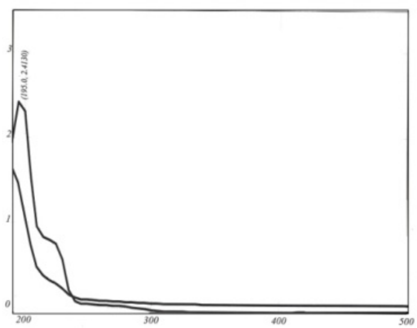
These obtained results were confirmed again based on the UV-spectral analysis of the mentioned raffinate samples and distillate taken as raw materials.



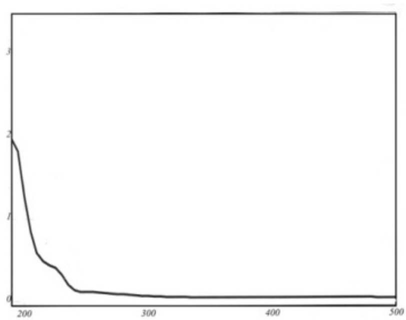
(a)



(b)



(c)



(d)

Figure 4. UV spectra of АМГ-10 hydraulic liquid distillate (a) and raffinate samples obtained by selective purification of this distillate by ionic liquid (b), N-methylpyrrolidone (c) and acid-contact method (d)

The results of UV spectral analysis also reveal that ionic liquid containing N-MPA has a higher extraction capacity. Thus, the amount of residual aromatics in the raffinate obtained by ionic-liquid extraction method is significantly reduced compared to the distillate (Table 10).

Table 10

UV spectral analysis results

Samples	Mol.weight	Monocyclic aromsatic hydrocarbons, % mas.	Bicyclic aromati hydrocarbons, % mas.	Total, %
1. Balakhany oil distillate	218	7,9	6,5	14,4
2. Raffinate obtained by one-step ionic-liquid extraction method	190	0,074	0,21	0,95
3. Raffinate obtained by ionic-liquid extraction method by stages	182	0,51	0,13	0,64
4. Raffinate obtained by organic solvent N-methylpyrrolidone	180	1,94	0,62	2,56
5. Raffinate obtained by acid-contact method	200	1,8	0,71	2,51

The raffinate samples obtained by selective purification of the taken distillate with N-methylpyrrolidone via acid-contact method are characterized by the presence of a significant amount of residual aromatics in the composition (2,56, 2,51% mas.).

Thus, the obtained distillate with different approaches:

- using ionic liquid containing N-MPA as an extractant;
- using organic solvent N-MP as an extractant;
- using acid contact purification analysis of the structure-group composition of the raffinate samples by IR and UV spectral analysis methods proves efficiency of ionic-liquid extraction method and high selectivity of the ionic-liquid composition.

Evaluation of economic efficiency of selective purification of АМГ-10 hydraulic liquid distillate by ionic liquid extraction method

The possibilities of regeneration and reuse of N-MPA-containing ionic liquid recommended for use as an extractant in the АМГ-10 HLD ionic-liquid extraction method, mathematical modeling of the process, technical and economic evaluation were carried out, and the results were compared with the known acid-contact method and evaluated.

It was determined that the regeneration of the extractant is ensured by adding water to the extract phase. Water is removed from the obtained solution by distillation and returned to the system, to the regeneration stage. The composition and structure of the ionic liquid extractant regenerated by this method was studied by IR and NMR spectral analysis methods and compared with the corresponding spectra of N-MPA. Based on the study of the effectiveness of reusing the ionic liquid extractant regenerated by the mentioned method in the selective purification of the extracted distillate, it was determined that it has the same effect and its repeated reuse (more than 100 cycles) is purposeful.

The experimental results obtained from the extraction of distillate by N-MPA-containing ionic liquid were used for mathematical modeling of selective purification process of АМГ-10 HLD by ionic-liquid extraction method.

Solving the issue of optimization process proved that obtaining АМГ-10 hydraulic base oil meeting the requirements of ТУ 0253-021-46693103-2006 is ensured by HLD extraction process using N-MPA-containing ionic liquid as an extractant.

Table 11

Physico-chemical properties of АМГ-10 hydraulic base oil purified by ionic liquid extraction method

Properties	TC 0253-021-46698103-2006	АМГ-10 hydraulic liquid	Test methods
1.Fractional composition, °C: IBP, no less than EBP, no more than	 210 315	 225 302	GOST 2177-99
2.Flash point, °C, in an open crucible, no less than	93.0	114	GOST 4333-2014
3.Freezing point, °C, no more than	- 72	doesn't freeze at 75°C	GOST 20287-91
4.Aniline point, °C, no less than	78.0	79.0	GOST 12339-77
5.Corrosion, testing on Cu		endures	GOST 2917-76
6.Sulfonated components amount, % mas., no more than	1.5	0.0	GOST 6994-74
7.Acid number, mg/KOH gr substance, no more than	0.03	0.006	GOST 5485-76
8.Total amount of sulfur, % mas.	-	0.0257	ASTM D 4294
9. Density, at 20°C	860	839	GOST 3900-85
10.Kinemtatic viscosity, mm^2/s at 50°C at minus 55°C,no more than	 2.2 220	 2.25 185	GOST 33-2016

A technical and economic evaluation of the studied processes was carried out for the purpose of determining the efficiency of the ionic liquid extraction method. For this purpose:

- the cost of the synthesized ionic liquid proposed for use as an extractant was determined;
- a preliminary technical and economic assessment of АМГ-10

HLD single-stage and two-stage selective purification process by ionic-liquid extraction method was carried out.

The cost calculation of obtained target product was carried out according to the methodology applied for this purpose in oil refineries, and the cost of the target product in the single-stage purification with HLD N-MPA containing ionic liquid was 342.27 man./t, but in the 2-stage purification it was 407.10 man.

The cost of the product for the purpose of selective purification by acid-contact method in the presence of HLD acetic anhydride was 135.5 man/t lower than the known purification process by acid-contact method and was 415.12 man/t against 550.62 man/t.

Based on the studies carried out by this way, it was found that N-MPA-containing ionic liquid is more effective in the АМГ-10 HLD selective purification process, the yield of raffinate is significantly higher compared to acid-contact method and is 82.5% mas. to 67.5% mas., the process is ecologically efficient, i.e., acid waste products and sewage aren't practically produced, and is economically efficient (the cost of the extraction process of ionic liquid as an extractant compared to the acid contact method is 208.35 man/t by one-step purification, 143.52 man/t by two-step purification), a technologically simple selective purification method waas developed.

The principle technological scheme of the selective purification process by ionic liquid extraction method developed by HLD is given in Fig.5.

HLD, taken as raw material, is first fed to heat exchanger (2) by pump (1) and heated by the heat of raffinate and transferred to contactor (6). The temperature in the top and bottom parts of the contactor is regulated due to the heat of the extractant and the raw material. Thus, the raffinate removed from the system adjusts the temperature of the distillate supplied to the contactor. Counter current flow principle in the contactor is created by supplying the extracted ionic liquid from the upper part of the contactor at a temperature of 50-60°C, and the counter current flow of the ionic liquid towards the distillate ensures the mixing of the components in the contactor and the selective purification process. The density difference in the raffinate and extract solution obtained from the extraction process ensures that the raffinate is collected in the top of the contactor, but the extract solution in the bottom part of the contactor. The raffinate collected during the process is fed to the mixer (3) from the top part of the contactor.

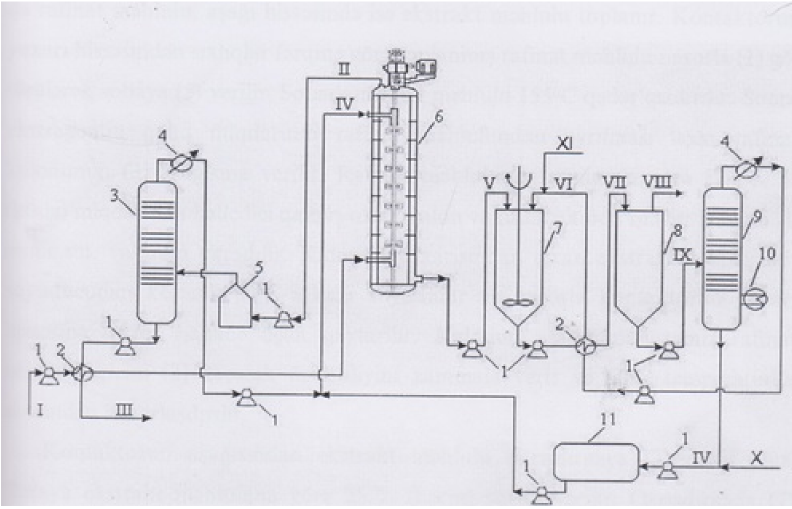


Fig. 5. The principle technological scheme of the АМГ-10 hydraulic liquid distillate purification process using ionic liquid extraction method
1 – pump; 2 – heat exchanger; 3 – raffinate column; 4 – cooler; 5 – heater; 6 – contactor; 7 – mixer; 8 – precipitator; 9 – water distillation column; 10 – reboiler; 11 – extractant capacity.
I – distillate; II – raffinate solution; III – raffinate; IV – extractant; V – extract solution; VI – water; VII – mixture of water and extract solution; VIII – extract; IX – mixture of extractant and water; X – extractant supplied to the system from outside; XI - water supplied to the system from outside.

The extract obtained by the extraction process is transferred to the mixer (7) from the lower part of the contactor (6) and stirred by adding 20-30% water and transferred to the precipitator (8). Phase separation in the precipitator is completed within 30 minutes, and the extract from the components removed during the extraction process from the upper layer distillate and in the lower layer the aqueous solution of the ionic liquid used as an extractant is collected. After the stratification is completed, the water extractant mixture from the lower part of the precipitator is pumped (1) to the water distillation column (9) equipped with a heater.

The water distilled from the mixture passes through the cooler (4), transfers its heat to extraction solution, and returned to the system, mixer (7) for reuse with water from the outside. In this way, the regenerated liquid is removed from the bottom part of the column, collected in the extractant capacity (11) and sent to the contactor for reuse. The extract mixture separated from the distillate is removed from the system by the top of the precipitator.

Thus, HLD purification process by the proposed ionic-liquid extraction method is based on a very simple, easy-to-implement environmentally friendly technology and ensures the development of a refining technology meeting the requirements of "green chemistry".

CONCLUSIONS

1. The HLD obtained by compounding based on the fractions separated from the oils of Balakhany and Neft Dashlary oil fields, was studied by one-step or multi-step selective purification using ionic liquid extraction method, and the conditions for obtaining АМГ-10 hydraulic liquid base product meeting the requirements of TU 0253-021-46698103-2006 were determined [6,8-10,13,15-16].
2. By using N-MPA as an extractant, the amount of ionic liquid in one-step process was 3 times more than the distillate, the components were contacted for 2 hours at a temperature of 60°C, and when the process was carried out by steps - in two or three steps, at equal mas. ratio of the components in each step and 1 hour contact time, complete removal of aromatic hydrocarbons from the content of the distillate was ensured; sulfur content decreased from 884 ppm to 218 ppm [2,6,8-10,13-16,18-19].
3. A similar result was obtained by selective purification process of the distillate obtained by compounding on the basis of the oil

fraction obtained from the fractions separated from the oil of "Neft Dashlary" oil field, using ionic liquid compounds synthesized on the basis of formic and acetic acid as extractants. It was determined that 2.5-3:1 mass ratio of N-methylpyrrolidoneacetate to distillate practically ensures a complete dearomatization, and the distillate obtained on the basis of oil produced from the "Neft Dashlary" oil field is suitable as a raw material for this purpose [2,4,6,8-10,12-16,18-19].

4. It was shown that the residual amount of aromatic hydrocarbons in the raffinate obtained with a mas. yield of 81.6% under similar conditions using ionic liquids containing di- and triethylamine acetate as an extractant in HLD selective purification process is 2% mass [17].
5. Hydrocarbon group composition of the raffinate and extract samples obtained by the extraction process with HLD ionic-liquids, organic solvents of different composition was carried out by IR, UV and chromo-mass spectral analysis methods, and the obtained results revealed effectiveness of the ionic-liquid extraction process [2,5,8,11].
6. The HLD selective purification process was carried out by ionic-liquid extraction process simultaneously with the acid-contact method in the presence of N-methylpyrrolidone and acetic anhydride as a water-absorbing component, which is applied as a selective solvent in the purification of oil fractions on an industrial scale, and ionic-liquid extraction method was determined to be more promising due to the extraction of the target product, regeneration and reuse of an extractant, economic and environmental efficiency [1,3,6-8,10,12,20].
7. The technical and economic calculation of the selective purification process by HLD ionic liquid extraction method was compared with the acid-contact method, and depending on the implementation of the process by one- or multi-steps, the economic efficiency was determined for one-step (207.8 AZN) and two-steps (143.0 AZN) processes [14,20].

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