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Biodegraded bitumens dispersed in Vendian (Neoproterozoic) rocks of the Khatyspyt Formation, Northeastern Siberia

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Abstract. The organic matter of the Vendian (Neoproterozoic) Khatyspyt Formation was investigated. The new data obtained from the GC and GC-MS analyses include: high UCM humps and heightened peaks of steranes and terpanes against the backdrop of C_{27}+ alkanes on chromatograms of saturated hydrocarbons fractions of five samples; predominance of C_{21}-C_{26} in n-alkanes distribution; the presence of 12- and 13-monomethylalkanes on chromatograms and demethylated terpanes on filtered chromatograms (m/z 191 and 177). Besides, dispersed bitumens with biodegraded hydrocarbons have been for the first time identified in carbonate and carbonate-siliceous rocks of the Khatyspyt Formation. Also 8,14-sekohopanes resistant to biodegradation and gammacerane were established.

The revealed features of the composition and distribution of hydrocarbons showed that their source was the autochthonous (syngenetic) organic matter of the Khatyspyt Formation, one of the potential hydrocarbon source rock of Northeastern Siberia. The composition and content of bitumen are found to be controlled by the inputs of primary organisms (eukaryotes and prokaryotes) which lived in Vendian seas, by changing redox conditions of sediment deposition and transformation, as well as by relatively little effect of temperature and high degree of biological oxidation in hypergenesis.

The patterns of bitumen distribution throughout the section show, that besides the overlying deposits, their accumulations can be discovered in the Khatyspyt Formation.

Keywords: organic geochemistry, potential source rock, dispersed bitumens, saturated hydrocarbons (biomarkers), biodegradation, Khatyspyt Formation, Vendian (Neoproterozoic), Siberian Platform

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Introduction

Extensive geological and exploration works within Olenek Uplift which were conducted in the 30s of the 20th century revealed bitumen occurrences in rock cavity and pore space in Turkut Formation, Kessysa Formation and Upper Paleozoic deposits (Gusev, 1950). It has been known since 1950s that Khatyspyt Formation of Vendian (Neopreterozoic) age is developed on the Olenek Uplift slopes (Zhuravlev, Sorokov, 1954). Presently, the age of the Khatyspyt Formation (its lower limit) can be estimated at ~553-558 Ma based on paleontological data (Grazhdankin, 2004; Rogov et al., 2012; Rogov et al., 2013; Soldatenko et al., 2019). However, geochemical data also indicate that the age of the Formation may be younger, ~545 Ma (Kaufman, 2019). Thus, the Khatyspyt Formation is of Vendian age and is overlain by the Turkut Formation of Nemakit-Daldyn age. The latter contains skeletal fossils *Cambrotubulus decurvatus* (Karlova, 1987). In turn, the Turkut Formation is overlain by stratiform breccias being the product of tuff breccias decay, whose age is 543.9 Ma (Bowring et al., 1993; Rogov et al., 2015). The Khatyspyt Formation provides a unique object of study – the Ediacaran biota along with its habitat and burial setting (Rogov et al., 2012; Nagovitsin et al., 2015; Cui et al., 2016).

Some of the research conducted in the 1960s showed that the Khatyspyt Formation rocks are enriched with organic matter (OM) (Natapov, 1962). For more than 50 years, they have been viewed as source rock in the northeast of the Siberian Platform (Bazhenova et al., 1981; Kashirtsev, 1988; 2004; Kontorovich et al., 1995, 2000; Parfenova et al., 2010). Even today, the question about the role of the Khatyspyt Formation as possible oil and gas source in the Russian Arctic sector remains actual (Stupakova et al., 2017; Kashirtsev et al., 2018,

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2019). Some researchers point out, that the Khatyspyt limestones and dolostones depleted and poorly enriched with OM include carbonate siliceous rocks and black shales, whose generative potential has been realized and they therefore are interpreted as the main source for Olenek and East Anabar bitumen fields (Kashirtsev, 1988; Parfenova et al., 2010; Kashirtsev et al., 2018, 2019; Melnik et al., 2019).

The present work sets out to study the patterns of bitumen occurrences in rocks within the Khatyspyt section, along with geochemical signatures of hydrocarbons-biomarkers.

Materials and methods

The Khatyspyt rocks were sampled in the Neoproterozoic section outcropping along Khorbusuonka River and its tributaries (Fig. 1), and previously studied by geologists (Rogov et al., 2012; Nagovitsin et al., 2015; Cui et al., 2016). The collection including 43 samples was analyzed at the Laboratory of Petroleum Geochemistry of the Trofimuk Institute of Petroleum Geology and Geophysics of the Siberian Branch of the Russian Academy of Sciences (IPGG SB RAS) with an aim of studying the organic geochemistry as part of the project supported by the Russian Science Foundation (grant 17-17-01241). Rocks were powdered in >0.25 mm fraction. Obtaining an insoluble residue (IR) involved dissolution of small weights of ground sample material in 10% HCl. Total organic carbon (TOC) content was determined using express analyser AN-7529 by burning insoluble residues in oxygen flow. Bitumen was extracted with chloroform using a centrifuge and was subsequently purified from elemental sulfur and fractionated by column chromatography. Bulk bitumen content (b_{chl}), bitumen coefficient ($\beta = 100\%*b_{chl}/(1.33*TOC)$) and fraction composition of bitumens were calculated. Saturated fractions of bitumens were analyzed using gas liquid chromatograph Agilent 5890 series II with quartz capillary column 30 m*0.25 mm with HP-5 phase and carrier gas helium. Terpane and sterane hydrocarbons (HCs) were investigated using a gas chromatography-mass spectrometry instrument Agilent (6890 series gas chromatograph and mass-selective detector 5973N). The identification of individual compounds was carried out using data from the NIST 02 mass spectra library and published spectra. The new results of geochemical studies were presented at scientific conferences (Melnik et al., 2019a, b).

Five samples (Fig. 2) showed elevated the bitumen coefficient values, and molecular signs of biodegradation of saturated HCs in bitumen extracts. It is known, that the biological oxidation of HCs by microorganisms is possible only for natural bitumens concentrated in open pore space (also caverns, fissures) at the contact with water (Peters et al., 2005). This primarily was the reason why we concluded that the microscopic bitumen occurrences had been studied. The presence of bitumen is established in limestones (samples K604-1.4 and K602-42.9), siliceous limestones (samples K601C-(-2.5) and K602-53.3), calcareous silicite (sample K602-38.2) (Fig. 2). Additional study of thin sections using petrographic microscopic Carl Zeiss AxioScope A1 showed the presence of microcracks and caverns filled with redistributed organic matter (Fig. 3). Further in this paper, we will discuss only established characteristics of rocks containing dispersed biodegraded bitumens and of their organic compounds.

Fig. 1. Sample collection area

Results and discussion

Occurrences of free bitumens are reported not only in lowermost and uppermost horizons of the Khatyspyt Formation, but also in the middle part of the section (Fig. 2). The insoluble residue constitutes 4-57% (19% on average) in rocks, TOC content is 0.07-0.87% (Table 1). Bitumen content varies from 0.009 to 0.125%. Calcareous silicite is the most enriched with OM. It is known, that average TOC is 0.3-2.0% within the Khatyspyt section, rising occasionally up to 4-6% and 12-14% in limestones and black shales, respectively (Natapov, 1962; Kashirtsev, 2004; Parfenova et al., 2010; Cui et al., 2016; Melnik et al., 2019). The values of the bitumen coefficient vary from 8.5 to 11.3%, while β values for syngenetic bitumens of the Khatyspyt Formation are generally lower (Kashirtsev, 1988; Parfenova et al., 2010; Melnik et al., 2019).

Bitumens differ by the bitumen group composition (Table 1). Usually, resins dominate. Saturated HCs constitute 22%, aromatic compounds 13% and their sum 35%, accordingly; the sum of asphaltenes and

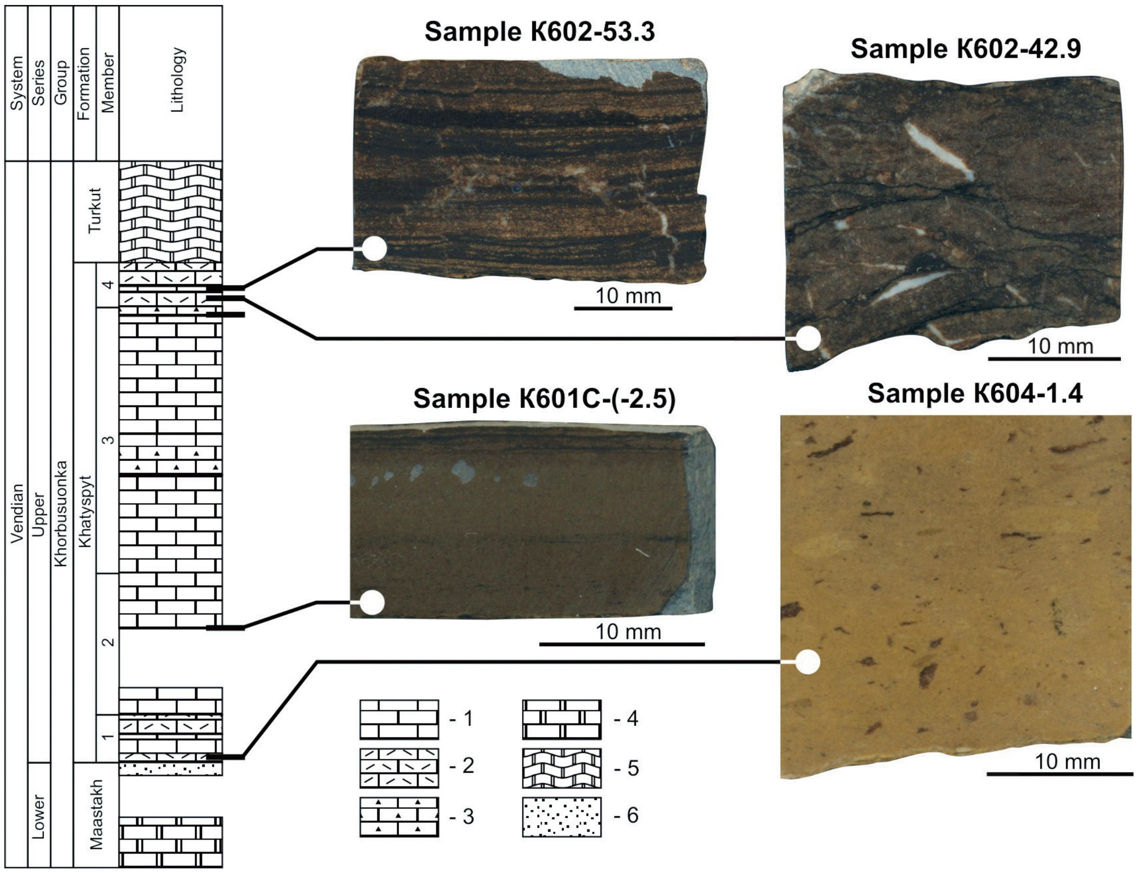

Fig. 2. Stratigraphy and lithology of the Khatyspyt Formation (according to Nagovitsin et al., 2015, modified). 1 – limestone, 2 – intraclastic limestone, 3 – siliceous limestone/calcareous silicite, 4 – dolostone, 5 – microbialitic dolostone, 6 – sandstone.

Sample		K604-1.4	K601C-(-2.5)	K602-38.2	K602-42.9	K602-53.3
IR, %		8	10	57	4	14
TOC, %		0.07	0.18	0.87	0.23	0.33
b_{chl}, %		0.009	0.02	0.125	0.034	0.045
β, %		9.1	8.5	10.8	11.3	10.3
The content in bitumen, %	Saturated HCs	20.3	48.2	15.1	15.2	11.9
	Aromatic HCs	1.4	5.0	16.7	23.9	16.5
	The sum of HCs	21.6	53.2	31.8	39.0	28.4
	Resins	78.4	46.8	59.3	54.5	61.1
	Asphaltenes	undefined	undefined	8.9	6.5	10.5

Table 1. Geochemical characteristic of rocks and dispersed bitumens of the Khatyspyt Formation

resins accounts for more than 60%. Syngenetic bitumens are commonly characterized by a higher content of saturated hydrocarbons against lower concentrations of aromatic hydrocarbons, while resins and asphaltenes remain at approximately the same level (Kashirtsev, 1988; Parfenova et al., 2010; Melnik et al., 2019a, b). The revealed elevated contents of resins and asphaltenes allows us to attribute most of the studied bitumens to asphalts, and one sample (K601C-(2.5)) to "malts" (according to Bazhenova et al., 1981).

Below we consider characteristics of the saturated hydrocarbons from free bitumens.

The normal alkanes distribution is typically characterized by a maximum at n-C_{23-25}. The n-C_{27}/n-C_{17} ratio varies between 0.5-1.6. Such a pattern was noted earlier for some of the Khatyspyt bitumen samples. Usually, n-C_{17-20} predominate among their alkanes (Kontorovich et al., 1995; Kashirtsev, 2004; Parfenova et al., 2010; Melnik et al., 2019). The GL chromatograms exhibit high humps of unresolved complex mixture (UCM) and high steranes and terpanes peaks against the background of alkanes (Fig. 4). The Pr/Ph isoprenoids ratio varies from 0.3 to 1.0 (Table 2). The Pr/n-C_{17} and Ph/n-C_{18} ratios are generally higher, amounting to 0.34-0.37 and 0.39-0.51 respectively. The ratio of the sum of n-alkanes to the sum of isoprenoids, averaging 12.7, is lower, as compared to syngenetic bitumens (Parfenova et al., 2010; Duda et al., 2016; Melnik et al., 2019).

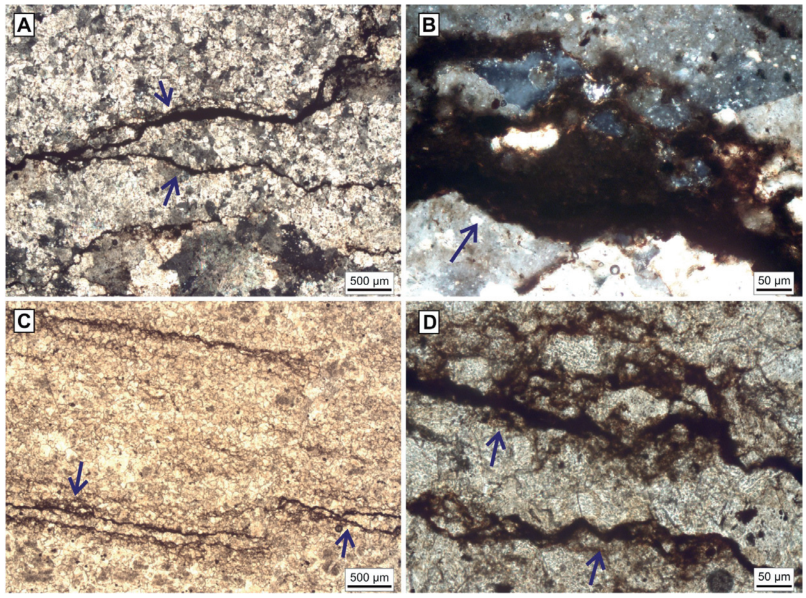

Fig. 3. Typical bitumen occurrence in microfissures in the Khatyspyt rocks. Thin sections, transmitted light microscopy: A, B – sample K602-42.9 (limestone); C, D – sample K602-53.3 (siliceous limestone).

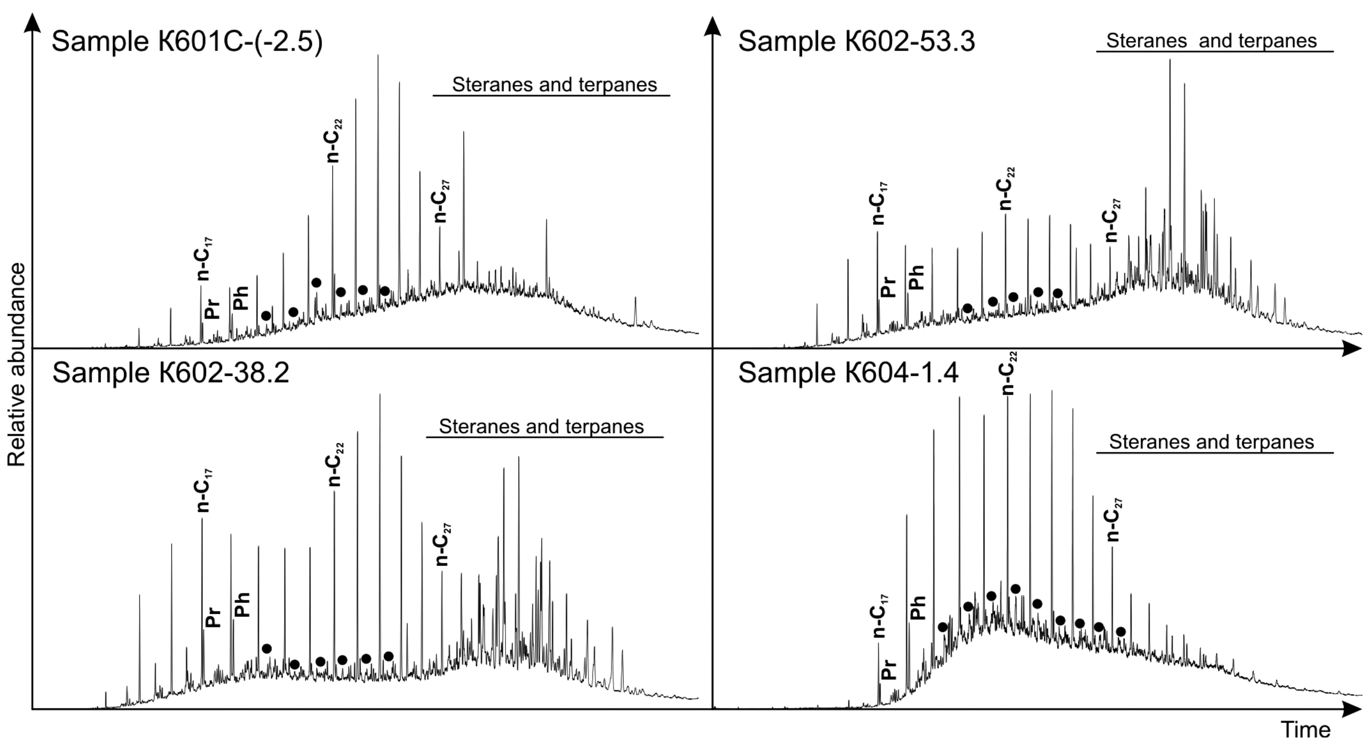

Fig. 4. Chromatograms of the saturated fractions of bitumens from the Khatyspyt Formation rocks. n-C_i – normal alkanes, Pr – pristane, Ph – phytane, black dots – 12- and 13-monomethylalkanes and their low molecular homologues.

The carbon preference index (CPI, Table 2) values for studied bitumens average 1.1, likewise for syngenetic bitumens of the Khatyspyt Formation (Parfenova et al., 2010; Duda et al., 2016; Melnik et al., 2019).

The analyzed chromatograms of saturated fractions of bitumens displayed low concentrations of 12- and 13-monomethylalkanes (Fig. 4). They were first noted in several syngenetic bitumen samples (Melnik et al., 2019). Previously, the possibility for diagnosing these hydrocarbons of the Precambrian OM from the available single samples (Petrov, 1984; Peters et al., 2005) had been dismissed (Kontorovich et al., 1995; Kashirtsev, 2004; Parfenova et al., 2010 etc.).

Analysis of steranes on m/z *217*, *218*, *231* mass-chromatograms permitted to establish two types of steranes distribution both for free bitumens, and syngenetic bitumens. The representative contents include: cholestane between 21 and 35% (per sum of C_{27}-C_{30} steranes), methylcholestanes(16-27%), ethylcholestanes (35-59%), propylcholestanes (1-4%). The C_{29}/C_{27} ratio varies from 1.0-1.3 to 2.4-2.9. The presence of 4-methylstigmastane (according to m/z *217*

	Sample	K604-1.4	K601C-(-2.5)	K602-38.2	K602-42.9	K602-53.3
Alkanes	Pr/Ph	0.31	0.78	0.91	0.83	0.96
	Pr/n-C_{17}	0.36	0.37	0.35	0.35	0.34
	Ph/n-C_{18}	0.41	0.51	0.43	0.39	0.44
	n-C_{27}/n-C_{17}	1.6	1.2	0.6	0.7	0.5
	∑n-C/∑izo-C	9.5	13.8	12.9	16.3	10.8
	CPI*	1.2	1.5	1.0	1.0	1.1
Steranes	C_{29}/C_{27}	1.0	1.3	2.4	2.9	2.6
	20S/(20S+20R)	0.4	0.4	0.4	0.5	0.5
	βα/(αα+ββ)	0.6	0.5	0.2	0.2	0.2
Terpanes	Ts/Tm	0.8	0.9	0.5	0.4	0.4
	Hopanes C_{29}/C_{30}	0.9	0.9	0.8	0.9	0.9
	Hopanes C_{35}/C_{34}	0.8	0.9	1.3	0.9	0.9
	Moretane C_{32}S/R	0.9	0.5	1.4	1.2	1.1
	Ga, %	0.02	0.5	7.2	7.3	6.4

Table 2. Characteristics of hydrocarbons of saturated fractions from bitumens of the Khatyspyt Formation. Note: Asterix (*) denotes Carbon preference index. CPI=0.5((C_{25}+C_{27}+C_{29}+C_{31}+C_{33})/(C_{26}+C_{28}+C_{30}+C_{32}+C_{34})+((C_{25}+C_{27}+C_{29}+C_{31}+C_{33})/(C_{26}+C_{28}+C_{30}+C_{32})) (according to Peters et al., 2005).*

and *231*), acting as dinoflagellate biomarker (Peters et al., 2005), was established. Previously, this HC was identified in bitumen and HyPy-pyrolisate of the Khatyspyt rock sample (Duda et al., 2016). One of the objectives of the Russian Science Foundation project was searches for biomarkers of ancient sponges, however neither 24-isopropylcholestan (Peters et al., 2005) nor 26-methylstigmastan (Zumberge et al., 2018) have thus far been identified. The cholestane isomers ratio C_{29} 20S/(20S+R) remains at the level of 0.4-0.5. The ratio of diasteranes to regular steranes ((βα/(αα+ββ)) varies from 0.15 to 0.55 (Table 2).

Analysis of hopanes and homohopanes, moretanes and gammacerane studied among terpanes, cheilantanes, tetracyclanes, on m/z *191* mass-chromatograms (Fig. 5) revealed the dominance of hopanes and homohopanes (up to 81%). The Ts/Tm and C_{29}/C_{30} hopane ratios are 0.6 and 0.9, on average. The established two types of homohopanes distribution are: $C_{35}>C_{34}$, $C_{32}\geq C_{31}$ and $C_{31}>C_{32}>C_{33}>C_{34}>C_{35}$, the homohopane index C_{35}/C_{34} (Vaz Dos Santos et al., 1998; Kashirtsev, 2004) values vary from 0.8 to 1.3. The cheilantanes are presented in the range from 12-13 to 22-28% in the sum of terpanes, with homologs C_{21} and C_{23} prevailing among them; the tricyclane index ($2*\sum C_{19-20}/\sum C_{23-26}$) values vary within 0.3-1.1 (the average is 0.7). The content of moretanes usually is not more than 5% by the sum of terpanes, with the C_{32}S/R isomers of moretane averaging 1.0. Gammacerane was detected both in high (7.3%) and low (0.02%) concentrations with respect to the sum of terpanes.

The distributions and ratios of HCs established for alkanes, steranes and terpanes of syngenetic bitumens of the Khatyspyt Formation have demonstrated close affinity (Kashirtsev, 2004; Parfenova et al., 2010; Duda et al., 2016; Kashirtsev et al., 2018; Melnik et al., 2019 a, b). This indicates that their source was OM of the Khatyspyt deposits.

A wide range of Pr/Ph ratio and homohopane index values and gammacerane content of HCs in bitumens suggests primarily that their source was sediments formed under changing redox conditions in the Vendian marine basin in the north-east of the Siberian Platform. The distributions of alkanes, isoprenoids, terpanes and steranes of bitumens indicate that archaea, algae and bacteria served as initial inputs of the biomaterial for the organic matter (Petrov et al., 1984; Peters et al., 2005).

Results of this study of bitumens the saturated fractions include pioneering identification of not only demethlated hopanes and homohopanes, but also tetracyclanes and tricyclanes on *m/z 177* mass-chromatograms (Fig. 5). It is commonly noticed, that the latter elute before regular terpanes, since their molecular weight is 14 units lower. Their presence is indicative of a high degree of HCs biodegradation (Petrov, 1984; Kashirtsev, 1988; Peters et al., 2005). The 8,14-sekohopane resistant to biological oxidation of HCs was diagnosed based on molecular mass 414 and fragment-ions 123 and 193 (Peters et al., 2005).

It is known that normal alkanes are the first to undergo bacterial oxidation, then isoprenoids, then hopanes and homohopanes and finally tetracyclanes and tricyclanes (Petrov, 1984; Kashirtsev, 1988; Peters et al., 2005). The fact that both demethylated terpanes and alkanes were found in saturated fractions of the studied samples probably attests to the mixing of bitumens from different stages of generation and filling

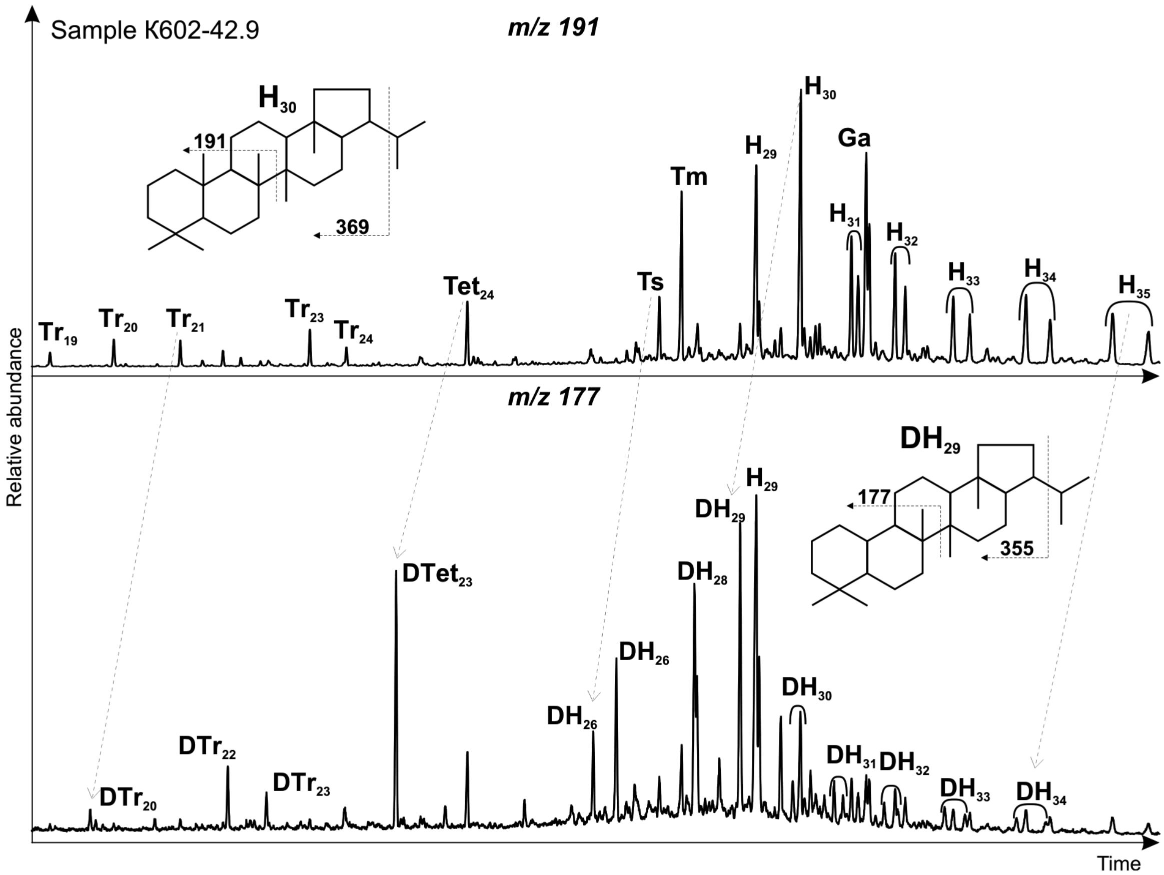

Fig. 5. Mass-chromatograms (m/z 191 and 177) of the saturated fraction of the bitumen from the Khatyspyt Formation rock. Ts – trisnorneohopane, Tm – trisnorhopane, Tr_i – cheilantanes (tricyclic terpanes), Tet_{24} – tetracyclic terpane C_{24}, H_i – hopanes and homohopanes; demethylated terpanes: DTr_i – tricyclic, $DTet_{23}$ – tetracyclic, DH_i – hopanes and homohopanes; Ga – gammacerane.

of the pore space. Bitumens that formed during the first stage were exposed to strong bacterial oxidation, with demethylated terpanes remaining in their saturated fractions. Hydrocarbon generation and migration that followed led to the secondary enrichment of bitumens with alkanes and isoprenoids. The chromatograms (gas-liquid chromatography, Fig. 4) show the ratio of alkanes and terpanes in bitumens to differ significantly, which indicates different intensities of the repeated processes of filling the pores with bitumens and their biodegradation.

Previous studies of the atomic and molecular composition of bitumens from the Turkut Formation of Vendian and Kessysa group of Vendian and Cambrian age within the Olenek Uplift allowed to establish signatures of their HCs composition (high gammacerane content, steranes distribution with ethylcholestanes dominance, absence of 12- and 13-monomethylalkanes). This proves the OM of the Khatyspyt Formation to be their source (Kontorovich et al., 1995, 2000; Kashirtsev, 2004; Parfenova et al., 2010, etc.). Recently updated characteristics of bitumens (oils) generated by Vendian rocks enriched with OM with the geochemistry of aromatic biomarkers (Kashirtsev et al., 2018) have shown that Vendian and Lower Cambrian natural bitumens within the East Anabar and Central Olenek fields owe their origin to the realization of generative potential of the Khatyspyt Formation rocks. Our materials, which supplement the previous results, demonstrate the expanded molecular parameters complex characterizing bitumen genetically associated with autochtonous OM of the Khatyspyt Formation (Melnik et al., 2019a, b). The bottom lines of our findings include: new distribution of steranes with similar concentrations of cholestanes and ethylcholestanes; presence of 12- and 13-monomethylalkanes in low concentrations; low gammacerane content, etc; oil and bitumen accumulations genetically related to the organic matter of the Khatyspyt Formation can be found in sedimentary basins in the Arctic section of Eastern Siberia.

Conclusions

Results of the conducted geochemical study of the organic matter from the Khatyspyt Formation of Vendian age have prompted the following conclusions.

1. The identified occurrences of dispersed bitumen in the Khatyspyt Formation rocks are interpreted as a direct sign of petroleum potential. Most features of the bitumen HCs composition are largely inherited from the OM of the Khatyspyt Formation oil source rock. The maturity

of bitumen and parent OM during mesocatagenesis (early oil window) was estimated from average values of Ts/Tm ratio (0.6), CPI (1.1), and cholestane C_{29}20S/(20S+R) ratio (0.5).

2. The revealed geochemical signatures of the saturated biomarkers indicate that bitumens of the Khatyspyt Formation are biodegraded in hypergenesis. The group composition of bitumens and HCs composition of their saturated fractions resulted from the bitumens mixing which occurred during several stages of their generation, primary migration and biological oxidation.

3. The placement of dispersed bitumens in different parts of the Khatyspyt Formation section within the Olenek Uplift area demonstrates that under favorable geological conditions, oil or bitumen accumulations can be detected in the Khatyspyt section, rather than in the overlying Vendian and Cambrian successions alone, as it was commonly noticed earlier.

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