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Geochemical characteristics of terrestrial organic matter in the Upper Paleozoic complex of the Vilyui syneclise and some features of its transformation under thermobaric conditions at great depths

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Abstract. A combination of geochemical methods were used to study organic matter from Permian rocks in the central part of the Vilyui syneclise (East Siberia) penetrated by the Srednevilyuiskaya-27 ultra-deep well in the depth range of 3370-6458 m. This study discusses variations in the pyrolysis indices (HI, T_{max}), hydrocarbon type content (hydrocarbons-resins-asphaltenes), vitrinite reflectance (R^0_{vt}, %), organic carbon content (C_{org}), as well as some trends in the saturated and aromatic hydrocarbon compositions of bitumen extracts from the Upper Paleozoic rocks. Below a depth of about 4.5 km (late mesocatagenesis), the hydrocarbon type composition is characterized by a sharp decrease in the content of asphaltenes from < 30 % (at 4.5-5.0 km) to < 15 % (at 5.0-5.5 km), which are not detected at greater depth. In turn, the resins became the dominant constituent (~ 50-70 %), whereas hydrocarbons account for < 20 % at depths down to 5 km and < 40 % at greater depth. These depths are also characterized by a predominance of saturated hydrocarbons over aromatic compounds with a decrease in the relative contents of high molecular weight compounds in both fractions, as indicated by mass chromatograms. The hydrocarbon index (HI) of organic matter decreases to the first tens from the depth of 4.9 km and to the bottomhole (6519 m); the temperature of the maximum hydrocarbon yield (T_{max}) varies between 570-580 °C, showing a slightly increasing trend. Our results show that the generative potential of organic matter from the rocks within the studied depth range (4.9-6.5 km) has been exhausted and that the terrestrial organic matter undergoes significant changes under severe temperature and pressure conditions at great depths.

Keywords: terrestrial organic matter, hydrocarbon type composition, pyrolysis, catagenesis, ultra-deep well, Vilyui syneclise, chromatography-mass spectrometry

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Depleting oil and gas reserves in the upper horizons of sedimentary basins are becoming a pressing issue worldwide. This requires a thorough evaluation of hydrocarbon accumulations at great depths: the lower boundary of their distribution, the peculiarities of their conversion under severe pressure-temperature conditions, changes in their composition, etc. The deepest well in Siberia, Srednevilyuiskaya-27, was drilled in 1984-1986 down to 6519 m and penetrated the sedimentary cover to the top of the Carboniferous to study the geological structure and assess a potential for deeply buried hydrocarbons. This well is located within the Khapchagai megaswell, to which the largest fields of this petroleum area are confined: Srednevilyuiskoe, Tolon-Mastakhskoe, Sobolokh-Nedzhilinskoe (Fig. 1). In this study, we present the results of the analysis of organic matter (OM) in the Upper Paleozoic strata represented by irregular interbedding of sandstone, siltstone, and mudstone. Permian strata grade into relatively deep marine, coal-rich Carboniferous facies deposited in a shallow-water marine environment (Kontorovich et al., 1994; Tectonics, geodynamics..., 2001). The current understanding of the lithologic, stratigraphic and structural setting of the studied part of the sedimentary section of the Vilyui syneclise is based on the data of Grausman et al. (1980). Core samples collected from the 3370-6458 m interval characterize the following strata: kn – Kyundei (3226-3480 m); hr – Kharyya

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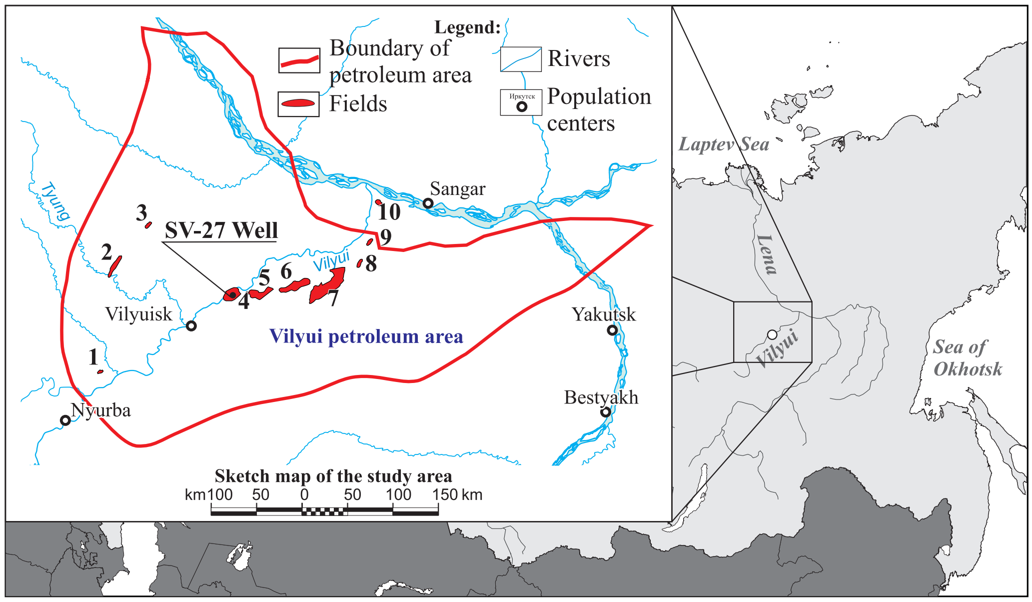

Fig. 1. Location of the Srednevilyuiskaya-27 (SV-27) well in the territory of Vilyui petroleum area. Fields: 1 – Nizhnetyukyanskoe; 2 – Srednetyungskoe; 3 – Andalakhskoe; 4 – Srednevilyuiskoe; 5 – Tolonskoe; 6 – Mastakhskoe; 7 – Sobolokh-Nedzhelinskoe; 8 – Badaranskoe; 9 – Nizhnevilyuiskoe; 10 – Ust-Vilyuiskoe.

(3480-3887 m); hm – Khomustakh (3887-4305 m); kb – Kubalangda (4305-4696 m); hrb – Kharbalakh (4696-5143 m); cc – Chochos (5143-5663 m); jn – Yunkyur (5663-6073 m); jr – Yuren (6073-6519 m).

Since the late 1980s, much of the attention of the researchers has focused on the analysis of dispersed organic matter from Upper Paleozoic sediments of the Vilyui syneclise. Different research teams collected unique data on the variation of predominantly terrestrial organic matter in a relatively homogeneous section during its uniform catagenetic transformation. Kontorovich et al. (1988) presents the data on bitumen extracts, redistribution of hydrocarbon-type composition, and thermodynamic boundary of sharp compositional changes. In addition, these authors proposed a scheme for the destruction of hydrocarbons (HC) during apocatagenesis, including degradation, simplification of their structure, which occur simultaneously with condensation reactions of the structural blocks of asphaltenes (mainly aromatic) and their conversion into an insoluble phase. Melenevsky et al. (1989) examined the variations in pyrolysis parameters and presented the results of electron paramagnetic resonance (EPR) study. Bodunov et al. (1990) discussed changes in the composition of individual hydrocarbons and chromatogram patterns. Subsequent studies revealed the presence of unique compounds in the zone of strong thermal transformation (Kashirtsev et al., 2016, 2017). Polyakova et al. (1999) presented a summary on the hydrocarbon potential of deeply buried horizons by comparing data collected from ultra-deep wells (Tyumenskaya SG-6, Srednevilyuiskaya-27 and Bertha Rogers, USA). The authors analyzed in detail the variations trends in the generative potential with increasing degree of catagenesis.

In this study, we employ a larger number of samples than has been previously used to give a general geochemical characterization of the studied interval, to redefine the boundary of changes in the hydrocarbon composition of hydrocarbons, to trace the transition zone, and to calibrate the obtained parameters with the vitrinite reflectance data.

Methods

In this study, we used the results of the geochemical analysis of organic matter from 71 core samples collected from the Srednevilyuiskaya-27 well, including the level of organic maturity (R^0_{vt}, %), Rock-Eval pyrolysis parameters (HI, T_{max}), hydrocarbon-type composition (hydrocarbons, resins, asphaltenes), and organic carbon content (C_{org}) per rock. Organic matter was extracted from sedimentary rock samples with chloroform and asphaltenes were precipitated with petroleum ether. Maltenes were further separated into fractions by column chromatography. The saturated and aromatic fractions were analyzed by chromatography-mass spectrometry using the Agilent 6890 gas chromatograph with 5973N mass-selective detector. Vitrinite reflectance measurements were performed on a MSFP-2 microscope spectrophotometer. Quantification of organic matter was performed by Rock-Eval pyrolysis in the absence of oxygen, by double analysis of each sample. The total organic carbon content of rocks was determined on an AN-7529 carbon analyzer.

Results and discussion

In modern petroleum geology, catagenesis of organic matter is one of the principal indicators used to assess the hydrocarbon potential of a sedimentary basin, the degree of preservation of the trapped hydrocarbons within the accumulation, and the hydrocarbon generative potential of source rocks, because the relationship between the organic matter transformation and distribution of hydrocarbon accumulations has been established in many regions of the world. Based on the depth zonation of hydrocarbon generation and maturity levels of source rocks proposed by Vassoyevich (1967), Kontorovich (1976) and Neruchev (1973), we defined the boundaries of possible oil and gas generation in Upper Paleozoic sediments of the Vilyui syneclise (Fig. 2). Three zones of hydrocarbon generation were identified in the studied section based on 71 vitrinite reflectance sample analyses. The first zone of predominantly liquid petroleum generation comprises the Kyundei and much of the Kharyya sequences at depths ranging from 3370 (first measurement) to 3800 m. According to the classification of A.E. Kontorovich, this zone corresponds to late MC_2. Despite the fact that liquid hydrocarbons are produced during this catagenesis substage, the intensity of petroleum generation is much lower than that within the oil window, corresponding to substages MC_1^1-MC_1^2. A transition to the wet gas/gas-condensate generation zone is identified down the section. This zone comprises the lower part of the Kharyya, Khomustakh and almost the entire Kubalangda sequences (3815-4700 m) and corresponds according to the above classification to MC_3^1, MC_3^2. Zone III represents early apocatagenesis (AC_1, AC_2) and comprises the basal parts of the Kubalangda, Kharbalakh and Chochos sequences (4700-5500 m). This level of thermal maturity suggests the likelihood of dry gas generation. In the deeply buried Yuren and Yunkyur sequences, the processes of petroleum generation became replaced by thermal cracking of the liquid hydrocarbons and methane formation.

Table 1 shows the distribution of the total organic carbon (C_{org}) in the studied interval. The average C_{org} values of most samples are higher than the average crustal values reported for mudstones (Vassoyevich, 1972) (53 samples >0.9 %) with the maxima lying between 0.9 and 1.9 % (31 samples). The highest C_{org} values are calculated for clay-rich rocks (43 samples), which also indicates their high generative potential. In addition, biomarker analysis also indicates a close link between the oil rims of gas condensate fields within the Vilyui syneclise and organic matter from the Upper Paleozoic rocks (Kashirtsev et al., 2009). The slightly higher values reported for the upper part of the Late Permian section suggest that the residual generative potential (HI) of these rocks is not completely exhausted.

Figures 3 and 4 show Rock-Eval pyrolysis data. A comparison of HI with the catagenesis substages based on R^0_{vt} values reveals that samples with the level of thermal maturity corresponding to MC_2-MC_3^1 have higher residual HI (50-190 mg HC/g C_{org}), which decreases considerably toward the substage MC_3^2. The Kyundei, Kharyya, Khomustakh and much of the Kubalangda sequences forming the Upper Paleozoic section in the Srednevilyuiskaya-27 well to 4.6-4.9 km depth retain their petroleum generative capacity. At greater depths, the HI values of 5-20 mg HC/g C_{org} suggest that the generative potential of these rocks is completely exhausted. The temperature index of the maximum hydrocarbon yield (T_{max}) naturally increases

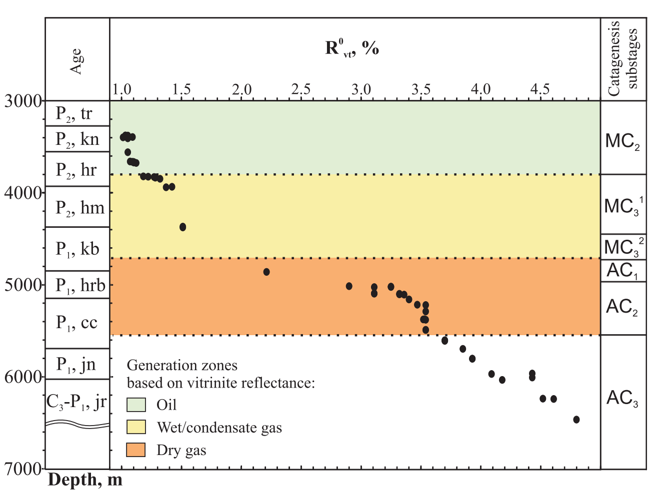

Fig. 2. Changes in vitrinite reflectance values (R^0_{vt}) in the Upper Paleozoic sediments

Distribution of organic carbon content by rocks [**average** (min-max) / number of samples]				
Sequence	Sandstone	Siltstone	Silt-rich mudstone	Mudstone
P_2, kn, Kyundei	-	**2.61** (*3.90-0.84*) / 6	**3.08** / 1	-
P_2, hr, Kharyyas	**0.45** / 1	**1.4** (*2.63-0.58*) / 6	**1.82** (*3.44-0.95*) / 11	-
P_2, hm, Khomustakh	-	-	**1.88** / 1	**1.48** (*1.80-1.15*) / 3
P_1, kb, Kubalangda	-	-	-	**1.33** (*1.44-1.22*) / 2
P_1, hrb, Kharbalakh	**0.16** / 1	-	**1.39** (*1.60-1.06*) / 5	**2.12** (*2.22-1.98*) / 6
P_1, cc, Chochos	-	-	**1.60** (*3.00-0.60*) / 14	-
P_1, jn, Yyunkyur	**0.2** (*0.2-0.1*) / 3	-	**0.70** (*1.40-0.20*) / 5	-
C_3-P_1, jr, Yuren	**0.1** / 1	-	-	**1.80** (*2.50-0.40*) / 3

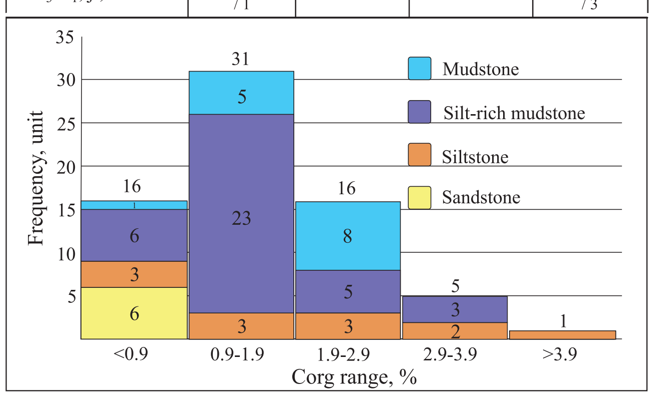

Table 1. Distribution of organic carbon content

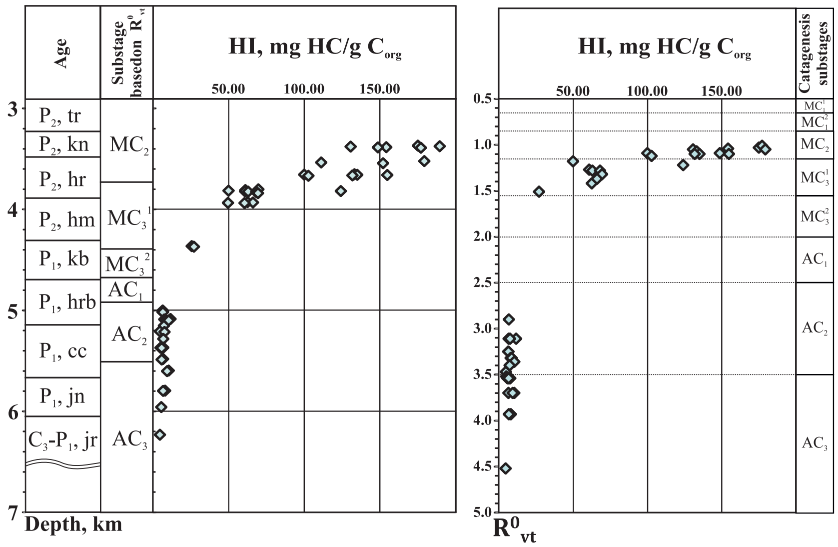

Fig. 3. Variation in residual HI in the well section (left) and with an increase in the vitrinite reflectance index (R^0_{vt}) (right)

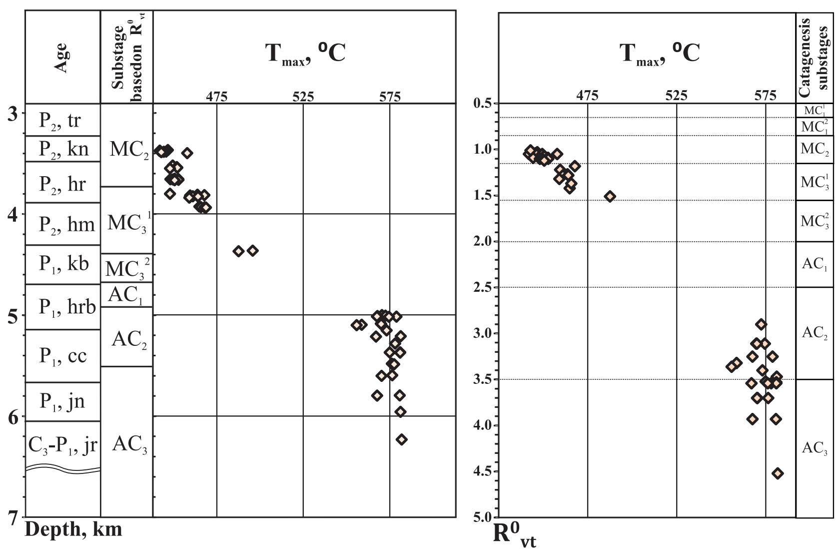

Fig. 4. Variation in T_{max} for the maximum yield of hydrocarbons (S_2 peak) in the well section (left) and with an increase in the vitrinite reflectance index (R^0_{vt}) (right)

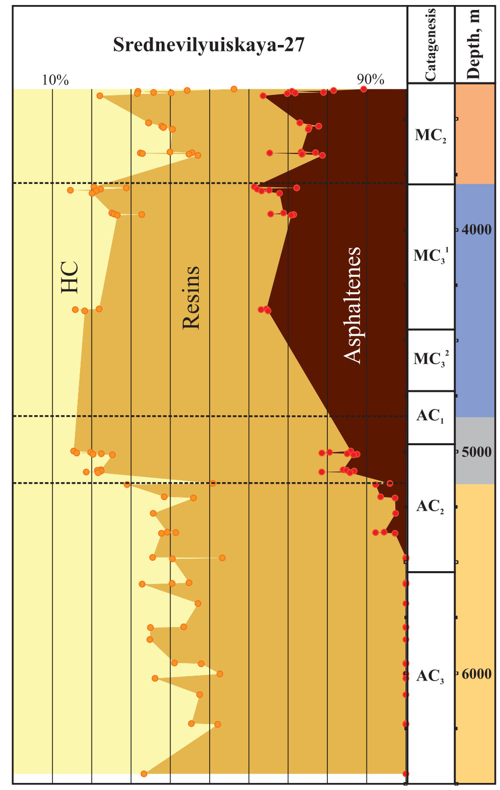

Fig. 5. Depth-dependent variation in the hydrocarbon type composition of bitumens

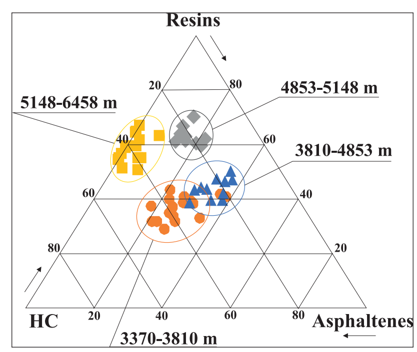

Fig. 6. Triangular plot showing variations in the hydrocarbon type composition of bitumens

with increasing depth and, accordingly, with the degree of catagenetic transformation. Additionally, a comparison of T_{max} and R^0_{vt} gives a correlation coefficient of 0.977. Therefore, the level of thermal maturity of the studied OM type (terrestrial OM from the Upper Paleozoic coal-bearing complex of the Vilyui syneclise) can be reliably evaluated using T_{max}. For example, T_{max} values of 440-460 ^{0}C represent the substage MC_2, T_{max} of 460-490 ^{0}C shows MC_3^1, T_{max} of 490-525 ^{0}C corresponds MC_3^2, and T_{max} >525 ^{0}C represents AC. Based on the Rock-Eval pyrolysis and vitrinite reflectance data, we propose the following depth zonation of liquid petroleum generation in the Upper Paleozoic section: oil window down to 3.6 km (R^0_{vt} – 1.1 %, average HI – 150 mg HC/g C_{org}); base of the gas window at 4.9 km (R^0_{vt} – 2.5 %, average HI – 60 mg HC/g C_{org}).

Figures 5 and 6 show variations in the hydrocarbon type composition of bitumens for depths in the range 3370-6458 m, whereas variations in the composition of individual hydrocarbons are given in Fig. 7.

Close attention was paid to the distribution maxima and the ratio of higher homologues to lower homologues. Using this approach, we estimated the effect of severe thermobaric environment on the destruction of compounds with alkyl chain substituents of various lengths.

Chromatograms of saturated hydrocarbon fractions show n-alkanes and those of the aromatic fractions show n-alkylbenzenes as an example. These compounds in the analyzed samples represent a homologous series that displays the depth variation in the ratio of lower to higher molecular weight parts.

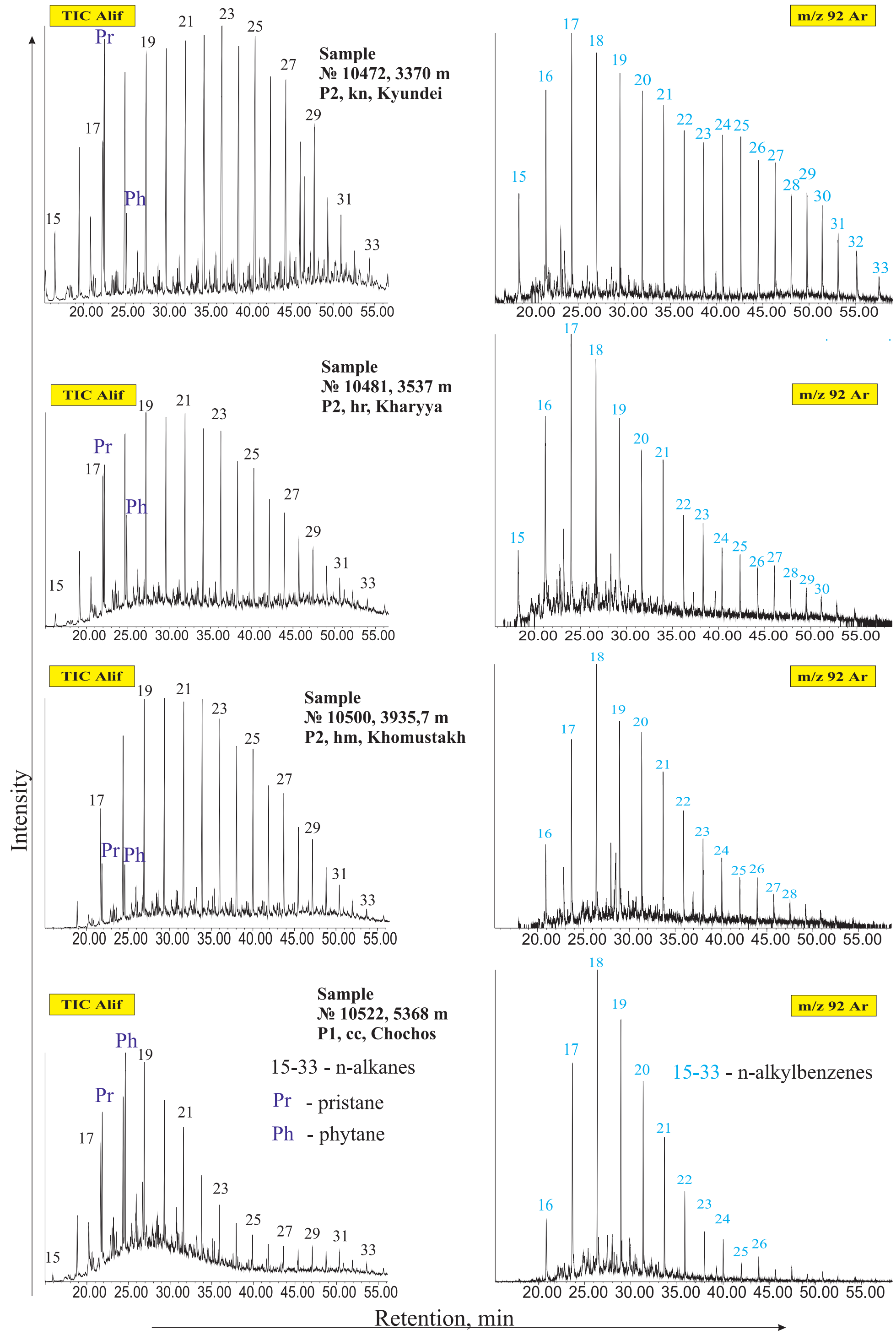

Fig. 7. Total ion current chromatograms (TIC) of normal alkanes and characteristic ions of n-alkylbenzenes (m/z 92)

At depths of 3370-3810 m, representing mid-late MC_2, the composition is dominated by hydrocarbons (35-45 %), while resins and asphaltenes account for <35 % and 25 %, respectively (Fig. 5). This interval is represented by rocks that retained their potential to generate hydrocarbons (they contain allochthonous bitumens, and primary migration is assumed within the Kyundei Formation based on a variation of the hydrocarbon content). For depths, representing the middle part of this substage, the total ion current chromatograms of the saturated fractions show the distribution typical of the continental OM, with peaks at C_{21-25} and a prevalence of pristane (Pr) over phytane (Ph) (Fig. 7). The n-alkylbenzenes have a bimodal distribution with a major peak at C_{16-20} and a small secondary peak at C_{23-26}. At the base of this substage, the main peak of n-alkanes is shifted toward the range C_{19-23}, Pr/Ph decreases, and the n-alkylbenzenes have lower relative content of the compounds with carbon numbers >22. In this depth interval, the processes of hydrocarbon cracking are observed toward the base of the substage, organic matter has high residual generative potential (HI), and processes of petroleum generation take place.

Down the section, at a depth representing late mesocatagenesis-apocatagenesis, we identified two intervals separated by a transition zone, which exhibit a drastically different distribution of these three components (Figs. 5, 6). The first interval, at a depths of 3810-4853 m, corresponds to MC_3^{1-2} – early AC_1. The bitumens display a decrease in the relative content of hydrocarbons (15-25 %) and a slight increase in the relative content of resins (45-50 %). The asphaltene content is 30-35 % and decreases toward late mesocatagenesis. At this depth, the content of saturated hydrocarbons decreases as compared to aromatic hydrocarbons (Fig. 8). This can be caused both by migration and minor petroleum generation, as well as by aromatization of saturated hydrocarbons. The peak in the n-alkane distribution displays a shift toward C_{19} (Fig. 9) and when Pr/Ph reaches 1 phytane tends to dominate over pristane. Such variations in the composition of the saturated fraction have been described earlier (Kashirtsev et al., 2017).

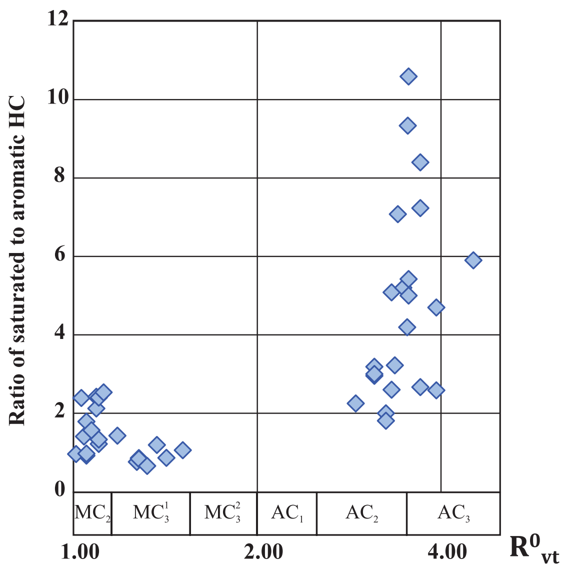

Fig. 8. Variations in the ratio of saturated to aromatic hydrocarbons with increasing catagenesis

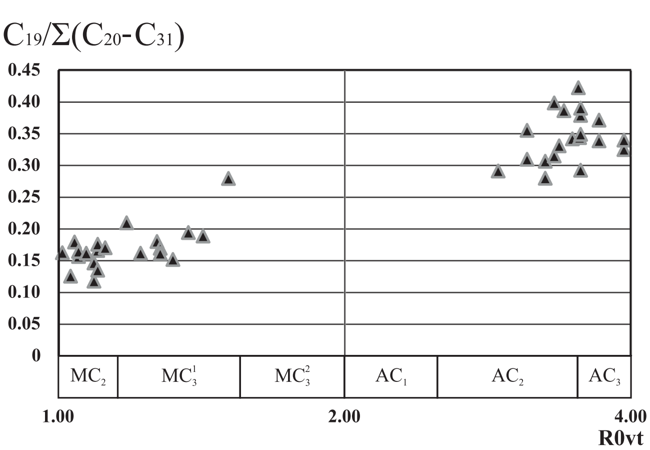

Fig. 9. Variations in the ratio of the relative contents of C_{19} n-alkanes to the sum of C_{20-31} n-alkanes with increasing catagenesis

N-alkylbenzenes have a distribution maximum at C_{18}, whereas the relative contents of compounds with lower carbon numbers decrease, and the peak heights of high molecular weight homologues continue to decrease. In this interval, the processes of cracking or condensation are not observed explicitly at the level of hydrocarbon type composition, but are evident in the distribution of the studied homologous series. The latter suggests that the restructuring of the substance at high temperatures begins before changes in the ratios of the components of the bitumen extracts (HC-resins-asphaltenes) become evident, as will be shown below.

The transition zone corresponds to the beginning of the substage AC_1 (thermobaric boundary at 4853 m). The hydrocarbon type composition is characterized by a dominance of resins (> 60 %), a sharp increase in the content of the saturated compounds (Fig. 8), and a lower relative content of n-alkanes with carbon numbers > 22. This substage is characterized by a sharp increase in resin content due to asphaltene destruction, and, probably, condensation of aromatic structures, as indicated by a decrease in their content of the hydrocarbon component. The homologous series of alkenes, dimethylkanes, alkylcyclohexanes with an odd-to-even-number predominance and four new diastereomers of C_{27} monoaromatic steroids are present only in trace amounts at this thermobaric boundary, but reach concentrations comparable to those of the common biomarkers at greater depths (Kashirtsev et al., 2016, 2017). These authors suggest that these unusual compounds are formed by the destruction of asphaltenes at high temperatures and the removal of occluded compounds.

The deepest interval (5148-6458 m) is represented by rocks with exhausted generative potential. This interval representing middle AC_2 is characterized by a dominance of hydrocarbons (40-45 %) and resins (55-60 %), and a decrease in the content of asphaltenes (<10 %), which are not detected below 5482 m. At the same time, a slight increase in the content of hydrocarbons (~ 5 %) and a decrease in the content of resins is observed with increased maturity level. This zone reflects a further structural simplification, e.g., dealkylation of large structures (mainly asphaltenes), resulting in the increased content of the hydrocarbon component, to form high molecular weight compounds, which then precipitate in an insoluble phase. At the same time, the saturated compounds are the dominant constituents. This can be caused either by enrichment due to the destruction of more complex structures, or by condensation of aromatic compounds, and their further structural incorporation into the resinous component. Kontorovich et al. (1973) noted that the number of paramagnetic centers (NPCs) in terrestrial organic matter increases with increasing degree of catagenesis, and two minima are observed.

In the Srednevilyuiskaya-27 well, one minimum corresponds to late AC_2 (Melenevsky et al., 1989). At this depth the predominance of saturated hydrocarbons over aromatic compounds was reported (Fig. 8). This ratio tends to decrease gradually toward the beginning of AC, which corresponds to an increase in the number of paramagnetic centers. Both saturated and aromatic compounds are present in almost equal concentrations by late mesocatagenesis, whereas the concentration of the aliphatic compounds increases sixfold and more at the beginning of AC_2-AC_3 (Fig. 8). Asphaltenes tend to precipitate completely at the same boundary. It is important to note that based on the data from electron paramagnetic resonance (EPR) spectroscopy (Dindoin, 1973), the aromatic compounds are the most probable paramagnetic centers in organic matter. Also interesting is the increasing number of paramagnetic centers after the above-mentioned minimum may be associated with the consolidation, polymerization, and aromatization of the kerogen structure under severe thermobaric conditions at great depths.

The above transitions in the hydrocarbon type composition of the bitumen follow the scheme proposed by Kontorovich et al. (1988): "...destruction of the liquid products played a prominent role during late mesocatagenesis and was the predominant process during apocatagenesis. The destruction, in turn, occurs in two directions. On the one hand, this can be described as further desintegration and simplification of the structure, following the scheme asphaltenes – resins – hydrocarbons and, on the other hand, condensation of individual blocks, mainly aromatic, and enlargement of the structure, according to the scheme hydrocarbons – resins – asphaltenes, up to the partial transformation of the soluble phase into insoluble one and its precipitation into kerogen." All transitions occur through the resins, because they represent a metastable component under thermobaric conditions. For example, Dobryanskii (1948, 1961) made the following conclusions on the resinous component of crude oils: this component "...is thermally unstable and can easily undergo polymerization, decomposition and overall compositional changes...." In addition, the author described an example when tar oils gave a bituminous distillation residue, while in methane oils the resins coagulated and precipitated as a solid phase or remained in solution in fractions enriched in complex polycyclic hydrocarbons.

Conclusions

A combination of geochemical methods was used to study organic matter in the Upper Paleozoic rocks within the Khapchagai megaswell of the Vilyui syneclise. Based on Rock-Eval pyrolysis and vitrinite reflectance data, we identified several zones of liquid petroleum generation with different phase composition in the sediments in which the generative potential has not been exhausted yet. In this study, we give the correlation of the determination of the level of thermal maturity based on temperatures corresponding to the maximum hydrocarbon yield (T_{max}) and vitrinite reflectance (R^0_{vt}), compare these indicators with those for organic matter from Upper Permian rocks, and provide characteristics of the distribution of organic matter in the rocks within the studied interval.

It was shown that the hydrocarbon type composition of bitumen extracts undergoes significant changes upon maturation (severe thermobaric conditions), which have been described in detail in the previous works (Kontorovich et al., 1988): disintegration and simplification of the structure, taking place together with condensation of the individual blocks and their transition to an insoluble phase. In this study, we traced these changes using a larger number of samples to redefine the depth and maturity levels. In addition, based on the differences in the hydrocarbon type and molecular composition, we identified four intervals in the well section, which differ in the effects of the destruction and condensation processes. We propose possible transition mechanisms between components and describe the mechanisms of noticeable transformations of bitumens at the molecular level during late mesocatagenesis, before sharp changes in the hydrocarbon type composition occur (increase in resin content, asphaltenes discharge). The results of this study can be used to predict the degree of preservation of hydrocarbon accumulations in deeply buried horizons.

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