STUDY OF THE HYDROCARBONS' STRUCTURE-GROUP COMPOSITION OF FRACTIONS UP TO 450°C OF NEW WELL OILS OF THE KHARABAGHLY FIELD

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Abstract: *Samples were taken from new well oils No. 243, 252 and 451 of the Karabakhli field. After complete dehydration by adding demulsifier, certain quantities of samples were prepared, and the compositions of the residues obtained after drying were determined by X-ray phase analysis, provided that metal compounds that can sublimate are not lost. As a result, it was estimated that the dry residues contained V, Ni, Fe, Al, S elements and a small amount of* $C_{30.00}N_{16.00}N_{2.00}$; $S_{8.00}O_{8.00}N_{16.00}C_{36.00}H_{48.00}$; $C_{68.00}H_{128.00}O_{.16.01}N_{16.00}$; $O_{16.00}C_{44.00}H_{40.00}$ *compounds. The oils were extracted in the ARN-2 unit, and 50°C fractions boiling up to 450°C were obtained, their physical and chemical properties were studied, and their structural and group compositions were determined by infrared and proton magnetic resonance spectroscopy analysis methods. According to the densities of the oil samples studied, oil from well No. 243 (*d_{20}^4*= 844.5 kg/m³) is light, while oil from well No. 451 (*d_{20}^4*= 892.4 kg/m³) and oil from well No. 252 (*d_{20}^4*= 900.5 kg/m³) are medium-density oils. It was determined from the yield percentages of the 50°C fractions from those oil samples that well oils No. 252 and 243 contain mostly light-coloured products (kerosene, diesel fuels); well oil No. 451 is rich in oil products. Residual oil products boiling at >450°C constitute ~2.0% in well oil No. 252, ~48.7% in well oil No. 451, and ~78.3% in well oil No. 243. Acidic oxygenated compounds are observed only in the fractions of well oil No. 451 boiling between 300-450°C. The component group-structural composition of the 50°C fractions of the oils was studied using infrared and proton magnetic resonance spectroscopy. It was determined that with increasing temperature, the amount of protons, terminal, and gem-methyl groups in the saturated structures of the components also increases, which indicates the enrichment of the fractions with saturated naphthenic - paraffin hydrocarbons. The results obtained play a significant role in choosing an efficient oil processing method, obtaining high-quality commodity products from it, and assessing the quality of oil.*
Keywords: *oil, fraction, new well oils, light, medium density, components, group-structural composition, infrared, proton magnetic resonance, methyl, gem-methyl groups.*

1. Introduction

Oil and natural gas are important energy sources formed during the geological and biological evolution of the Earth. Due to their long formation period and harsh conditions, oil is generally considered a non-renewable energy source. Oil and natural gas reserves directly or indirectly meet the development needs of countries around the world in various fields [1].

Petroleum engineering involves the application of scientific theories, methods, and technologies to extract oil and gas from underground deposits using modern equipment, and to process these resources to meet the demand for petroleum products and natural gas. Since the start of the global public health emergency, the share of oil and gas consumption and production has declined [2, 3].

However, the global economy is now gradually recovering, which is likely to prompt countries to increase their efforts for oil and gas extraction. Due to the depletion of non-deep reserves, oil and gas extraction is gradually moving deeper into the Earth. The deep subsurface environment is characterized by complex geological structures, high temperature, and high stress conditions, and is of great concern in scientific research and engineering fields [4-6]. Even using currently known methods of increasing oil production, it is possible to recover an average of 10-15% or 30-40 billion tonnes of oil from the remaining oil reserves. Therefore, the residual oil reserves remaining in exploited fields are considered a large reservoir and the application of modern methods for increasing

production is important [7-9]. Oil is a complex organic mixture of hydrocarbons and non-hydrocarbons (heteroatom-containing compounds). Natural hydrocarbons in crude oil mainly include alkanes, cycloalkanes and aromatic hydrocarbons, but unstable olefins and alkynes are very little or absent. After refining, oil contains not only saturated but also unsaturated hydrocarbons. Non-hydrocarbons mainly include organic compounds containing nitrogen, sulfur, oxygen, and metals.

Due to the diversity of types of compounds in oil, the wide range of molecular weights and different polar properties, the complex characterization of hydrocarbons has become a complex issue [10-14]. Complex analysis at the molecular level is one of the leading directions in the petrochemical industry to obtain high-quality, pure and high-value petroleum products. Aromatic compounds constitute a large part of the organic matter in oil and sediment. During thermal transformations of organic substances, methylation, demethylation and regeneration of groups of aromatic compounds are controlled by their thermal stability [15, 16].

The type, structure, and amount of hydrocarbons determine the properties of petroleum products. For example, high-quality gasoline requires more isoalkanes than n-alkanes, since isoalkanes have a higher octane number and a lower cetane number, while diesel fuel has the opposite properties. Olefins affect the stability of petroleum products, and an excess of aromatic hydrocarbons not only causes coke formation, but also causes environmental pollution. Therefore, the amount of olefins and aromatic hydrocarbons must be strictly controlled during petroleum refining [17, 18].

In the field of geochemical research, the characterization of petroleum hydrocarbons contributes to a deeper study of source rocks and allows for a better understanding of their current state and evolutionary history, since biomarkers such as steranes, terpenes, and diamondoids preserve the hydrocarbon structure of their biochemical components and evidence of evolution from living organic matter to sedimentary organic matter. With increasing temperature, high-temperature-resistant aromatic compounds are less likely to undergo the above processes; therefore, their relative abundance gradually increases, while the relative abundance of aromatic compounds with poor thermal stability gradually decreases [19, 20].

Thus, the content of aromatic compounds in oil is a good indicator of its maturation ability. Finally, it should be noted that the complex molecular characterization of hydrocarbon compounds in oil is very important and of great importance [21]. Distillate fractions obtained from oil refineries can facilitate the determination of information about the properties of crude oil. However, a detailed assessment of the chemical composition of crude oil reveals large differences that are directly related to the properties and behaviour of the oil throughout the entire production chain [22, 23].

However, knowing the complete chemical composition of oil and its derivatives at the molecular level remains a challenge for the petroleum industry. In order to better understand the composition of crude oil and reduce its complexity, many researchers have analyzed petroleum fractions over the years [24]. In oil refineries, crude oil can be separated into various distillation fractions, such as naphtha, gases, middle distillates, gas oil, lubricating oils, waxes, and residues, depending on their boiling point and carbon content. One of the main properties of oil and its fractions after distillation is not only the study of their chemical composition, but also the identification of different types of oils and their properties [25].

In the article, as a continuation of previous studies, the study of the structural-group composition of the fractions boiling at temperatures above 350°C, 50°C to 350°C, obtained from new well oils No. 243, 252 and 451 of the Kharabaghly field in the Salyan region of the South Caspian basin, Lower Kur Depression, is considered a key factor in determining the type of oil and choosing the most suitable option for its processing.

2. Experimental part

Demulsification of oils taken from the Salyan Kharabaghly wells, which contain 70% water, was carried out in the following manner:

100 g of oil was poured into the container used for demulsification, one gram of 2% solvent was added to it and mixed for 30 minutes. Then this oil was heated in a water bath for 5 hours, and the amount of water remaining in the oil was determined. It was determined that the amount of water remaining in the oil after demulsification was 0.03%.

Then the oil was separated into 50^0C fractions in the ARN-2 distillation unit. The distillation device for separating oil into fractions was assembled together with a single metal frame [1000x2290x720 mm in size] consisting of technical and electrical blocks at a temperature of 450-500ºC. The device is equipped with BH-461-M or 2HBp-5DM plastic - rotor type or other vacuum pumps providing a residual pressure of 1.3·102 Pa (1 mm Hg) in a continuous operation mode for 16 hours, and 2 cubes with a volume of 1.9-3.0 dm^3. The surface-insulated rectification column with a diameter of 50 mm and a height of 1016 mm has an electroheater and 20 theoretical plates capable of separating the vapor into its constituent parts.

3. Results and discussion

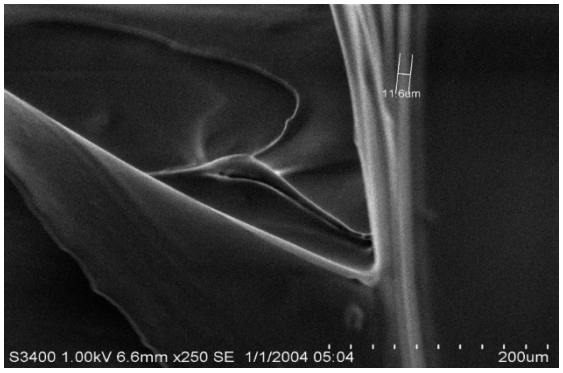
The analysis of the elemental composition of the oils was analyzed by Energy Dispersive X-ray Spectroscopy (EDS) and is given in Fig. 1-4. Oil samples were taken from wells No. 243, 252 and 451 of the Kharabaghly field in the Salyan region and their dried residues were obtained. Their compositions were analysed with a "PANalytical Empyrean" diffractometer and a microscope "Hitachi S-3400 N" atomic analyzer, "OXFOPD Instruments" scanning electron. Through X-ray phase analysis, it was determined that these samples contained 2 "halo" or X-ray amorphous substances in the areas º20 = 36-50º.

Element	Weight, %
C	91.50
O	8.15
Al	0.13
S	0.25
V	0.02
Fe	-0.03
Ni	-0.02
Totals	100.00

Fig. 1. Elements observed in dried residues of well oils No. 243

However, peaks belonging to the crystalline structures are also observed in these compounds, and according to the results of the analysis, these weak peaks are considered to belong to the following compounds:

- In the dry residue obtained from well oil No. 243, $C_{30.00}H_{46.00}O_{.4.00}N_{2.00}$
- (Cyclotoluammonium).(benzoate) indicates the detection of a compound with a triclinic structure;
- In the dry residue obtained from well oil No. 252, $S_{8.00}O_{8.00}N_{16.00}C_{36.00}H_{48.00}$ orthorhombic structures;
- It is estimated that the dry residue obtained from well oil No. 451 contains a small amount of compounds with orthorhombic $C_{68.00}H_{128.00}O_{.16.01}N_{16.00}$ and $O_{16.00}C_{44.00}H_{40.00}$ and triclinic $N_{20.00}C_{240.00}H_{264.00}$ (4-ethunul-phenol)-dimethul-amine structures.



Element	Weight, %
C	97.00
O	2.53
S	0.23
V	0.03
Fe	0.09
Ni	0.12
Totals	100.00

Fig. 2. Elements observed in dried residues of well oil No. 252

Element	Weight, %
C	88.73
O	10.71
Al	0.17
S	0.32
V	0.06
Ni	0.00
Totals	99.99

Fig. 3. Elements observed from dried residues of well oil No. 451

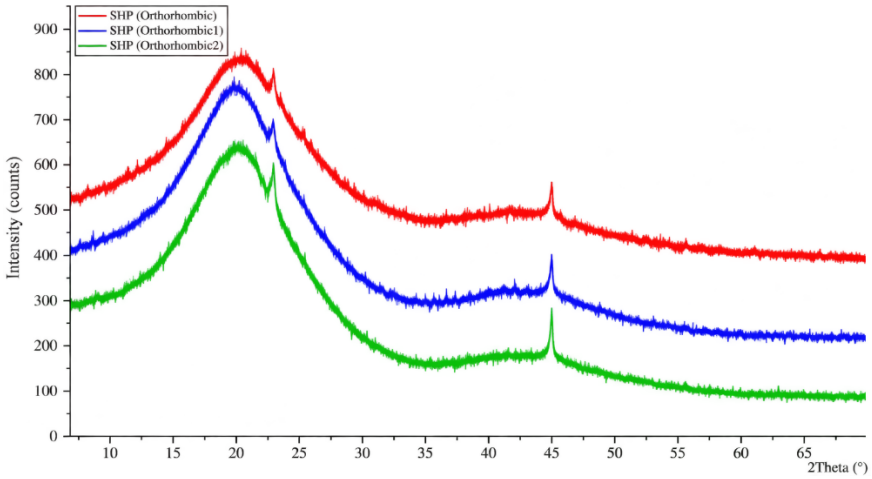


Fig. 4. Types of compounds likely to be found in the dried residues of wells 243, 252 and 451 according to the analysis results

As a result of the combined analysis of SEM (scanning microscope) and atomic force microscope (AFM) in the residues remaining after removal of light fractions from oil samples, provided that sublimely metal compounds are not lost, various elements were found in the composition of the residues. The results obtained show the content of these elements in dry residues in %. The residual weights after evaporation of the samples were taken into account in calculating the content of elements relative to crude oil and are given in Table 1.

Table 1. The content of elements found in the dried residues of oils, in %

Element	Well 243		Well 252		Well 451	
	By Dry Residue	By Oil	By Dry Residue	By Oil	By Dry Residue	By Oil

Al	0.13	0.0122	-	-	0.17	0.0409
S	0.25	0.0235	0.23	0.0431	0.32	0.0770
V	0.02	0.0019	0.03	0.0056	0.06	0.0144
Fe	-	-	0.09	0.0169	-	-
Ni	-	-	0.12	0.0225	-	-

The indicators of the physicochemical properties of the studied oil samples are given in Table 2. The oil samples taken are light oils with densities d_{20}^4= 844.5 kg/m^3 (well 243); d_{20}^4= 0892.4 (well 451) and d_{20}^4=900.5 kg/m^3 (well 252) according to their density.

Table 2. Physicochemical indicators of oils from wells 243, 252, and 451 of the Kharabaghly field.

Well №	Name of indicators				
	Density, kg/m^3, 20°C	refractive index of a beam, η_D^{20}, 20°C	Freezing temperature,, °C	Acid value, mgKOH/g	Iodine value, iodine g/100g
Device name	DMA-4500M	Abbemat - 500	Methodology	Methodology	Methodology
Methodology	ASTM D45002	Methodology	GOST 20287-91	ASTM D3242 GOST 5985	GOST 2070-82
243	844,5	1.5185	+24	-	1.07
252	900,5	1.5121	+9	-	2.59
451	892,4	1.5022	+8	-	2.97

Newly dewatered oil samples from wells 243, 252 and 451 of the Kharabaghly field were distilled in the ARN-2 distillation unit at 450°C according to GOST -II0IV-86: 50°C fractions. The material balance of the obtained narrow fractions is given in Table 3.

Table 3. Material balance of distillation of oils from wells 243, 252 and 451 of the Kharabaghly field into 50°C fractions.

Fractions °C	Well №					
	243		252		451	
	Q	%	Q	%	Q	%
Oil was taken	3.595	100	2163.7	100	3.183	100
200-250	175	4.768	751.2	34.718	360	11.20
250-300	240	6.539	463.3	21.412	260	8.087
300-350	255	6.948	562.7	26.006	85	2.644
350-400	50	1.362	53.4	2.468	300	9.331
400-450	-	-	290.9	13.446	645	20.06
Total distilled	720	19.617	2121.5	98.049	1650	51.322
Residual >450	2875	78.338	42.2	1.951	1533	48.678
Loss	75	2.045	0	0	0	0

As can be seen from the results obtained, the lightest oil from well No. 243 in terms of density was expelled up to 400°C by ~19.62%, while the medium-density oils were expelled up to 450°C: oil from well No. 252 - 98.05%; oil from well No. 451 - 51.32% to 50°C fractions. According to the comparison of the expulsion percentages up to 400°C, oil from well No. 252 - 84.60%, and oil from well No. 451 - 31.26% differ from the others in being richer in the lightest components. In well oil No. 243, components boiling at a temperature of 250-350°C, in well oil No. 252 at a temperature of

200-250°C, and in well oil No. 451 at a temperature of 400-450°C are more abundant than the others in terms of yield. From this it can be seen that well oils No. 252 and 243 are mostly light-coloured (kerosene, diesel fuels); well oil No. 451 is rich in oily oil products. Residual products boiling at > 450°C are ~2% in well oil No. 252; ~48.7% in well oil No. 451; and the highest ~78.3% in well oil No. 243.

The physicochemical parameters of the obtained 50°C fractions are given in Table 4. From the determined parameters, it was determined that components boiling in the range of 400-450°C are almost absent in the oil of well No. 243. In general, there are no or few acidic oxygen compounds in all three of these oils studied, only in the 300-350°C fraction obtained from well No. 451, an acid number of 0.17 was determined; in the 350-400°C fraction 0.01; and in the 400-450°C fraction 0.03 mgKOH/g was determined. With an increase in the boiling point of the fractions, the iodine number and freezing point increase relatively. In particular, with the increase in temperature during distillation, unsaturated hydrocarbons are most commonly observed in the 400-450oC fraction of well oil No. 252 (iodine number = 0.43 g/100g).

Table 4. Physicochemical parameters of 50ºC fractions separated from new well oils of Karabakhli field No. 243, 252 and 451

Well №	Name of parameters				
	Density, in 20ºC, kg/m^3	Refractive index of a beam, η_D^{20}, 20°C	Freezing temperature, °C	Acid number, mgKOH/g	Iodine number, iodine g/100g
Device name	DMA 4500M	Abbemat 500	Methodology	Methodology	Methodology
Methodology	ASTM D45002	Methodology	GOST 20287-91	ASTM D3242 GOST 5985	GOST 2070-82
200-250ºC fr					
243	1.8090	1.4466	Does not freeze at -60ºC	-	0.03
252	0.8052	1.4508	Does not freeze at -60ºC	-	0.03
451	0.8143	1.4369	Does not freeze at -60ºC	-	0.01
250-300ºC fr					
243	0.8175	1.4614	-40	-	0.05
252	0.8270	1.4640	Does not freeze at -20ºC	-	0.04
451	0.8118	1.4524	-45	-	0.03
300-350ºC fr					
243	0.8369	1.4691	-20	-	0.14
252	0.8480	1.4747	-20	-	0.15
451	0.8182	1.4569	-32	- 0.17	0.05
350-400ºC fr					
243	0.8662	1.04691	-10	-	0.28
252	It is solid	1.4960	+15	-	0.41
451	0.8280	1.4615	-25	- 0.01	0.03
400-450ºC fr					
243	-		-	-	-
252	It is solid	1.4917	+10 like paraffin	-	0.43

451	0.8408	1.4705	~6	- 0.03	0.08

The component structural-group compositions of the obtained 50ºC fractions were recorded on an ALPHA IR Fourier spectrometer of the German company BRUKER. As an example, the spectrum of a certain fraction from oil obtained from well No. 243 is given in Fig. 5.

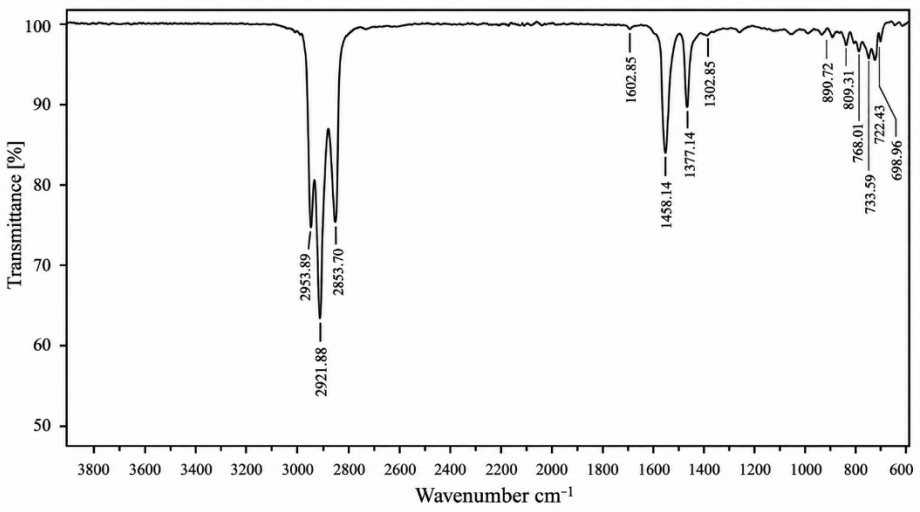


Fig. 5. Well 243 fr.200-250ºC

-698, 739, 769, 809 cm^{-1} - deformation vibration of the C-H bond; -722 cm^{-1} - mathematical vibration of the C-H bond of the CH_2 group; -1302, 1377, 1458, 2853, 2921, 2953 cm^{-1} - deformation and valence vibrations of the C-H bond of the CH_3, CH_2 and CH groups; -1602 cm^{-1} - valence vibration of the C=C bond of the aromatic carbo-hydrogen group HC=C-group;

According to the IR spectrum, the amount of aromatic hydrocarbons, their size, and the valence vibrations of the C=C bond of the ring are determined by the 1600-1605 cm^{-1} bands; the amount of paraffins is determined by the skeletal vibrations of the C-C bond in the long chain -720-727 cm^{-1} bands.

The component group-structural composition of the 50ºC fractions obtained from the oils of wells No. 243, 252 and 451 of the newly commissioned Kharabaghly field, the distribution of protons on individual hydrocarbon groups, was studied by PMR-spectroscopy analysis method. The spectra were recorded on a BRUKER-F Fourier 300.18 MH3 (DCl_3) spectrometer. As an example, the spectra of the 200-250 and 350-400ºC fractions of well No. 252 are given in Fig.s 6 and 7.

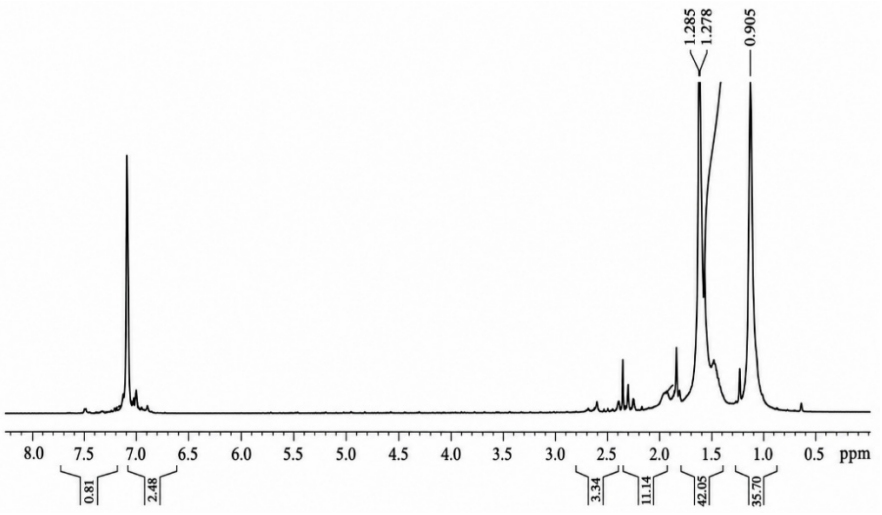


Fig. 6. PMR spectrum of well 252 fr.200-250ºC

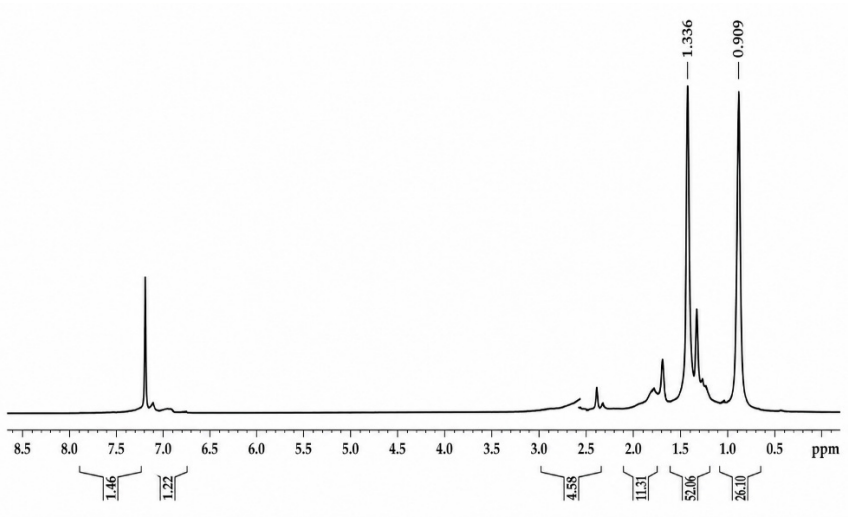


Fig. 7. PMR spectrum of well 252 fr. 350-400ºC

Table 6 shows the identification of the spectra of the samples and the distribution of hydrogen atoms on structural groups, and it was found that the PMR spectra of the 50^0C fractions are almost similar.

Table 6. Distribution of protons in different structural groups of 50ºC fractions of oils.

Fraction, ºC	Relative distribution of hydrogen by structural groups, %					Degree of aromaticity, %	Isoparaffin index
	H_{AZ}	Ha	Hn	Hp	Hγ	f_a	i
Well №252							
200-250ºC	3.44	3.50	11.67	44.02	37.97	11.0	0.57
350-400ºC	2.86	4.72	11.67	53.87	26.97	10.0	0.33

In the PMR spectra, it was determined that the resonance signal recorded at 0.70-1.08 m.h., depending on the boiling points of the 50˚C fractions, belongs to the CH_3 (H) group; 1.08-1.50 m.h. to the CH_2 group (HP); 1.50-2.11 m.h. to the CHn of naphthene rings; 2.11-3.30 p.m. to the CH_2, CH groups located in the α-position relative to the aromatic rings; and the resonance signals observed at 6.75-8.10 ppm. belong to the hydrogen atoms of the aromatic rings. As can be seen from the spectra, the most intense peaks for the 200-250˚C fractions are observed with absorption bands at 0.7-1.1 and 1.1-1.5 mln^{-1}, the first of which, including hydrogen belonging to aromatic rings, belongs to the protons of the methyl group (H), and the second to the protons of the methylene and methine (H) groups.

Relatively weakly intense bands for the fractions are 2.1-2.6 ppm in the 200-250ºC fractions; 350-400; 400-450ºC fractions, which are related to the protons of the groups (H) located in the α-position of the aromatic rings. The main result obtained from the PMR analysis of the fractions is that the amount of protons in the saturated structure of their components is ~91.9-95.31% for the 200-250ºC fractions; ~92.42-92.68% for the 350-400ºC fractions; ~93% for the 400-450ºC fraction.

The presence of terminal groups (CH_3) in the fractional components of the studied oils at 200-250ºC up to 35-41% and with increasing temperature (~28% in the fractional components at 350-400ºC and 400-450ºC), and the presence of ~55% (Hp) in paraffin structures with increasing temperature (up to 450ºC) indicate a high content of terminal and hem-methyl groups, which indicates the enrichment of the fractions with saturated naphthenic paraffin hydrocarbons.

Conclusion

1. Well oils No. 243 and 252 are mostly rich in light-colored fuels (kerosene, diesel), and well oil No. 451 is rich in oil products, so it can be used as a valuable raw material for the production of these products.

2. According to PMR spectra, the isoparaffin index of well oils No. 243, 252 in the 200-250ºC fractions is 0.56; 0.57; in the 350-400ºC fractions is 0.34; 0.33; in the 400-450ºC fraction of well oil No. 451 is 0.33; in the light fraction of 200-250ºC it is 0.66, which is different. The degree of aromaticity was determined differently from the others, being 11.0; 10.0% in the 200-250ºC and 350-400ºC fractions of well oil No. 252.

3. The main result obtained from the PMR analysis is that the amount of protons in the saturated structure of the components of the fractions reaches ~ 92-93% as the temperature increases (from 450ºC), which indicates their enrichment with saturated paraffin-naphthenic hydrocarbons.

Conflict of Interests

The authors announced that there are no conflicts of interest regarding this current work.

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