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Biomarker hydrocarbons of oils from the Labagan field of the Timan-Pechora oil and gas province

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Abstract. The results of geochemical studies of four oils samples from Paleozoic deposits of the Labagan field of the Timan-Pechora oil and gas province are presented. It is shown that the organic matter, which generated the oil of the Lower Devonian and Tournaisian deposits of the Labagan field, is sapropelic, its accumulation occurred in a marine sub-reducing environment. The oils of the Artinskian and Ufimian deposits are substantially biodegraded. Type of collectors is fissure-porous, secondary-porous. Carbonate reservoirs of the Devonian, Carboniferous and Permian (Artinian) age are characterized by good and medium reservoir properties. Terrigenous reservoirs of the Ufimian deposit have good reservoir properties.

Keywords: Timan-Pechora province, Paleozoic deposits, biomarker hydrocarbons, oil, genetic typification, correlation, reservoir

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Introduction

The Labagan oil field is geographically confined to the Sorokin swell of the Varandey-Adz'va structural zone of the north of the Timan-Pechora oil and gas province (TPOGP) (Fig. 1). Currently, the field is developing 15 oil deposits located in reservoirs from the Lower Devonian to the Triassic (Kleshchev, Shein, 2010). The confinement of the deposit to the Varandey-Adz'va fault zone is of interest from the point of view of the genesis of petroleum hydrocarbons (HCs) and the formation of the field itself: what was exactly source of the hydrocarbons? What is their nature and trap filling time? The study of the properties and composition of oils from different oil and gas complexes and their genetic typification are an important stage in the comprehensive geological and geochemical study of the region's oil and gas potential in order to assess prospects and determine further directions for the search and exploration of deposits. The article is a continuation of the study we conducted earlier in this field (Ryabinkina, Valyaeva, 2018).

Short geological sketch

The Labagan structure is composed of Silurian to Quaternary sediments. Stage boundaries were drawn according to Stratigraphic Code (2006). The productive

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horizons of the Labagan deposit are confined to the Lower Devonian, Upper Devonian, Lower Carboniferous, Lower Permian and Lower Triassic deposits (Fig. 2). We studied only the rocks confined to the carbonate deposits of the Ovin-Parmian horizon of the Lower Devonian, the Tournaisian stage of the Lower Carboniferous and the Artinian stage of the Lower Permian, as well as to the terrigenous deposits of the Ufimian stage, which is currently attributed to the Lower Permian (Kotlyar, 2009; Kotlyar et al., 2013).

Lower Devonian sediments were intersected by well 76 in the central part of the Labagan structure at a depth of 3500-4130 m. The oil pool is confined to the anticline trap and is a reservoir, vault, with size of 5.0 km \times 2.5 km, the height of the pool is 74 m. The enclosing deposits are represented by dark-gray strong dolomites, weakly cavernous and fractured with interlayers of greenish-gray, fractured mudstones. The rocks are oil-saturated with spots and vertical cracks. The average reservoir porosity is 8 %, and the extraction coefficient is up to 30 %. The reservoir is sealed by overlying Lower Devonian sulphate-dolomite deposits.

Carboniferous deposits conformably occur on Upper Devonian limestones and are overlain by Permian carbonate deposits with a stratigraphic gap. <u>*Tournaisian*</u> <u>sediments</u> are characterized by a core in all deep wells of the Labagan field. An industrial oil pool was identified and explored in the section. A mudstone member occurs at the base of the Tournaisian strata. The Tournaisian oil pool (C_1 t) is confined to carbonate reservoirs of

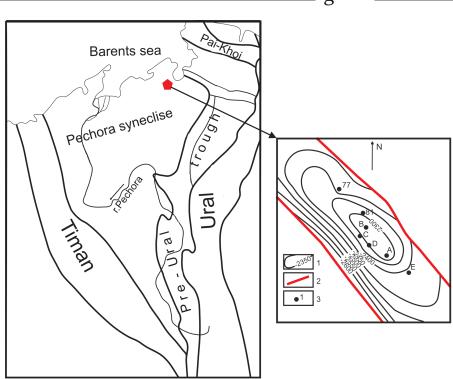


Fig. 1. General scheme of the TPOGP and structural map of the roof of the Tournaisian carbonate deposits. 1 - isogypses of the roof of carbonate deposits; 2 - tectonic disturbances; 3 - wells.

pore-fractured, rarely pore types, it has dimensions of $6.6 \text{ km} \times 3.2 \text{ km}$, the height of the pool is 82 m, the extraction coefficient is 34 %, the average porosity of the reservoirs is 12 %. All carbonate rocks are oil-saturated. In addition, minor layers of mudstone, anhydrite, siliceous rock, and clay siltstone are occasionally found in the section. The thickness of the Tournaisian stage ranges from 84 to 91 m.

The lower part of **Permian deposits** of the *Asselian, Sakmarian, and Artinskian stages* with stratigraphic unconformity occurs on carbonate rocks of the Middle and Upper Carboniferous divisions. The Lower Permian deposits are clearly divided into two strata according to their lithofacies features: the upper one is terrigenous and the lower one is carbonate.

At the base of the Permian productive layer in the sections of wells 73, 75 and 81, the development of a member of organogenic limestones of the *Asselian and Sakmarian age* is noted, which are replaced by fine-grained limestones with a more clay composition, often marl-like. The thickness of the strata is 60-120 m.

The main productive horizon of the *Artinskian age* is composed of permeable carbonate rocks and lies in the roof of the thickness. It is mainly represented by fine-grained limestones and organogenic detritus, porous and fractured, layered, clay-silty, to varying degrees, silicified, with numerous inclusions of fauna. Sometimes in the thickness of limestones there are single strata of marls. The thickness of the productive horizon varies from 26 to 57 m. The Artinskian oil pool (P_1ar) is

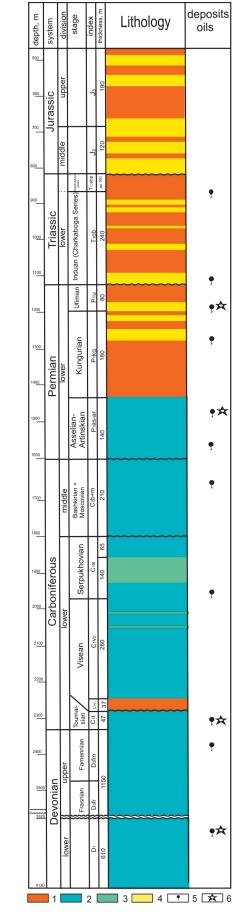


Fig. 2. Summary lithological column and location of oil deposits of the Labagan field. 1 - mudstones, 2 - limestones, dolomites, 3 - anhydrites, 4 - sandstones, 5 - oil deposits, 6 - analyzed oil samples.

confined to carbonate pore and pore-fracture reservoirs. It has a size of 12.0 km \times 3.2 km, the height of the deposit is 97 m. The average porosity of the reservoirs is 22 %. Below the productive layer, small- and finegrained clay and marl-like fractured limestones begin to play a dominant role in the section of the considered thickness, with prints and fragments of fauna, with inclusions of plant detritus and pyrite. Mudstones of the Kungurian age are a seal for the pool. The total thickness of undivided carbonate deposits (Asselian-Sakmarian-Artinskian) within the area varies from 167 to 202 m.

As part of the terrigenous strata of the Lower Permian, the *Kungurian age* is established, which is represented by sandstones, siltstones and mudstones that conformably occur on the Artinskian carbonate deposits. Argillites are clearly dominant in the section of the Kungurian stage. Thickness of the terrigenous strata is from 172 to 193 m.

The Ufimian stage is represented by an alternation of terrigenous rocks of continental, lagoon-marine and lagoon-freshwater facies with single layers of dense light-gray limestone based on the study of core from wells 74, 75, 82 and 141. Wells 75 and 73 received oil inflows from the Ufimian deposits (1235-1247 m in 73; 1112-1140 m in 75). The rocks are represented by fine-medium-grained light-gray polymictic sandstones with clay and carbonate cement. There are layers of coal and black mudstones. Coal and carbonaceous inclusions contain the imprints of plants. There are small layers of limestone light gray, massive, dense. The thickness of the Ufimian deposits is 80-236 m. The Ufimian oil pool (P₁u) is a reservoir, vault, and confined to terrigenous pore reservoirs. It has a size of $12.0 \text{ km} \times 3.2 \text{ km}$, the average porosity of the reservoirs is 28 %, and the extraction coefficient for development using steam-thermal (PTV) methods is 45 %.

Materials and methods

The collection of the studied oils includes samples from 5 wells located in the depth range of 1235-3980 m and in a wide stratigraphic range of deposits from the Lower Devonian Lohkovian to Permian Ufimian (Table 1). The studied oils differ in density, sulfur content, waxes and asphaltenes concentrations. So, in the Lower Devonian and Lower Carboniferous deposits, there are oils belonging to the class of heavy oils, and in the Permian deposits – to the class of bituminous oils. Oil in the P₁ar formation, as well as in the overlying P₁u formation, belongs to low-wax, sulfur, and hightar. Classification of oil is given by (Guidelines for the application of the Classification of reserves..., 2016).

Fractionation of oil. Asphaltenes were separated from a weighted oil sample through precipitation by n-hexane (oil and n-hexane were used in the volume proportion of 1:40). The obtained maltene fraction was separated into apolar (50 ml of 20 % dichloromethane solution in n-hexane) and polar (resins, 50 ml of ethanol-benzene mixture (1:1)) on a column filled with aluminum. The apolar fraction was separated into fractions saturated HCs (eluent – n-hexane) and aromatic HCs (eluent – benzene) on a silica gel column.

The gas chromatography (GC) analysis was made by Kristall-2000M chromatograph ($30 \text{ m} \times 0.25 \text{ mm}$ column HP-5, 0.25 µm thick stationary phase). Temperature was programmed from 110 to 300 °C at a rate of 5 °C/min. The injector and detector temperatures were 300 °C.

Gas chromatography–mass spectrometry (GC-MS) analysis was carried out by Shimadzu QP 2010 Ultra ($30 \text{ m} \times 0.25 \text{ mm}$ column HP-5, 0.10 µm thick stationary phase). Temperature was programmed from 110 to 300 °C at a rate of 5 °C/min. The injector temperature was 300 °C, detector temperature was 250 °C. Mass-chromatograms were reconstructed to m/z 217 for sterane hydrocarbons and m/z 191 for terpane hydrocarbons.

Results and Discussion Geochemical characteristics of oils *Acyclic hydrocarbons*

On chromatograms obtained by the GC method, the distribution of n-alkanes in the oil deposits, studied by us from the Lower Devonian and Lower Carboniferous deposits, is generally quite similar (Fig. 3a, b).

In the saturated fraction, n-alkanes of the C_{11} - C_{34} composition were identified, which were characterized by a single-mode distribution dominated by n- C_{11} -n- C_{18}

	Well	А	В	С	D	Е
Parameters	Age	$D_1 l$	$C_1 t$	$C_1 t$	P ₁ ar	P ₁ u
	Depth, m	3936-3980	2338-2369	2326-2350	1375-1390	1235-1247
	Density, g/cm ³	0.876	0.878	0.876	0.936	0.963
	Sulfur, %	0.42	0.66	0.63	2.25	2.7
	Waxes, %	1.09	4.80	5.01	1.26	0.6
	Resins, %	8.82	9.53	13.63	15.21	19.14
	Asphaltenes, %	1.45	2.77	6.77	6.01	3.83

Table 1. Physico-chemical properties of the Labagan field oils

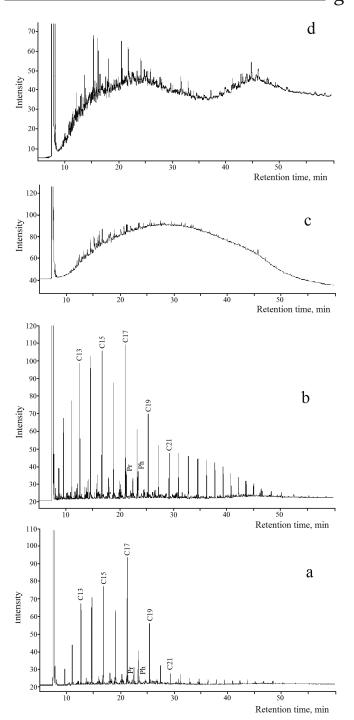


Fig. 3. Chromatogram of the distribution of normal and isoprenoid alkanes in the oils of the Labagan field: a) well A, b) well B, c) well D, d) well E. C (number) – n-alkanes, Pr – pristane, Ph – phytane

(Table 2). Thus, the relative concentration of low molecular weight alkanes of the composition $n-C_{11}$ - $n-C_{18}$ varies from 54.36 to 71.38 %. Then there is a marked decrease in the content of high-molecular n-alkanes: $n-C_{25}$ - C_{34} accounts for 5.39 to 15.45 %. This distribution of alkane HCs indicates that the accumulation of the initial organic matter (OM) of the studied samples occurred under marine conditions (Tissot, Welte, 1981; Ilyinskaya, 1985). Oils are characterized by the prevalence of $n-C_{17}$ over neighboring homologs; the odd

ratio of $2 \times C_{17}/(C_{16}+C_{18})$ varies from 1.71 to 2.51, which may indicate the contribution of algal OM to the initial biomass (Tissot, Welte, 1981; Hunt, 1982). The value of the odd ratio of high-molecular n-alkanes $2 \times C_{29}/C_{28}+C_{30}$ for sapropelic OM, the formation of which took place under reducing conditions, rarely exceeds 1 (Peters, Walters, Moldowan, 2005). The CPI (Carbon Preference Index) is in the range of 1.13-1.30, characterizing oil as mature (Peters, Walters, Moldowan, 2005).

The content of iso-alkanes is very low (iso/nalkanes – 0.08-0.11). The Pr/Ph ratio is close to 1, which may indicate that accumulation of the initial OM occurred most likely in a reducing environment (Peters et al., 2005). The indicators Pr/n-C₁₇ and Ph/C₁₈ are characterized by small values: 0.07-0.21 and 0.3-0.5, respectively. The ratio (Pr + Ph)/(C₁₇ + C₁₈) is low (0.120-0.30).

According to the authors (Reed et al., 1986; Jacobson et al., 1988), the prevalence among the alkanes of the normal structure of the odd homologues of the composition C_{15} - C_{19} (sometimes C_{21}) in combination with low concentrations of higher molecular n-alkanes and low concentrations of isoprenoids is widespread in OM of the Ordovician source rocks and the oils produced by them.

The dominance of n-alkanes of C_{17} and C_{19} together with low concentrations of isoprenoid HCs, recorded in the Lower Paleozoic TPOGP oils, allowed us (Bushnev et al., 2018) to suggest the possibility of generating these oils by deposits containing *G. Prisca* microalgae typical of Ordovician deposits.

For the oils of the Timan-Pechora basin T.A. Kiryukhina (Kiryukhina, 1995) gives a classification based on the distribution of n-alkanes and isoprenoids; according to this classification the oils we studied are of type I, that can be confirmation of generative accessory oils of the Labagan field to Ordovician-lower Devonian oil and gas complex (Fig. 4).

According to the formation-genetic typification proposed for the Timan-Pechora sedimentary basin oils by the authors (Prischepa et al., 2011), the oil from the wells A-C can be attributed to the family B (oil and gas source rock horizon S_2 -D₁), a detailed description of which is given by O.M. Prischepa and co-authors.

There are no n-alkanes and isoprenoids on the oil chromatograms from Permian deposits (Fig. 3c, d), indicating biological oxidation of oils involving microorganisms (Bailey et al., 1973; Reed, 1977; Jobson et al., 1979; Connan, 1984; Petrov, 1984; Kashirtsev et al., 2001; etc.). Intensive biodegradation of oils has changed their hydrocarbon composition and makes it difficult to carry out their genetic typing. It is not possible to determine the type of initial OM as well as the conditions of its precipitation by GC analysis results for these oils.

Well	А	В	С	D	Е
Age / Geochemical parameters	D_1l	C ₁ t	C ₁ t	P ₁ ar	P ₁ u
$\sum C_{11}$ - C_{18}	71.38	61.32	54.56	-	-
$\sum C_{19}$ - C_{24}	15.97	19.24	21.45	-	-
$\sum C_{25}-C_{34}$	5.39	9.82	15.45	-	-
iso-/n-alkanes	0.08	0.11	0.09	-	-
Pr/Ph	1.01	0.98	0.94	-	-
(Pr+Ph)/(C ₁₇ +C ₁₈)	0.12	0.29	0.30	-	-
Pr/C ₁₇	0.07	0.21	0.21	-	-
Ph/C ₁₈	0.30	0.49	0.50	-	-
$2*C_{17}/(C_{16}+C_{18})$	2.51	1.73	1.71	-	-
$2 C_{29}/(C_{28}+C_{30})$	0.88	0.86	0.89	-	-
CPI	1.13	1.30	1.26	-	-
$C_{27}: C_{28}: C_{29}$	35:26:39	31:28:41	31:29:40	31:21:48	32:22:46
Dia/reg	1.23	0.59	0.61	0.39	0.42
Steranes/hopanes	0.10	0.15	0.15	0.18	0.26
Tri/penta	0.07	0.07	0.07	0.26	0.39
$C_{35}/(\sum C_{31-35})$	0.15	0.14	0.12	0.15	0.17
H ₂₉ /H ₃₀	0.52	0.59	0.71	1.30	1.31
*K1	0.41	0.50	0.49	0.46	0.50
**K2	0.48	0.50	0.51	0.56	0.55
βα, % C ₃₀	8.50	8.40	9.41	6.65	8.08
22S/22S+22R	0.60	0.60	0.61	0.54	0.53
Ts/Tm	1.15	1.37	1.42	0.48	0.47

Table 2. Geochemical characteristics of the saturated fraction of the Labagan field oils. $*K_1 = 20S/20S+20R$ (C29 $5\alpha(H), 14\alpha(H), 17\alpha(H)$ steranes); $**K_1 = abb/abb+aaa$ (C29 $5\alpha(H), 14\beta(H), 17\beta(H)-and 5\alpha(H), 14\alpha(H), 17\alpha(H)-steranes)$.

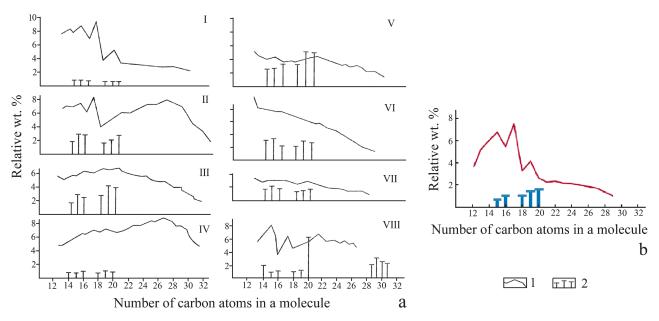


Fig. 4. Molecular weight distribution of n-alkanes (1) and isoprenanes (2) in high boiling fractions of oils: a) of different geochemical types (I-VIII), by (Kiryuchina, 1995); b) in the oil of Labagan field (average values for wells A-C)

Polycyclic hydrocarbons

In oils, the composition of polycyclic HCsbiomarkers has been studied, which contains important information on the composition of the initial organic matter of oils, conditions of its accumulation and thermal transformation (Peters et al., 2005). Their distribution and the resulting coefficients are shown in Table 2.

gr ⁄⁄⁄

The distribution of steranes is shown in m/z 217 mass chromatograms (Fig. 5). Cholestane (C_{27}) varies from 31 to 35%. There is a slight prevalence of ethylcholestane (C_{29}) over neighboring homologs, its oil content varies from 39 to 48%. Triangular diagram shows $\alpha\beta\beta$ steranes of the C_{27} - C_{29} distribution (Fig. 6). Figure 6 clearly shows that the oils from Lower Devonian Lokhkovian deposits and Lower Carboniferous Tournaisian deposits have a slightly different composition of the initial OM than Permian oils, but are characterized by similar sedimentation conditions of the initial OM, which occurred in coastal-marine environments.

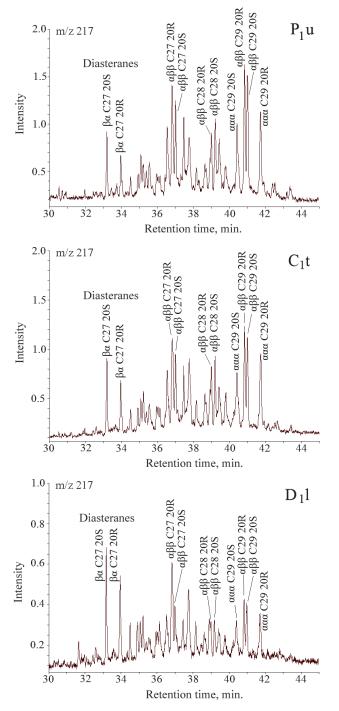


Fig. 5. Mass chromatograms of steranes (m/z 217) of methanenaphthene fractions of oils from deposits of different ages

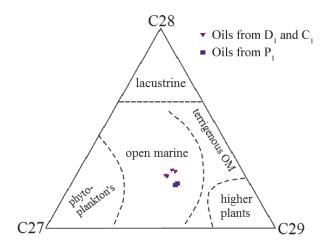


Fig. 6. Diagram of the relative distribution of C_{27} - C_{29} isosterans in oils

One indicator of the facial conditions of sediment accumulation (Brassell et al., 1984; Petrov, 1991) is the ratio of diasteranes to regular steranes (dia/reg). The increased values of this coefficient (0.59-1.23) indicate the prevalence of the clay component in source rocks (which is consistent with the data on the lithological composition of the rocks).

To assess the contribution of algal and bacterial organics, the authors (Peters et al., 2005) suggested using the ratio of steranes/hopanes. The ratio steranes/hopanes varies from 0.10 to 0.26, which indicates an insignificant or moderate bacterial processing of the initial organic matter in an early diagenesis.

The distribution of terpanes is shown in m/z 191 mass chromatograms (Fig. 7). Tricyclic HCs, hopanes, moretane are clearly identified on mass chromatograms. The D_1l and C_1t oils are low in tricyclic hydrocarbons (tri/penta ratio is 0.07), and in biodegradated oils this value rises to 0.39.

Hopanes are represented by compounds from H_{27} to H_{35} . The distribution of $\alpha\beta$ hopanes of C_{31} - C_{35} composition – the homohopane index $(C_{35}/C_{31}+C_{35})$ – is characterized by low values, which indicates the existence of sub-reducing conditions in sedimentation of the initial OM in early diagenesis.

The ratio of adiantane (C_{29}) to hopane C_{30} for oils, the genotype of which we have defined as the Ordovician-Lower Devonian, varies from 0.52 to 0.71, and for oils from Permian deposits – more than 1. In 1994, Al.A. Petrov (Petrov, 1994) found that for TPOGP oils the prevalence of adiantane over hopane is most often characteristic only of the Upper Permian. The C_{29}/C_{30} ratio <1 is characteristic of the OM of the Cambrian and Phanerozoic of Siberia and other regions of the world, as well as in a number of others (Höld et al., 1999; Filndani et al., 2005; Yandarbiev et al., 2017), data are presented for oils and OM when the hopane ratio C_{29}/C_{30} exceeds unity.

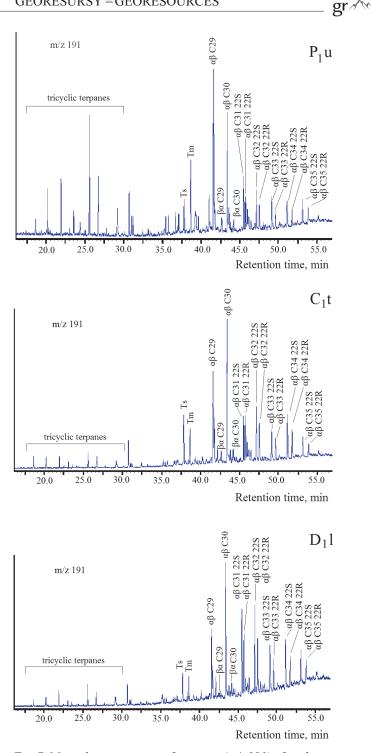


Fig. 7. Mass chromatograms of terpanes (m/z 191) of methanenaphthene fractions of oils from deposits of different ages

T.M. Parfenova believes that "biochemistry of microorganisms that occupied some ecological niche in Cambrian seas was presumably inherited from organisms of Precambrian biospheres. ... The biochemical composition of prokaryotes of some Proterozoic, Phanerozoic, and Quaternary communities was characterized by the elevated adiantane-hopane coefficient" (Parfenova, 2018). According to the authors (Clark, Philp, 1989), the ratio $C_{29}/C_{30} > 1$ indicates the presence of a significant fraction of the carbonate component in the initial source rocks.

The degree of biodegradation (Vinogradova, Punanova, 2012) of Permian oils is defined as medium (moderate). Formation of a series of demethylated 25-norhopanes (reliably identified by the fragment ion m/z 177) characteristic of high-stage biodegradation oils (Volkman et al., 1983) is not observed here.

To determine the degree of the maturity of the OM, the relationship between the initial biological steranes (configuration $\alpha\alpha\alpha 20R$) of C₂₉ composition and isosteranes that have been newly formed as a result of catagenetic processes ($\alpha\beta\beta$ 20R + 20S) (coefficient K_1) is often used; as is the ratio of geosteranes – $5\alpha(H)14\beta(H)17\beta(H)$ – to biosteranes – $5\alpha(H)14\alpha(H)17\alpha(H)$ (coefficient K₂); as well as the relative content of moretane ($\beta \alpha C_{30}$); the ratio of neohopane C_{27} (Ts) to the regular hopane C_{27} (Tm) (0.47-0.48); and the coefficient 22S/22S+22R for homohopane C_{31} .

Ts/Tm ratio differs slightly in the studied oil groups. Thus, Ts/Tm for oils from D₁l and C₁t deposits correspond to values of 1.15-1.42, while for oils from P₁ar and P₁u deposits they do not exceed unity (Table 2). However, based on other coefficients, we can say that the studied oils are equally catagenetically transformed. The maturity of all the studied oils is low, probably corresponding to the beginning of the main phase of oil formation. These differences may well correspond to the formation of A-C and D-E oils in various source rocks. The effect of biodegradation on the composition of biomarkers is not excluded.

Oil-oil correlation

For the oil-oil correlation, we built so-called star diagrams. For correlation of oil M.V. Dakhnova and co-authors propose to use ratios between concentrations of hydrocarbon pairs close in chemical structure (Dakhnova et al., 2007). As such parameters we used the ratios: n-C₁₁/n-C₁₂, n-C₁₃/n-C₁₄, i-C₁₅/i-C₁₆, n-C₁₆/n-C₁₇, $Pr/n-C_{17}$, $Ph/n-C_{18}$ (Fig. 8). The results obtained by the distribution of normal and iso-alkanes show the identity of diagrams, which indicates a common genotype of the Lower Devonian and Lower Carboniferous oils and good vertical fluid communication of reservoir rocks. These conclusions are confirmed by the tectonic structure of the investigated territory.

A comparative analysis of diagrams of oils based on sterane and hopane coefficients (Fig. 9) revealed insignificant differences between the Lower Devonian and Carboniferous oils from Permian oils, which are most likely associated with hypergene changes in the latter.

Conclusions

According to the set of data, obtained by hydrocarbon composition, two oil genotypes are determined at gr M

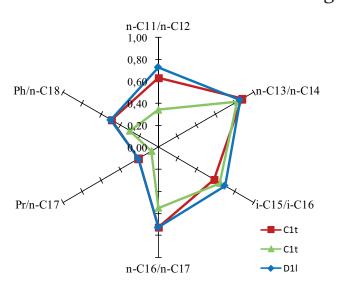


Fig. 8. Correlation of oils by the ratio of the concentrations of pairs of close acyclic hydrocarbons

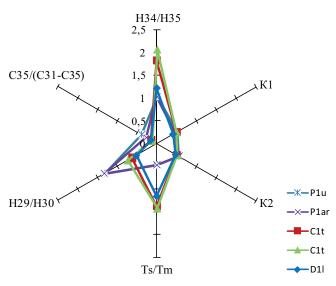


Fig. 9. Star diagram by sterane and hopane coefficients

Labagan field. Oils from the deposits of the Lokhkovian stage of the Lower Devonian and Lower Carboniferous belong to the same genotype – the Ordovician-Lower Devonian. The initial organic matter of oils is sapropelic, the accumulation of which took place in a sub-reducing environment. It is assumed that oil migrated from the Silurian-Lower Devonian sediments to overlying sediments in the decompression zones.

Due to biodegradation, it is not possible to reliably determine the genotype of oils from Permian deposits. This requires additional geochemical studies, such as thermolysis of oil asphaltenes. However, by the distribution of polycyclic hydrocarbon biomarkers we determined that the oil had a slightly different composition of the initial OM.

The maturity of oils of all the oil and gas complexes, studied by us, established by the sterane and hopane coefficients, corresponded to the beginning of the "oil window".

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