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Composition of Mono-, Bi- and Tricyclic Aromatic Hydrocarbons in Oils from the Middle part of the East Siberia

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Aromatic hydrocarbons composition of oils was determined by the GC-MS method. Analysis of the results showed that there is a wide variety of alkyl-substituted benzenes, naphthalenes, fluorenes and phenanthrenes. Relative concentrations of special types of arenes varies in different oils; but in the most part of oils alkylbenzenes are the most representative type of arenes.

Keywords: areny, biomarker, East Siberia, oil, aromatic hydrocarbons.

Introduction

As usual aromatic hydrocarbons (HC) are the part of all crude oils; they present in oil in some definite concentrations. The properties of oil and the ways of oil refining depend on the quantity of aromatic HC in crude oil. Oil arenes are investigated in details; the results are reported in numerous papers but as usual all articles are concerned with the western parts of Russia including the territory of the West Siberia. In spite of the East Siberia has a sizeable discovered recoverable reserve of HC and famous oil pipeline "East Siberia – Pacific Ocean" is constructed in this area, the data of arenes concentrations in the East Siberia oils are quiet limited.

The information of linear alkylbenzenes C17-C23 in three types of crude oil of Nepa-Botuobinskaya oil-and-gas bearing region (OGBR) is presented in [1]. These oils mostly contain linear alkylbenzenes with an odd number of C atoms [1]. In papers [2, 3] the data of arenes content in oil and natural bitumens of Devonian deposits in Khakassia are presented. It is shown that 3-methylalkylbenzenes with isoprene chain C4-C13 are prevalent in oil arenes and polyphenyles (polycyclic aromatic HC with 3-4 phenyl radicals) are found in bitumens [2, 3].

Paper [4] applies to the detailed investigation of aromatic HC in the Jurassic and the Paleozoic oils of the West-Siberian oil-and-gas province

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(OGP) which is on the edge of the East Siberia. It was shown that the major part of aromatic HC in these oils is mono- and bi-arenes [4]. In the course of oil thermal catagenesis the concentration of mono-arenes is reduced but relative content of bi- and tri-arenes is increased [4].

The data of individual and group composition of aromatic HC in oils from the main oil-and-gas bearing formations of the Middle part of East Siberia are discussed in this paper.

Samples and methodology

Oil fields in the East Siberia are found within Riphean, Vendian and Cambrian deposits and they are almost the oldest ones on the planet. Typical oils from three of the main East Siberian OGBRs (Nepa-Botuobinskaya, Baikitskaya and Katangskaya) were investigated in order to get special characteristics of aromatic HC composition in them. Geographically this investigated area is situated within Krasnoyarsk and Irkutsk Regions and Sakha-Yakutia Republic; the investigated area covers the so called "gold belt" of Lena-Tungusskaya OGP.

Nepa-Botuobinskaya OGBR is situated in the south-eastern part of Lena-Tungusskaya OGP and it is part of the anticlinal folding of the same name. Rocks of Vendian terrigene complex, overlying rocks of Vend-Cambrian carbonate complex and Lower Cambrian ($Є_1$) carbonates of Osinskiy productive Horizon of Usolskaya Formation are productive within the OGBR. Numerous showings of oil and gas are discovered in Lower-Cambrian carbonate-saliferous structures including the upper parts of Usolskaya, and full Belskaya, Bulaiskaya, Angarskaya Formations and their analogs.

Nepa-Botuobinskaya OGBR on its west part is contiguous with Katangskaya OGBR, which covers the peripheral south-eastern part of Tungusskaya syneclise and Katangskaya saddle. Baikitskaya OGBR is situated in the east part of Lena-Tungusska OGP and tectonically it is the part of the anteclise of the same name. Carbonates of Ryphean (R) complex and terrigene-carbonate Vendian complex are productive in these structures.

Alkanes composition was determined by the gas-liquid chromatography method according to the internal standard (STP IG-023-05) in a Hewlett Packard chromatograph with flame-ionization detector; helium was used as carrier gas. Capillary column «J&W Scientific» with fixed phase DB-1 (column length 50m, inner diameter 0,25mm, film thickness of fixed phase 0,25μm) was used for the compounds separation. Detection mode: linear programming of temperature 3 degrees/ minute from initial temperature 80˚C until 320˚C, then isothermal conditioning until complete cleaning of the column. The temperatures of the detector and of the evaporator are 310 and 320˚C, respectively.

Aromatic HC were extracted from oils by adsorption chromatography at alumina (IV class of adsorption activity according to Brockman scale) with hexane as eluent. Their composition was determined by gas chromatography with a mass-spectrometric detector (GC-MS) at quadrupole chromatograph-mass-spectrometer «NERMAG R-10-10 C» (France). Compounds separation was realized in a chromatographic capillary column «Supelco» with fixed phase CPB-5 (the column length 30m, inner diameter 0,32mm, film thickness of fixed phase 0,25 μm), carrier gas – helium, input pressure 0,5atm, evaporator temperature 250˚C. The program of thermostat heating: $T_{initial}$ =80˚C, isothermal conditions during 20 minutes, then heating 4 degrees per minute until 280˚C with the next isothermal conditioning until complete cleaning of the column.

Individual compounds were identified by the mass-spectra. Published in different papers spectrum-structural correlations were used for

this identification. Also a special electronic library of mass-spectra "WILEY" with more than 130 thousand titles was used for the identification of HC, so as additional different information about chain-length distribution of HC is published in [6, 7].

Physical and chemical properties of oils (density, sulfur content, fractional and group composition) were determined according to the standard techniques (Russian National Standards GOST 3900, GOST 2177, [5], internal attested standard method STP IG-022-05).

Results and discussion

Oils from 8 areas of Riphean-Vendian-Cambrian deposits from test and exploration drill boreholes were investigated (Table 1).

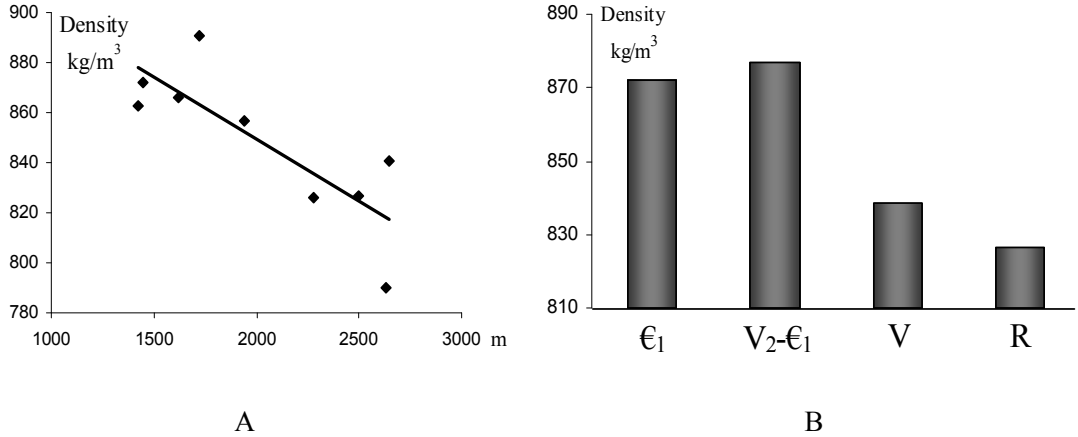
The most number of investigate oils according to the Russia National Standard (GOST P 51858-2002) may be classified as low-sulfur ones (total content of sulfur is less than 0,17-0,54). Oils from Middle-Botuobinskaya and Jelindukonskaya areas are the exceptions. The first one is high-sulfur oil and the second one is middle level sulfur oil. The minimal content of sulfur is in oils from Riphean strata.

According to the standard classification by density the oils from Nepa-Botuobinskaya OGBR are middle type oils, but oils from Baikitskaya and Katangskaya OGBR are light and especially light ones. The density of oils is going down with the depth of the field and the age of enclosing deposits (Fig. 1).

All investigated oils are methane-naphthenic ones according to the group composition of their HC. The content of saturated HC (Me-N) in these oils is 54-80 % and it is growing with the age of enclosing deposits but the content of tars and pyrobitumens is going down while the age of enclosing deposits is increasing (Fig. 2). The maximum content of naphthene-aromatic (N-Ar) HC is in Vendian-Cambrian oils of Nepa-Botuobinskaya OGBR (Table 1).

According to their alkanes composition, all investigated oils may be classified as ones of chemical type A1 [8]; n-alkanes are predominating over alkanes with isoprenoid type of molecule. The chain-length distribution is unimodal with the maximum on C_{15}-C_{17}. Pristane-phytane ratio (Pr/Ph) indicates that the original oil productive organic matter of all investigated oils was generated in reducing conditions (Pr/Ph < 1, 2). The oil from Riphean Vingoldin Formation of Baikit OGBR is the single exception (Pr/Ph = 1, 5); its Pr/Ph ratio indicates sub-oxidizing condition while initial organic matter generation.

Total content of mono-, bi- and tricyclic aromatic HC, which was detected by GC-



A B

Fig. 1. Correlation between oil density and its depth of occurrence (A) and the age of enclosing deposits (B)

Table 1. Data for the oils from the East Siberia

№ of oil	Area	Well number	Depth, m	Formation	Productive Horizon	Age of enclosing deposits	Density, kg/m^3	Content, %				
								Sulfur	Me-N HC	N-Ar HC	Tars	Pyrobitumens
Nepa-Botuobinskaya OGBR												
1	Kugasskaya	364-0	1315-1345,4	Olyokminskaya	Birkinsky	$Є_1$	No data					
2	Middle-Botuobinskaya	25	1425-1452	Bilirskaya	Osinsky	V_2-$Є_1$	872	1,1	54,0	16,1	26,7	3,2
3	Upper-Vilyuchanskaya	677	1709-1722	Yuryakhskaya	Yuryakhsky	V_2-$Є_1$	891	0,5	56,1	22,7	20,4	0,9
4	Upper-Chonskaya	55	1604-1620	Uspunskaya	Preobrajensky	V_2-$Є_1$	863	0,3	61,2	23,5	15,4	0,00
5	Tas-Yuryakhskaya	560	1939-1946	Byukskaya	Botuobinsky	V	857	0,5	63,8	19,1	14,7	2,2
6	Upper-Chonskaya	96	1612-1620	Parshinskaya	Khamakinsky	V	866	0,4	60,2	20,8	18,8	0,2
Katangskaya OGBR												
7	Jelindukonskaya	103	2587-2638	Vanavarskaya	I, IV	V	790	0,6	80,6	9,6	9,6	0,2
8	Sobinskaya	14	2643-2652	Vanavarskaya	II	V	841	0,3	71,6	13,1	15,3	0,0
Baikitskaya OGBR												
9	Yurubchonskaya	8	2271-2284	Yurubchonskaya	I	R	826	0,2	73,9	16,5	9,6	0,0
10	Yurubchonskaya	108	2500	Vingoldinskaya	I	R	827	0,2	72,6	15,7	11,6	0,0

Note: $Є_1$ – Lower Cambrian, V_2-$Є_1$ – Upper Vendian-Lower Cambrian, V – Vendian, R – Riphean, Me-N = methane-naphthenic, N-Ar = naphthene-aromatic, HC – hydrocarbons.

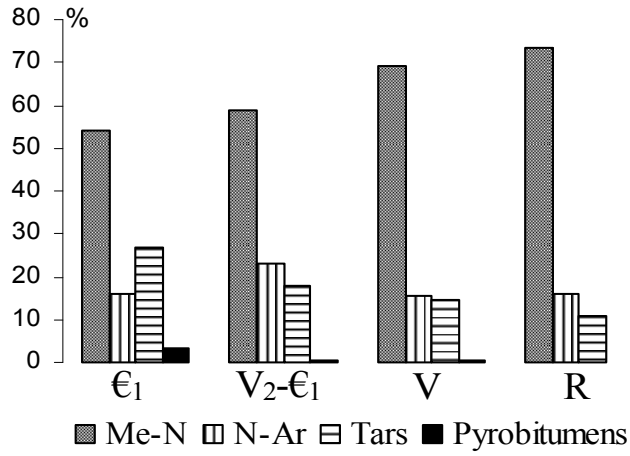


Fig. 2. Group composition of HC in oils from deposits of different age

MS technique, relative to the concentration of n-alkanes fluctuates from 6 % up to 34 %; it is maximum (15-34 %) in oils from Nepa-Botuobinskaya OGBR but it is much lower in oils from Katangskaya OGBR (5-16 %) and in oils from Baikitskaya OGBR (6-11 %). It is correlated with the data on the group composition of HC (Table 1). The analysis of the HC group composition showed the higher concentration of naphthene-aromatic structures in oils from Nepa-Botuobinskaya OGBR. The oil from Vendian-Lower Cambrian strata from Nepa-Botuobinskaya OGBR is characterized by higher concentrations of arenes.

Compounds of benzene, naphthalene, fluorene and phenanthrene types are identified in mono-, bi- and tri-cyclic aromatic HC of investigated oils. Monoaromatic HC are predominating in the major part of oils; but the oil from Sobinskaya area has 11 % of condensed arenes only (Fig. 3). The oil numbers 4, 5, 9 (see Table 1) are the exclusion of this rule; naphthalenes are predominating in these oils. Comparatively to all the other aromatic HC, fluorenes are presented in much lower concentrations (not more than 2 %, see Table 2). The maximum concentration was registered in the oil from Lower Cambrian strata (Kugasskaya area). The concentration of phenanthrenes in the most part of oils is 14-23 relative %; but it is much lower in oils from Sobinskaya and Middle-Botuobinskaya areas.

Monoarenes. Homological series of alkylbenzenes with normal composition and with one substituent radical (n-AB) are identified among monoarenes; this substituent is usually a linear aliphatic chain with 10-30 atoms of C (C_{10}-C_{30}). Also isomeric ortho-, meta- and para- methylalkylbenzenes (MAB), dimethylalkylbenzenes (DMAB) and trimethylalkylbenzenes (TMAB) were identified. Phytanilbenzene (PhB) (C_{26}) with isoprenoid aliphatic chain C_{20} and its methyl- and dimethyl-substituted homologs were identified in all oils. High relative concentration of PhB is in oil from Lower Cambrian deposits; all the other oils have from 0,04 to 2,1 % relative of PhB (Table 2). Methyl-phytanilbenzene is in the highest concentrations in oils comparatively with all others PhB-s.

DMAB is predominating in the monoarenes mixture in oils from Riphean deposits; their concentration is increasing up along a stratigraphic cross-section while the concentrations of TMAB are going down and the concentrations of MAB and n-AB are increasing. However in the top point

Table 2. Mono- and biarenes distribution in oils

N of oil *	Alkyl-benzenes**	Content of alkylbenzenes in mixture, % relative					Naphtha-lenes**	Content of naphthalenes in mixture, % relative						Fluo-renes**	Content of fluorenes in mixture, %relative		
		n-AB	MAB	DMAB	TMAB	PhB		N	MN	EN	DMN	TMN	TeMN		F	MF	DMF
1	45,5	12,5	27,3	32,3	13,9	13,9	29,5	0,0	4,7	6,7	15,0	33,3	40,3	2,1	11,3	28,8	59,9
2	54,1	31,2	35,1	23,0	8,2	1,4	41,1	3,4	18,7	1,5	38,2	37,7	0,5	0,3	5,5	4,8	89,8
3	46,6	30,0	37,6	24,7	6,9	0,7	31,7	2,9	16,9	1,4	26,5	33,9	18,3	0,1	2,5	7,2	90,3
4	31,3	23,4	33,3	35,0	7,8	0,4	46,8	0,3	4,7	0,5	22,4	44,2	27,9	0,3	3,5	13,9	82,5
5	28,8	24,3	31,9	32,2	11,0	0,5	48,8	0,9	7,6	0,9	21,4	45,3	23,9	0,6	25,9	26,8	47,3
6	42,9	20,2	34,2	33,2	11,8	0,6	36,1	0,9	6,3	0,5	14,6	46,8	31,0	0,3	15,5	9,8	74,7
7	52,3	27,0	33,3	26,8	12,3	0,6	25,6	2,9	9,9	0,9	17,3	38,1	30,9	1,3	17,5	21,4	61,2
8	89,5	20,2	38,3	37,0	3,9	0,5	6,5	4,5	19,5	0,0	32,8	38,0	5,2	0,1	1,3	1,4	97,4
9	39,0	11,5	32,2	40,4	13,7	2,1	46,3	0,8	10,9	0,9	22,4	47,8	17,2	0,9	13,2	54,5	32,3
10	55,0	18,5	31,4	40,3	9,7	0,04	25,5	1,8	15,4	0,3	29,6	36,3	16,5	0,8	23,7	41,9	34,4

Note: *Number of oil – see Table 1, ** – total content of appropriate group in a mixture of identified aromatic compounds, AB – alkylbenzene, n-AB – normal alkylbenzene, MAB – methylalkylbenzene, PhB – phytanilbenzene, N – naphthalene, MN – methylnaphthalene, EN – ethyl naphthalene, DMN – dimethylnaphthalene, TMN – trimethylnaphthalene, TeMN – tetramethylnaphthalene, F – fluorine, MF – methylfluorene, DMF – dimethylfluorene.

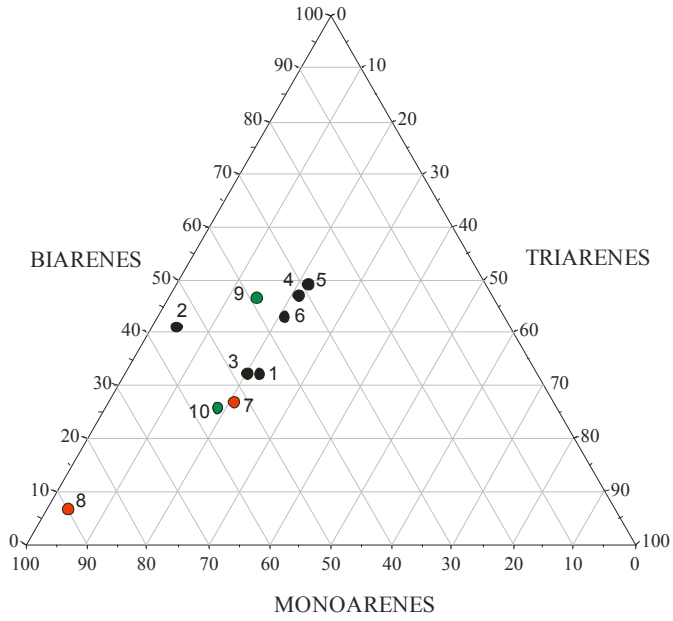


Fig. 3. Content of specified groups of aromatic HC in oils from the East Siberia. Sample number see Table 1

of the cross-section (Lower Cambrian zone), DMABs are predominating in oil. The content of PhB in this oil is more than the content of n-AB (Fig. 4).

N-ABs in the majority of oils form a wide series which include homologs C_{10}-C_{30}. In oils from Nepa-Botuobinskaya OGBR the maximum distribution is on C_{17} and C_{21}, mostly on C_{21}. In oils from Baikitskaya OGBR the main maximum is on C_{11}-C_{13}, but in oils from Katangskaya OGBR homologs with odd number of C are predominating in n-AB along the whole family of them.

Biarenes. Identified biarenes include naphthalene, fluorene and their alkyl-substituted homologs which have up to two methylene groups (fluorenes) and up to four methylene groups (naphthalenes) in their side alkyl chains.

The content of appropriate naphthalenes in oils from Lower Cambrian strata is growing up while the quantity of methylene groups in side substituents of aromatic rings is increasing. Naphthalenes with four atoms of C in side alkyl chain (TeMN) are in the maximum concentration but naphthalene without substituents was not identified in this oil (see Table 2). Going down along stratigraphic cross-section one can find similar concentrations of compounds with two (DMN) and three (TMN) atoms of C in oils from the top of nonsegmented Vend-Cambrian Stratum (Usolskaya Formation); naphthalene (N) and methylnaphthalene (MN) have high relative concentrations in these oils. TMN is the main compound in more aged oils. The oils from Katangskaya OGBR have the biggest concentrations of unsubstituted naphthalene. The distribution of different types of naphthalenes in oils shows that the low total concentration of naphthalenes in oil from Sobinskaya area is mainly because of a low concentration of TeMN in this oil.

Methylfluorenes are predominating in fluorenes (F) of Riphean oils from Baikitskaya OGBR (Table 2). Dimethyl-substituted fluorenes (DMF) are predominating in oils from more recent deposits. Dimethyl-substituted compounds content is growing while relative content of monomethyl-substituted compounds (MF) is going down. High concentration of unsubstituted

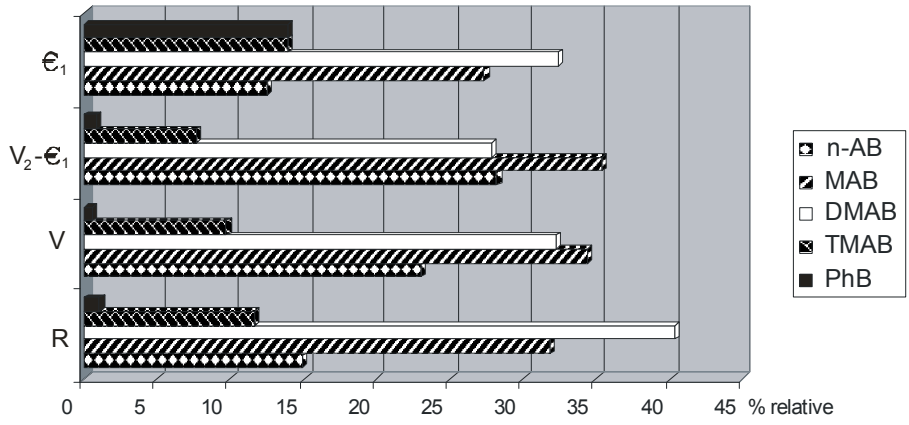


Fig. 4. Distribution of some groups of alkylbenzenes in oils of different ages

fluorene is characteristic of oils from Vendian strata from Nepa-Botuobinskaya OGBR and from Jelindukonskaya area also as of oil from Riphean Vingoldinskaya Formation.

Triarenes. Phenanthrene (P) and its alkyl-substituted homologs with 1-4 atoms of C were identified in investigated oils by the mass-spectrometric technique; they are the most usual representatives of tricyclic aromatic HC in oils. The concentration of phenanthrenes is changing from 4 up to 23 % relatively to total content of aromatic compounds (Table 3). In the most part of oils, the concentration of triarenes is within 18-20 %. The minimum concentrations of them are identified in oils from Middle-Botuobinskaya-25 and Yurubchonskaya-8 wells.

Compounds with two methylene groups in the side chains (DMP) are predominating among all homologs of phenanthrene in all investigated oils. Their concentrations are between 26 and 44 relative %. The content of methylphenanthrene (MP) and C3-phenanthrene (TMP) is comparatively high; but the content of unsubstituted phenanthrene and C4-phenanthrene (TeMP) totally is less than 10 %. Comparatively high concentrations of TeMP (more than concentrations of phenanthrene and MP) are characteristic for oils from Kugasskaya area (Lower Cambrian) and Jelindukonskaya area (Vendian). These compounds were not identified in oils from Sobinskaya area and in oils from Riphean strata.

Special parameter R_c (vitrinite reflectivity) was calculated as ratio between the content of some isomers of methylphenanthrenes in a mixture and the content of phenanthrene; its value is the characteristic of oil thermal maturity [9]:

$$Rc=0,6\times1,5\times (2\text{-}MP+3\text{-}MP)/$$
$$/(P+9\text{-}MP+1\text{-}MP)+0,4$$

Calculated for oils from Katangskaya OGBR parameters Rc range within 0,40 – 0,63; it indicates an immaturity of oils in this region. The same conclusion may be done from data of n-AB content; homologs with odd number of carbon in molecules are predominating in the mixtures. One of the Riphean oils from Baikitskaya OGBR is characterized by the signs of the highest degree of thermal maturity; but oils from Nepa-Botuobinskaya OGBR have intermediate values of Rc which is correlated with special features of their n-alkylbenzenes mixtures composition. In cross-section Lower Cambrian-Riphean strata the oil maturity is growing with the age of enclosing deposits. This trend is opposite to the one in Vendian oils from Katangskaya OGBR (Fig. 5A). It should be noted that oil's maturity doesn't depend on

Table 3. Distribution of triarenes in oils

Number of oil *	Phenanthrenes**	Content of phenanthrenes in mixture, %rel.					Rc
		P	MP	DMP	TMP	TeMP	
1	22,9	4,0	12,6	26,5	24,2	18,3	0,8
2	4,5	2,0	15,3	44,4	23,2	0,6	0,8
3	21,6	3,4	15,3	37,6	25,9	3,0	0,8
4	21,6	4,7	20,4	35,6	19,5	3,8	0,9
5	21,8	5,6	19,7	36,1	19,0	3,6	0,7
6	20,7	4,1	20,3	39,9	18,2	1,7	0,8
7	20,9	3,1	8,6	34,8	27,3	12,5	0,6
8	3,9	3,8	11,8	38,2	31,7	0,0	0,4
9	13,9	12,5	28,3	30,0	10,2	0,0	0,8
10	18,7	4,1	19,6	42,5	18,1	0,0	1,0

Note: *Number of oil – see Table 1, ** – total content of appropriate group in a mixture of identified aromatic compounds, P – phenanthrene, MP – metyilphenanthrene, DMP – dimetyilphenanthrene, TMP – trimetyilphenanthrene, TeMP – tetrametyilphenanthrene.

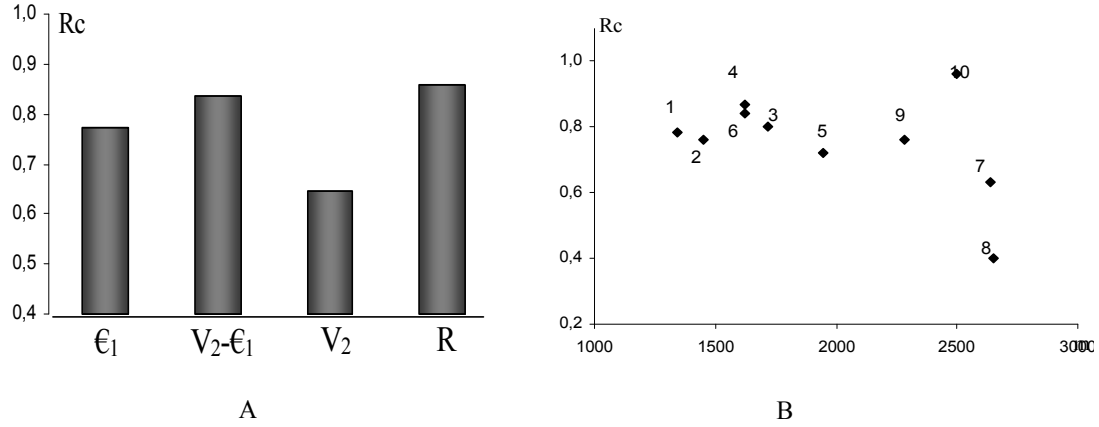


Fig. 5. Maturity of oils along oil-bearing cross-section (A) and in dependence on a field depth (B). Sample number see Table 1

occurrence of depth for the investigated oils (Fig. 5B).

Conclusion

Analysis of the data showed that there is a wide variety of alkyl-substituted benzenes, naphthalenes, fluorenes and phenanthrenes. The highest concentrations of these compounds were found in oils from Nepa-Botuobinskaya OGBR; the oil from Lower Cambrian strata has the maximum concentration. Relative concentrations of special types of arenes varies in different oils; but in the most part of oils alkylbenzenes are the most representative type of arenes. Oils from Riphean Urubchonskaya Formation, Vendian Byukskaya Formation and Vend-Lower Cambrian Uspunskaya Formation are characterized by predominating naphthalene structures. Oil from Sobinskaya area is characterized by low concentration of condensed aromatic compounds; C4-substituted molecules are in a very small concentration or

they are absent at all in these compounds. TeMN are dominating among all the other structures in oils from the Lower Cambrian strata while; DMN and TMN homologs are predominating in other oils.

Alkylbenzenes of oils are presented by homologs of n-alkylbenzene, ortho- meta- and para-methylalkylbenzenes, dimethyl- and trimethylalkylbenzenes, phytanilbenzene and its methyl- and dimethyl-substituted homologs. Methylphytanilbenzene has the highest concentration among all phytanilbenzenes and di- and tri-substituted compounds are predominating among all n-alkylbenzenes.

Methylfluorenes are predominating among all fluorenes in oils from Riphean deposits of Baikitskaya OGBR; but dimethyl-substituted fluorenes are predominating in the recent deposits. C2-substituted phenanthrenes are predominating in all oils.

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Состав моно-, би- и трициклических ароматических углеводородов в нефтях центральной части Восточной Сибири

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Состав ароматических углеводородов нефтей центральной части Восточной Сибири изучен методом хромато-масс-спектрометрии. Анализ результатов показывает присутствие в образцах широкого спектра метилзамещенных углеводородов ряда бензола, нафталина, флуорена и фенантрена. Относительные концентрации этих типов соединений варьируют для различных нефтей, но в большинстве образцов преобладают алкилбензолы.

Ключевые слова: нефть, ароматические углеводороды, моноарены, биарены, триарены, Восточная Сибирь, алкилбензолы, нафталины, фенантрены, хромато-масс-спектрометрия.