ГЕОЛОГИЯ, ПОИСКИ И РАЗВЕДКА ТВЕРДЫХ ПОЛЕЗНЫХ ИСКОПАЕМЫХ, МИНЕРАГЕНИЯ

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I.S. Guliev[1], G.S. Martynova[2], O.P. Maksakova[2], R.G. Nanajanova[2], N.I. Velimetova[2], S.G. Zeinalov[2*]
[1]Institute of Oil and Gas, Ministry of Science and Education of Azerbaijan
[2]Institute of Geology and Geophysics, Ministry of Science and Education of Azerbaijan
[*]zeynalov.samir.98@mail.ru

A STUDY OF GEOCHEMICAL FEATURES OF MEDICINAL OILS

Abstract. The article provides data on the geochemical properties of the fields officially recognized as medicinal crude oils in Croatia and Naftalan, Azerbaijan. Studies of hydrocarbon, biomarker and microelement compositions were carried out using the following methods: chromatography-mass spectrometry, mass spectroscopy with inductively coupled plasma, and the combined thermal analysis. Determination of the potential fractional composition of oil was carried out using a synchronous thermal analyzer and showed that medicinal Naftalan and Croatian medicinal crude oil have a similar fractional composition. The hydrocarbon composition and biomarkers analyses – determination of steranes, hopanes and adamantanes in the studied oils – was accomplished on a Clarus 680 Perkin-Elmer chromatography-mass spectrometer. It was defined that the medicinal and fuel oils of Naftalan differ significantly in hydrocarbon composition, while compared with the Croatian medicinal crude oil, the medicinal Naftalan oil is almost identical in hydrocarbon composition and biomarker-geochemical parameters. Using the ICP method, microelement data on the composition of the Croatian medicinal crude oil and the medicinal Naftalan oil was obtained; both of studied oils are of the ferruginous type. The Naftalan medicinal oil quantitatively exceeds the Croatian medicinal crude oil in terms of microelement content. Despite the difference in the genesis and geological structure of the medicinal oil fields in Croatia and Naftalan, Azerbaijan, the study of their hydrocarbon, biomarker and microelement compositions showed undoubted similarity. It can be suggested that the organic matter (OM) involved in the formation of the oils have almost the same origin.

Keywords: medicinal oil, Naftalan, geochemical parameters, hydrocarbon composition, biomarkers, sterane, hopane, adamantane, fractional composition, microelements.

ИССЛЕДОВАНИЕ ГЕОХИМИЧЕСКИХ ОСОБЕННОСТЕЙ ЛЕЧЕБНЫХ НЕФТЕЙ

И.С. Гулиев[1], Г.С. Мартынова[2], О.П. Максакова[2], Р.Г. Нанаджанова[2], Н.И. Велиметова[2], С.Г. Зейналов[2*]
[1]Институт нефти и газа Министерства науки и образования Азербайджана
[2]Институт геологии и геофизики Министерства науки и образования Азербайджана
[*]zeynalov.samir.98@mail.ru

Аннотация. В статье приведены данные о геохимических свойствах месторождений нефтей, официально признанных лечебными – Хорватия и Азербайджан Нафталан. Исследования углеводородного, биомаркерного и микроэлементного составов проводились с использованием следующих методов: хромато-масс-спектрометрии, масс-спектроскопии с индуктивно связанной плазмой и совмещенного термического

анализа. При определении потенциального фракционного состава нефти с использованием синхронного термического анализатора, показано, что лечебный Нафталан и Хорватская лечебная нефть имеют схожий фракционный состав. Анализ углеводородного состава и биомаркеров – определение терпанов, стеранов, гопанов и адамантанов исследуемых нефтей выполнялось на хромато-масс-спектрометре Clarus 680 Perkin-Elmer. Установлено, что лечебные и топливные нефти Нафталана существенно различаются по углеводородному составу, в то время как с Хорватской лечебной нефтью лечебный Нафталан практически идентичен по углеводородному составу и биомаркерно-геохимическим параметрам. С использованием метода ICP получены данные о микроэлементном составе Хорватской лечебной нефти и лечебной Нафталанской нефти, обе исследуемые нефти относятся к железистому типу. Лечебная Нафталанская нефть количественно превосходит Хорватскую лечебную нефть по содержанию микроэлементов. Несмотря на различие генезиса и геологического строения месторождений лечебных нефтей – Хорватии и Нафталана (Азербайджан), изучение углеводородного, биомаркерного и микроэлементного составов показало несомненное сходство. Можно предположить, что органические вещества (ОВ), участвующие в образовании нефтей, имеют практически одинаковое происхождение.

Ключевые слова: лечебная нефть, Нафталан, геохимические параметры, углеводородный состав, биомаркеры, стераны, гопаны, адамантаны, фракционный состав, микроэлементы.

Introduction

Medicinal oils are found in many places in the world; however, oil is officially used for medicinal purposes only in Azerbaijan and Croatia at special medical institutions. In 1989, in Croatia, in the small town of Ivanic Grad, located 30 km south of Zagreb, the balneotherapeutic sanatorium Naftalan was opened, which gained worldwide fame at the end of the 20th century. Rare sources of naphthalan were discovered while drilling an oil well in the Moslavina basin.

The purpose of this work is to consider and compare the properties of the Croatian medicinal crude oil with the Azerbaijani Naftalan medicinal and fuel oil.

The healing properties of Naftalan oil, the deposit of which is located in Azerbaijan in the city of Naftalan, not far from the large city of Ganja, have long been known throughout the world. There is a lot of literature on the origin of Naftalan oil, its genesis, physicochemical and geochemical properties, as well as unique balneological properties [1-7].

Methods and objects of research

The objects of research were the following samples:

- naphthalan oil from Croatian Naftalan Health Center in Ivanic Grad;
- crude oil from the Croatian medicinal oil field;
- medicinal oil from Naftalan field; I-II horizon, filter 151-586 m;
- fuel oil from Naftalan field; III-IV horizon well 1;
- refined naphthalan oil
- white naphthalan oil

The studies were carried out using the following methods: chromatography-mass spectrometry, inductively coupled plasma mass spectroscopy (ICP) and simultaneous thermal analysis (STA).

STA method. Determination of the potential fractional composition of oil was carried out using a synchronous thermal analyzer STA 6000 Perkin Elmer in the temperature range of 20–700°C. The oil samples were heated in a nitrogen atmosphere at a given constant speed from 5–40°C, as a result of which DSC curves were obtained in the coordinates $\Delta H=f(T)$ and TG curves in the coordinates $\Delta m=f(T)$, as well as their derivatives.

Chromatography-mass spectrometry method. Studies of the oils were carried out on a Clarus 680 Perkin-Elmer chromatography-mass spectrometer, which has an interface with a highly efficient mass selective detector Clarus SQ8T. Hydrocarbon chromatograms were obtained by total ion current (TIC) on a 100 m long, 0.25 mm diameter quartz capillary column impregnated with ZB-1MS phase. The analysis conditions were identical. Helium, with the flow rate 1 ml/min, was used as a carrier gas.

Evaporator temperature was at 300°C; programming the temperature rise from 80 to 290°C at a rate of 4°C/min, followed by an isotherm for 70 min. The ionizing voltage of the source was 70 eV, the source temperature was 250°C. Biomarker chromatograms were performed in the SIR, SIM mode. Methylene chloride was used as a solvent. During the study, chromatograms were also recorded on a column with a similar phase – DB – 5 M.

A trace element analysis of the oil samples was carried out on a **NexION 300D inductively coupled plasma mass spectrometer**. The detection limit for almost all elements of the periodic table was at the level of 9.1–10 ppm.

Results and discussion

The STA method was used to study the properties of the Croatian medicinal crude oil. Table 1 presents the quantitative content of fractions in the temperature ranges corresponding to the evaporation of gasoline – G, naphtha – N, kerosene –K, gas oil – G, paraffin –P, oils (light, medium, heavy) –M (1,2,3), resins –R and asphaltenes –Asph.

Data on the fractional composition of the medicinal and fuel Naftalan oil is also provided. As we can see, the oil from the Croatian field is naphthenic-paraffin, and after distillation we are dealing with a fraction (~97 % wt.), with boiling point = 280–425°C, in which light fractions (GNKG) and aromatics, which are toxic at treatment of both the musculoskeletal system and the skin and cause itching, burning and allergic reactions in patients. Medicinal Naftalan and Croatian medicinal crude oil have a similar fractional composition.

Table 1 – Fractional composition of medicinal oils

Таблица 1 – Фракционный состав лекарственных масел

Sample Name	Indicators	Fractions		P+O1, O2, O3	R+Asph
		GN	KG		
Croatian medicinal crude oil	T_1–T_2	b.p. –180°C	180–280°C	280–425°C	425–700°C
	Δ, % (wt.)	9.188	28.979	49.429	11.053
Naftalan medicinal oil	T_1–T_2	b.p. –180°C	180–280°C	280–425°C	425–970°C
	Δ, % (wt.)	8.302	32.098	43.636	16.183
Naftalan fuel oil	T_1–T_2	b.p. –180°C	180–280°C	280–425°C	425–790°C
	Δ, % (wt.)	11.221	40.427	38.603	9.506

Figure 1 presents chromatography-mass spectra of naftalan medicinal and fuel oil and the Croatian medicinal crude oil; Table 2 shows the calculated hydrocarbon composition data.

As you can see, the medicinal and fuel oils of Naftalan differ significantly in hydrocarbon composition, while with Croatian medicinal crude oil, medicinal Naftalan is almost identical in hydrocarbon composition.

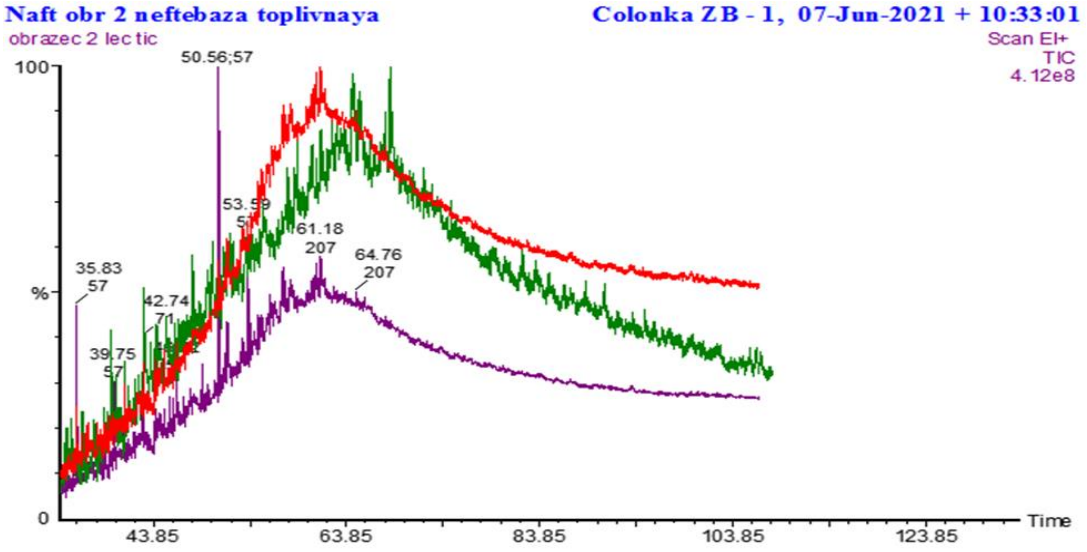


Fig. 1. Chromatogram of Croatian medicinal crude oil sample (green line) and Naftalan oil: medicinal (red line) and fuel (purple line)

Рис. 1. Хроматограмма образцов хорватской лекарственной сырой нефти (зеленая линия) и нафталановой нефти: лекарственной (красная линия) и топливной (фиолетовая линия)

Table 2 – HC composition of oils according to gas chromatography-mass spectrometry data

Таблица 2 – Углеводородный состав масел по данным газовой хроматографии-масс-спектрометрии

Sample name	n-alk %	i-alk %	isopre-noids %	∑ al-kanes	Naphtenes					∑ napht	Arenes			∑ arene
					mono	bi	tri	tetra	penta		mono	bi	tri	
Croatian crude oil	-	10.66	-	10.66	77.46	5.57	-	2.86	2.21	88.1	-	-	-	-
Naftalan medicinal	-	3.53	-	3.53	44.08	35.3	4.31	-	-	83.69	2.33	4.67	5.11	12.11
Naftalan fuel	6.81	16.12	Pr 4.94; Ph -2.11	22.93	41.7	17.14	-	0.53	-	59.37	3.7	1.76	12.02	17.48

In balneological centers of both countries, in addition to native oil, oils and certain fractions – oil distillation products – are used for medicinal purposes.

We examined the composition of naphthalan oil from the Croatian Naftalan Health Center, as well as oil from Naftalan oil: refined and white. Chromatograms of oils and calculation of their hydrocarbon composition are presented in Figure 2 and in Table 3.

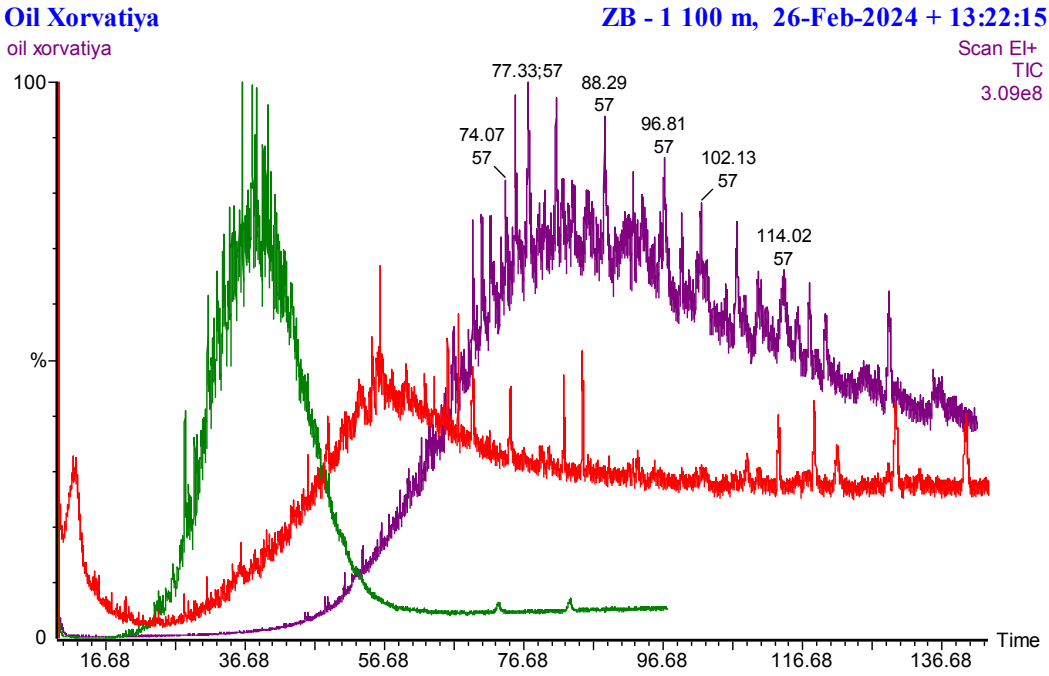


Fig. 2. Chromatogram of naphthalan oil from Croatian Naftalan Health Center (purple line) and from Naftalan oil: (red line – refined, green line – white)

Рис. 2. Хроматограмма нафталанового масла из Хорватского медицинского центра Нафталан (фиолетовая линия) и нафталановой нефти: (красная линия – очищенная, зеленая линия – белая)

Table 3 – Hydrocarbon composition of oils from Naftalan oil

Таблица 3 – Углеводородный состав масел по данным газовой хроматографии-масс-спектрометрии

Samle	n-alk %	i-alk %	isopre-noids %	∑ al-kanes	Naphtenes					∑ napht	Arenes			∑ arenes	diff
					mono	bi	tri	tetra	pen-ta		mono	bi	tri		
naphthalan oil from Croatian Naftalan Health Center	-	71.25	-	71.25	17.23	1.56	-	3.38	5.96	28.13		-	0.62	0.62	-
Refined naphthalan	-	33.85	-	33.85	54.7	3.5	-	3.08	2.44	63.72	-	-	0.72	0.72	-
White naphthalan	-	2.2	-	2.2	93.68	4.18	-	-	-	97.86	-	-	-	-	Tetralines 0.02

The Croatian oil from the Naftalan Health Center has a paraffin base, it corresponds to ~ 71 % isoalkane hydrocarbons; naphthenic hydrocarbons ~28 %. As for aromatic hydrocarbons, they are absent in white oil, and in the other two oils their content is approximately the same.

As can be seen, Naftalan oils have a wide range of naphthenic hydrocarbons ~ from 64 to 98 %; alkane hydrocarbons ~ from 2 to 34 %.

Of interest was a comparative assessment of the catagenetic transformation of medicinal oils based on biomarkers that were obtained in the SIR, SIM mode using gas chromatography-mass spectrometry.

Also presented are mass fragmentograms and calculation data for steranes and hopanes of Croatia oil (Fig. 3-4, Table 4-5).

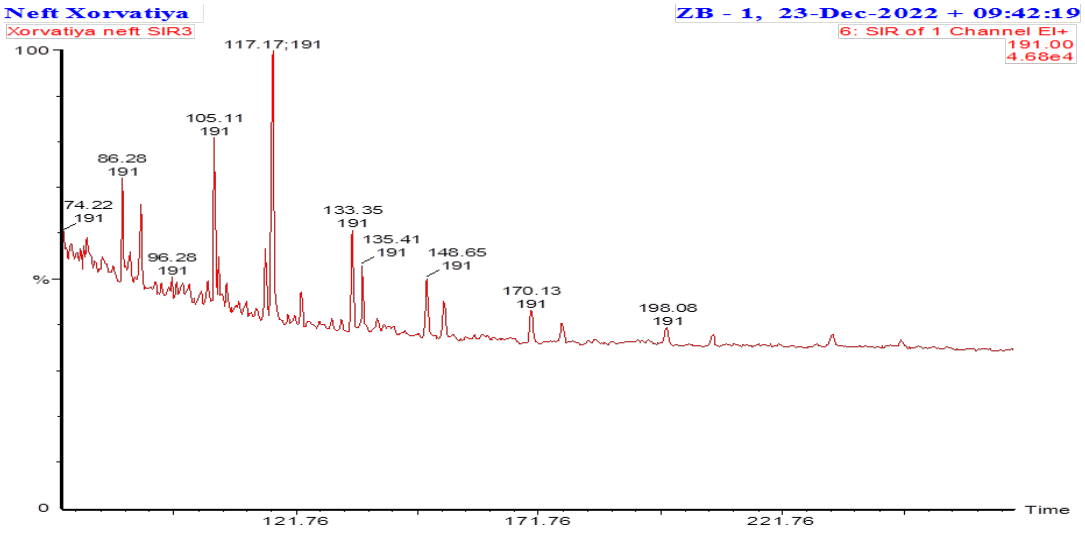


Fig. 3. Mass fragmentogram of hopanes m/z 191 of Croatian medicinal crude oil

Рис. 3. Массовая фрагментограмма hopanes m/z 191 хорватской лекарственной сырой нефти

Table 4 – Hopane content of medicinal oils

Таблица 4 – Содержание гопана в лекарственных маслах

Sample / Hopanes	Naftalan medicinal, %	Naftalan fuel, %	Croatian oil, %
Ts	4.09	4.21	5.43
Tm	4.02	3.46	5.70
H_{28}17αH, 18αH,21βH bisnorhopane	0.77	1.02	1.59
H_{29} Nor- 25- hopane	1.96	1.83	1.90
Adiantane	14.84	10.85	12.20
Diahopane H_{30}	2.2	2.04	2.07
Moretane H_{29}	0.92	1.11	-
Oleanane	7.47	5.64	6.04
Hopane H_{30}	19.19	24.52	25.22
Moretane H_{30}	3.06	3.03	-
Homohopane Hh_{31}	15.63	15.78	14.95
Bishomohopane H_{32}	10.14	10.34	10.16
Trishomohopane Hh_{33}	9.45	7.79	6.21
Tetrakishomohopane Hh_{34}	4.8	5.67	3.9
Pentakis homohopane Hh_{35}	3.35	2.73	2.75
Geochemical indicators			
Ts/Tm	1.01	1.22	0.95
$Hh_{35}/(Hh_{31}$-$Hh_{35})$ Homohopane index	0.08	0.06	0.07
Oleanane/H30	0.4	0.23	0.24
Adiantane/H_{30}	0.77	0.44	0.48

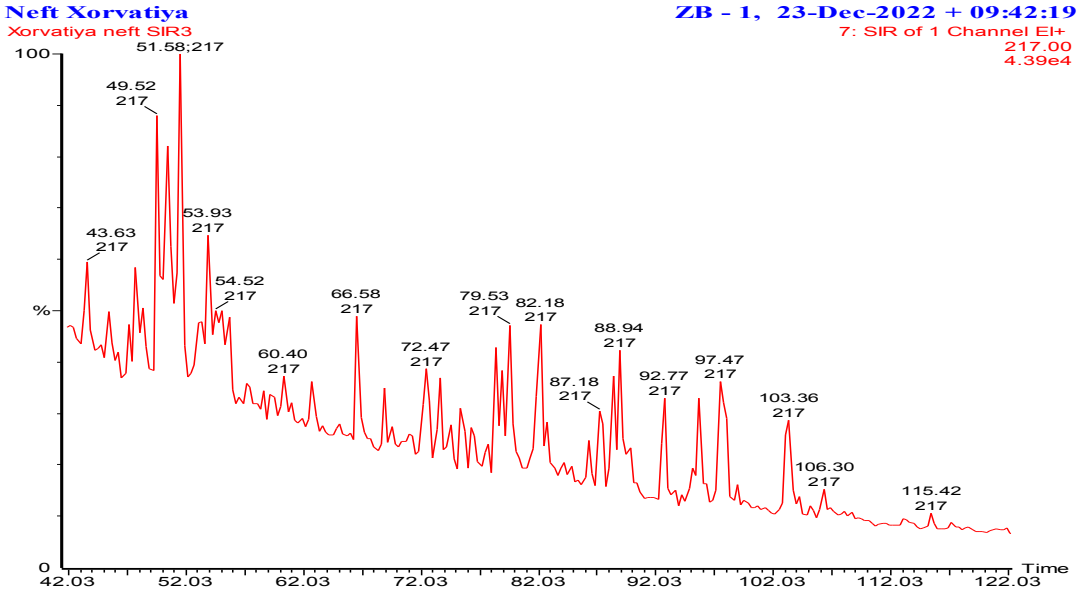


Fig. 4. Mass fragmentogram of steranes m/z 217 of Croatian medicinal oil sample

Рис. 4. Масс-фрагментограмма стеранов m/z 217 в образце хорватского лекарственного масла

Table 5 – Sterane content of Croatian crude oil

Таблица 5 – **Содержание стерана в хорватской сырой нефти**

Steranes	exit time	area	value %	∑ value
St 21 (A)	43.634	3485.785	3.65	∑6.56
St 22 (B)	47.753	2781.651	2.91	
holane (C)	53.931	2854.25	2.99	
dia St (D)	66.582	3797.049	3.98	∑ 18.74
dia St (E)	68.936	1764.897	1.85	
dia St (F)	72.466	5013.369	5.25	
dia St (G)	73.643	2941.292	3.08	
dia St (H)	75.408	2444.316	2.56	
dia St (I)	76.291	1928.22	2.02	
St 27 S1	77.762	1036.209	1.09	∑ 24.13
St 27 R1	78.351	3718.918	3.90	
St 27 S2	78.939	3545.709	3.71	
St 27 S2	79.527	5970.619	6.26	
St 27 R2	82.175	6679.938	7.00	
St 27 R2	82.764	2069.057	2.17	
St 28 S1	86.294	1606.67	1.68	∑ 22.34
St 28 R1	87.177	3456.995	3.62	
St 28 S2	88.354	3760.058	3.94	
St 28 S2	88.942	5701.3	5.97	
St 28 S2	89.825	1686.384	1.77	
St 28 S2	90.119	109.591	0.11	
St 28 R2	92.767	4541.981	4.76	
St 28 R2	93.649	468.964	0.49	
St 29 S1	95.12	1536.747	1.61	∑ 20.96

St 29 R1	95.709	3161.488	3.31	
St 29 S2	97.474	8385.082	8.79	
St 29 S2	98.945	558.988	0.59	
St 29 S2	99.534	234.189	0.25	
St 29 R2	103.358	5524.308	5.79	
St 29 R2	104.241	595.996	0.62	
St 30 S1	105.123	379.826	0.40	∑ 4.28
St 30 R1	106.3	1020.512	1.07	
St 30 S2	108.066	201.035	0.21	
St 30 S2	108.654	184.878	0.19	
St 30 S2	109.242	247.187	0.26	
St 30 S2	113.067	1184.188	1.24	
St 30 R2	115.421	646.663	0.68	
St 30 R2	117.186	223.069	0.23	

Table 6 – Steranes content (m/z 217) in Naftalan oils and Croatian oil
Таблица 6 – Содержание стеранов (m/z 217) в нафталановой нефти и хорватской нефти

Sample / Steranes	Naftalan medicinal, %	Naftalan fuel, %	Croatian crude oil, %
Diasterane	19.41	15.47	18.74
Sterane 27	22.31	22.79	24.13
Sterane 28	17.14	16.63	22.34
Sterane 29	21.6	20.55	20.96
Sterane 21+22	5.37	5.94	6.56
C_{27}: C_{28}: C_{29}	22:17:22	23:17:21	24:22:21

The distribution of C_{27}, C_{28}, and C_{29} sterane hydrocarbons is considered as an indicator of the type of organic matter (OM). The predominance of the C_{29} homologue indicates a large contribution of terrestrial vegetation to the original organic matter, whereas the dominance of C_{27} steranes indicates a significant contribution of zooplankton and algal, finally the dominance of C_{28} indicates a lacustrine environment. These parameters can be used in correlation using a ternary diagram as shown in Fig. [8].

The studied oil samples showed a relatively equal content of all three components, which can be seen as an evidence of mixed types of organic matter [9].

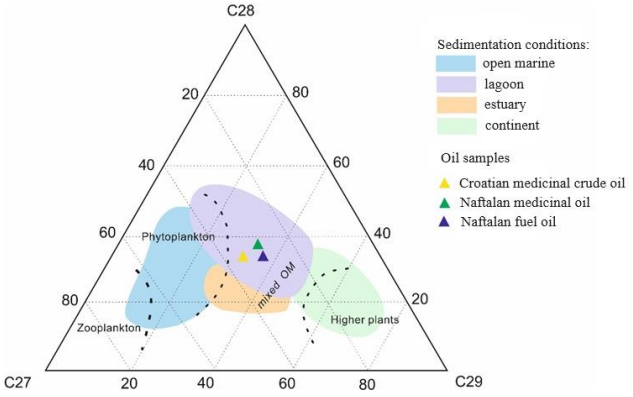


Fig. 5. Triangular diagram of regular steranes C27, C28 and C29 for medicinal oils

Рис. 5. Треугольная диаграмма обычных стеранов C27, C28 и C29 для лекарственных масел

According to the comparative star diagrams of the geochemical parameters of the Croatia medicinal crude and the Naftalan oils (Fig. 6), samples generally have almost the same biomarkers-geochemical features.

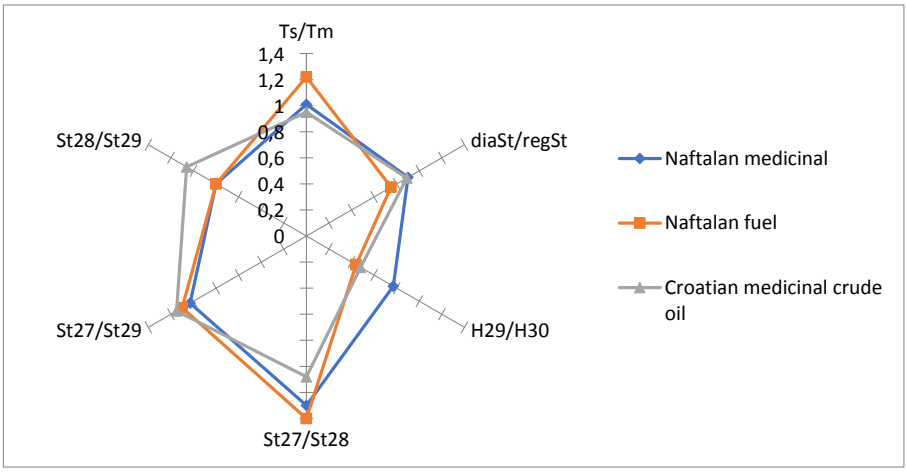
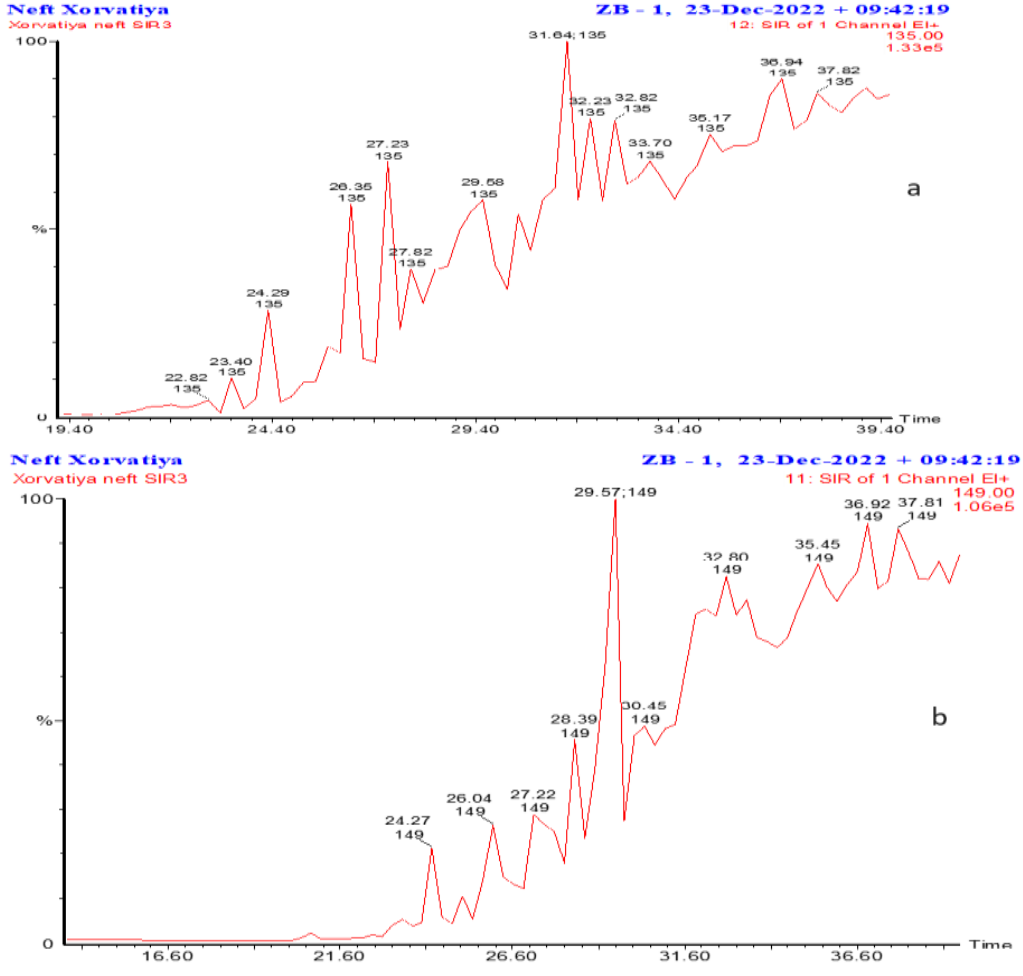


Fig. 6. Star diagram of several biomarker-geochemical parameters of Croatian and Naftalan oils

Рис. 6. Звездообразная диаграмма нескольких биомаркеров-геохимических параметров хорватской и нафталановой нефтей

The identification of adamantoids in samples of the Croatian oil was carried out by chromatography-mass spectrometry by m/z 135 – alkyladamantanes, m/z 149 – dialkyladamantanes and m/z 163 – trimethyladamantanes according to NIST (Fig. 7).



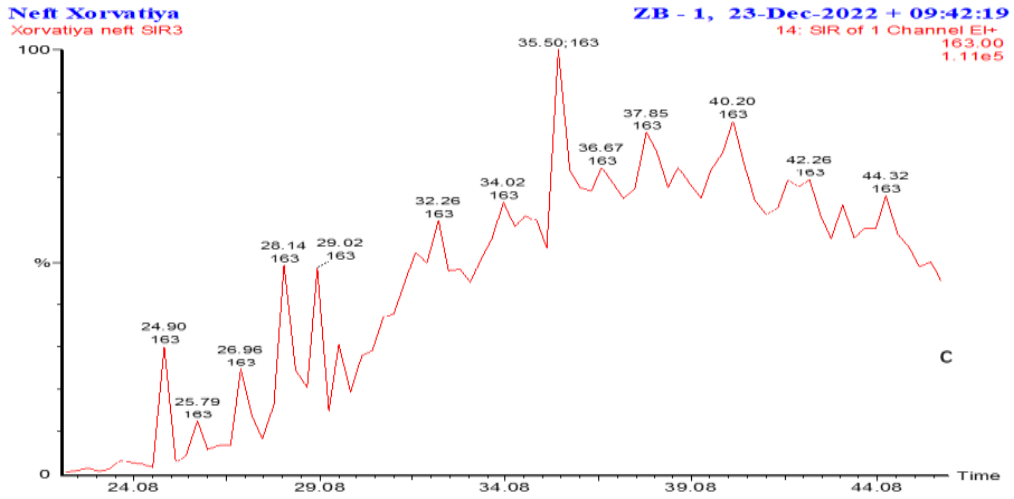


Fig. 7. Chromato-mass spectrum section of alkyladamantanes in Croatian oil
m/z 135 (a), m/z 149 (b) and m/z 163 (c)

Рис. 7. Хромато-масс-спектральный разрез алкиладамантанов в хорватской нефти
m/z 135 (a), m/z 149 (б) и m/z 163 (в)

Table 7 shows the total adamantoid content % in samples of the Croatian and Naftalan oils. The participation of adamantoids in the healing properties of Naftalan oil is already known to us [10]. For this reason, it is necessary to study adamantoids in the Croatian medicinal crude oil and compare it with the Naftalan oil. As can be seen from Table 6, dialkyladamantane is present in a higher percentage than other homologues. The alkyladamantane value of the Croatian medicinal crude oil is 2 times higher than that of the Naftalan oil; however trialkyladamantane is presented in the Naftalan oil in a 5 times higher percentage.

Table 7 – Content of adamantoids in Naftalan oils and Croatian medicinal crude oil

Таблица 7 – Содержание адамантуаза в нафталановой нефти и хорватской лечебной сырой нефти

Sample / Adamantanes	Naftalan Medicinal oil, %	Naftalan Fuel oil, %	Croatian crude oil, %
C11 (alkyladamantanes)	12.9	15.01	24.84
C12 (dialkyladamantane)	65.29	72.04	56.2
C13 (trialkyladamantane)	10.82	11.8	2.16

Table 8 presents the total quantitative content of biomarkers (%) in samples of Naftalan medicinal, Naftalan fuel and Croatian medicinal crude oil. The role of biomarkers in the therapeutic properties of Naftalan oil has already been established. Thus, the sharp difference in the biomarker content in fuel oil and the decrease in biomarker content in the oil used for therapeutic purposes for 1 year are proof of this [10]. As it is clearly seen that, the Croatian medicinal crude oil has 2.7 times less biomarker content than the medicinal Naftalan oil; however, the Croatian medicinal oil surpasses fuel oil in this indicator by almost 2 times. Based on the total percentage of hopanes, the Croatian medicinal crude oil and medicinal Naftalan oil are characterized by almost similar values. Yet the steranes of the Croatian medicinal crude oil are much higher than Naftalan oils.

Table 8 – The comparative content of biomarkers in Croatian crude oil and Naftalan oils
Таблица 8 – Сравнительное содержание биомаркеров в хорватской сырой нефти и нафталановой нефти

Samples	Triterpanes	Hopanes	Steranes	Adamantanes	Σ %, in oil
Naftalan medicinal	1.280	0.470	0.160	5.70	7.60
Naftalan fuel	0.250	0.040	1.07	0.020	1.60
Croatian crude oil	0.014	0.543	1.83	0.34	2.73

Using the ICP method, microelement data on the composition of Croatian medicinal crude oil and medicinal Naftalan oil was obtained, which is tabulated and shown in a histogram (Tab. 9, Fig. 8). As you can see, the Croatian medicinal crude oil is of the ferruginous type, and the Fe content is two times less than in the Naftalan medicinal oil. In second place is cobalt, the content of which is much higher than in the Naftalan medicinal oil. Next in quantitative content are strontium (Sr), titanium (Ti) and nickel (Ni). Comparing the quantitative content of microelements in the Croatian and Naftalan medicinal oils, their predominance in the Naftalan oil was noted (with the exception of cobalt).

Table 9 – Comparing the quantitative content of microelements in medicinal oils
Таблица 9 – Сравнение количественного содержания микроэлементов в лекарственных маслах

№	Sample	As g/t	Co g/t	Cr g/t	Cu g/t	Fe g/t	Mn g/t	Ni g/t	Pb g/t	V g/t	Zn g/t	Sr g/t	Ti g/t	Mo g/t	Cd g/t
1	Croatia oil	0,013	2,195	0.518	0,059	9,864	0,538	0,779	0,009	0,165	0,194	3,469	0,884	0,010	0,001
2	Naftalan medici-nal	0	0,068	1,056	0,294	17,907	5,396	2,659	1,586	0,062	1,653	4,191	1,918	0,172	0

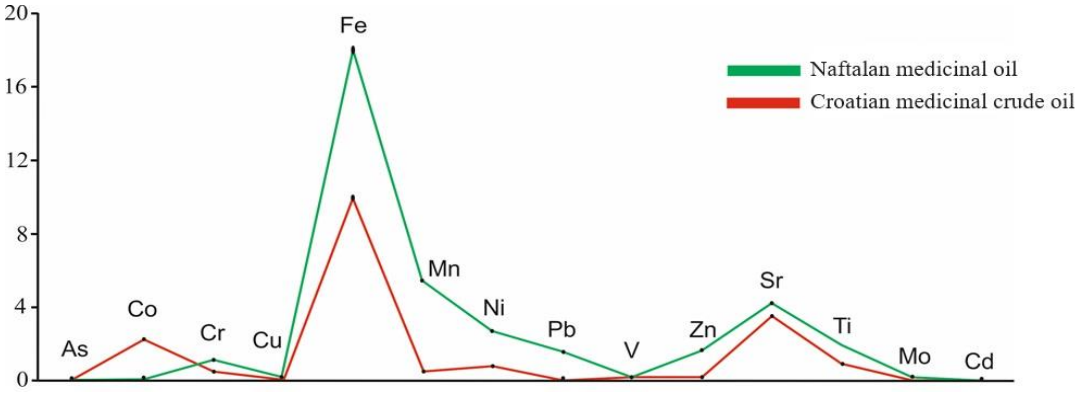


Fig. 8. Comparative diagram of the microelement composition of medicinal oils

Рис. 8. Сравнительная диаграмма микроэлементного состава лекарственных масел

Conclusion

Studies of the medicinal crude oils from Croatia and Azerbaijan using chromatography-mass spectrometry, inductively coupled plasma mass spectroscopy and combined thermal analysis showed that

• the medicinal oil from Naftalan field and the Croatian medicinal crude oil have a similar fractional and hydrocarbon composition

• as for the biomarker indicators, the studied oils generally have almost the same biomarkers-geochemical features

• the microelement composition of the medicinal oils showed that they belong to the ferruginous type

• a study of the genetic origin of organic matter showed that the medicinal oil samples demonstrate relatively equal contents of all three components C27 – cholestane, C28 –methylcholestane and C29 – ethylcholestane, which is evidence of mixed sedimentation.

Thus, despite the difference in genesis and geological structure of the medicinal oil fields in Croatia and Naftalan, Azerbaijan, the study of hydrocarbon, biomarker and microelement compositions showed an undoubted similarity of the studied medicinal oils. It can be suggested that the organic matter (OM) involved in the formation of oils have a similar origin. It is necessary to investigate the relationship between the healing properties and the received geochemical indicators with geological processes: sedimentation conditions, migration processes, and geological structure of the oil fields.

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Сведения об авторах

GULIYEV Ibrahim Said oglu – Academician, Vice-President of ANAS, Baku, AZ, e-mail: ibrahim.guliyev@science.az

ГУЛИЕВ Ибрагим Саид оглы – академик, вице-президент НАНА, Баку, AZ, e-mail: ibrahim.guliyev@science.az

MARTYNOVA Galina S. – Doctor of Physical and Mathematical Sciences, Head of the Laboratory of Petroleum Geochemistry, Institute of Geology and Geophysics, Ministry of Science and Education of the Republic of Azerbaijan, Baku, e-mail: martgs@rambler.ru

МАРТЫНОВА Галина Сергеевна – доктор физ.-мат.наук, Зав. лабораторией «Геохимия нефти» Институт «Геологии и геофизики» Министерство науки и образования, Баку, AZ, e-mail: martgs@rambler.ru

MAKSAKOVA Olga P. – Senior Researcher, Laboratory of Petroleum Geochemistry, Institute of Geology and Geophysics, Ministry of Science and Education of the Republic of Azerbaijan, Baku, AZ, e-mail: olya4747@mail.ru

МАКСАКОВА Ольга Петровна – старший научный сотрудник лаборатории «Геохимия нефти» Институт «Геологии и геофизики» Министерство науки и образования, Баку, AZ, e-mail: olya4747@mail.ru

NANAJANOVA Rahilya Gyulali gizi – Senior Researcher, Laboratory of Petroleum Geochemistry, Institute of Geology and Geophysics, Ministry of Science and Education of the Republic of Azerbaijan, Baku, AZ, e-mail: raxile_skorpion@inbox.ru

НАНАДЖАНОВА Рахиля Гюльали гызы - старший научный сотрудник лаборатории «Геохимия нефти» Институт «Геологии и геофизики» Министерство науки и образования, Баку, AZ, e-mail: raxile_skorpion@inbox.ru

VELIMETOVA Naida Iskender – Engineer, Laboratory of Petroleum Geochemistry, Institute of Geology and Geophysics, Ministry of Science and Education of the Republic of Azerbaijan, e-mail: v.naida.v@gmail.com

ВЕЛИМЕТОВА Наида Искендер – инженер лаборатории «Геохимия нефти», Институт геологии и геофизики Министерство Науки и Образования Азербайджанской Республики, e-mail: v.naida.v@gmail.com

ZEYNALOV Samir Gadir oglu– PhD student, Junior Researcher, Laboratory of Petroleum Geochemistry, Institute of Geology and Geophysics, Ministry of Science and Education of the Republic of Azerbaijan, e-mail: zeynalov.samir.98@mail.ru

ЗЕЙНАЛОВ Самир Гадир оглу – докторант, м.н.с. лаборатории «Геохимия нефти», Институт геологии и геофизики Министерство Науки и Образования Азербайджанской Республики, e-mail: zeynalov.samir.98@mail.ru