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Application of gamma-ray spectroscopy and IR-spectroscopy methods for the purposes of ore geology in the Timan-Pechora Oil and Gas Province (the case of Ukhta Region)

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Abstract. Two express methods are presented in this paper. The first method is a high-resolution gamma-spectroscopic method based on a germanium detector, the second method is an IR-spectroscopic method. The applied complex of methods allows to determine the sources of uranium and thorium, identify the rhythms of uranium accumulation associated with regional events; identify areas with a high content of uranium due to the influence of local sources (faults, hydrothermal, etc.); determine the amount of authigenous uranium in the composition of total uranium; determine thermal maturity of organic matter in shales without their preliminary demineralization. To identify levels of increased uranium intensity in the high-carbon strata, a set of indicators has been proposed, which includes both applied indicators in practice of geological work and new indicators.

New indicators have been tested on the collection of shale reference samples. For them, values were established that characterize the processes of uranium accumulation and uranium removal. On the example of Ukhta Region according to the proposed indicators, the sections from the Vendian-Riphean to Domanic inclusive were interpreted.

The performed work showed the possibility of comparing the calculated gamma-spectroscopic data with the data of other methods. This opens up a broader perspective for the use of express non-destructive gamma-spectroscopic method for detecting levels with a high content of uranium in the shale rocks, to which ore-bearing concentrations of a number of metals are also confined.

Keywords: uranium, thorium, domanic suite, shale, kaolinite clays, gamma-spectroscopy, IR-spectroscopy, neutron activation method, method of chromatography-mass-spectrometry data with inductively coupled plasma (ICP MS)

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Introduction

Ukhta Region of Komi Republic in North-Western Russia belongs to western part of the Timan-Pechora oil and gas province and is unique in terms of geological conditions. Geological study and the development of resources in the Ukhta Region was started in the 1930s and is associated with the discovery of deposits of titanium ores (Yaregskoye deposit) and oil fields in the Middle Devonian and Upper Devonian sediments. Tectonically, this region is represents an elongated in the meridional direction Ukhta anticlinal fold, which is located in the Northern part of the East Timan complex shaft (Fig. 1). Devonian sediments are considered promising for further investigation and exploration within Ukhta fold in order to obtain an increase in reserves of titanium ore and hydrocarbons.

In the stratigraphical section, the productive sediments belong to the Djerskaya and Timanskaya suites (Fig. 2). One of the attractive aspects of further investigation of this Devonian sediments is the fact that they occur at relatively small depths (in the central part at 0 – minus 200 m and in the western part of the fold up

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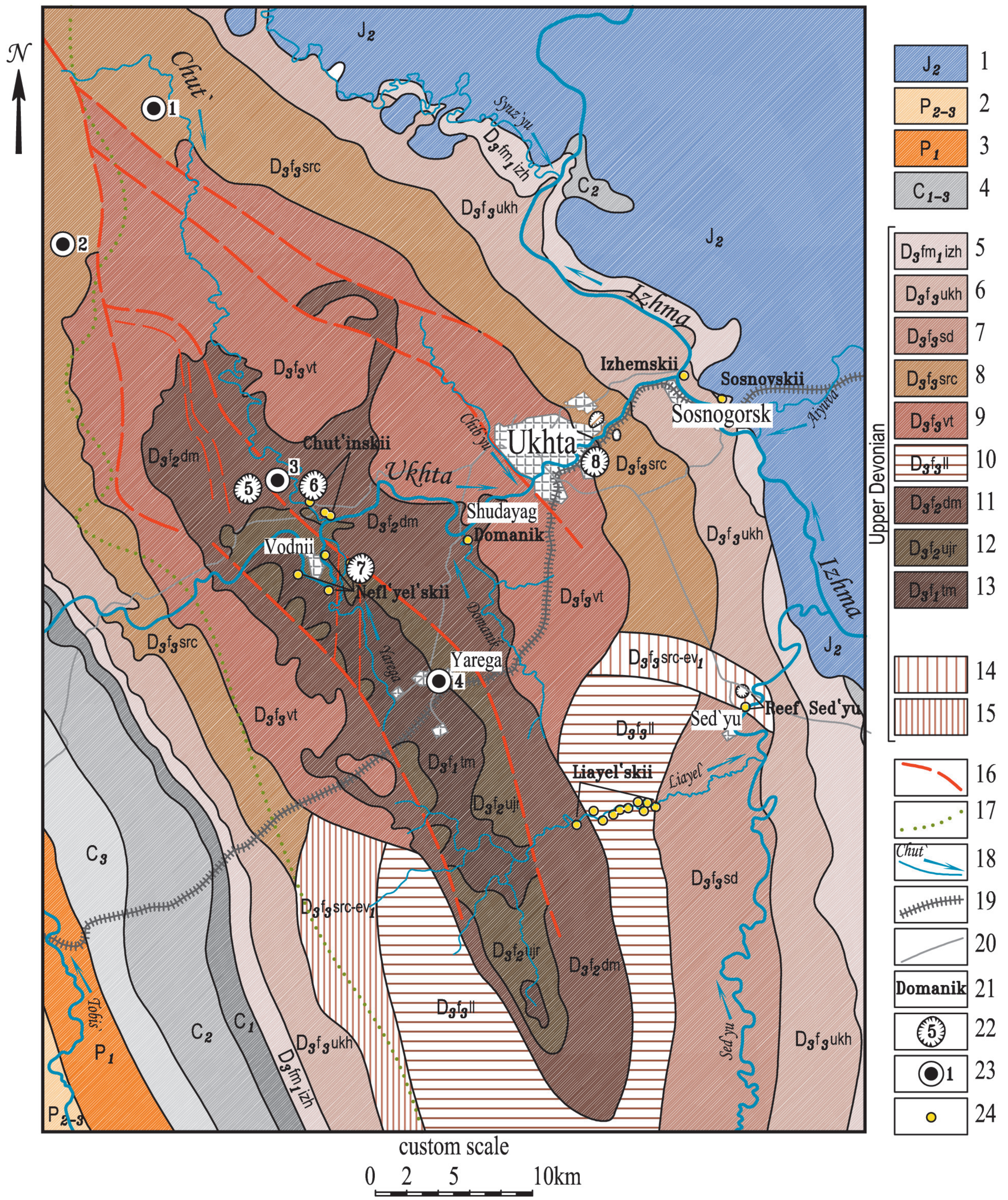

Fig. 1. Location of sampling points on the map of pre-Neogene formations of the Ukhta anticline (according to Yukhtanov et al., 2008). 1–13 – stratigraphic characteristics of sediments:1–4 – sediments in the volume of different systems and departments: 1 – middle jurassic, 2 – middle–upper permian, 3 – lower permian, 4 – carboniferous; 5–13 – upper devonian suites: 5 – izhemskaya, 6 – ukhtinskaya, 7 – sediyskaya, 8 – sirachoyskaya, 9 – vetlasyanskaya, 10 – lyaelskaya, 11 – domanicovaya, 12 – ustyaregskaya, 13 – timanskaya, 14–15 – barrier reefs: 14 – sediysky barrier reef, 15 – vezhavozhsky barrier reef; 16 – faults, 17 – western boundary of domanic depression facies extension, 18 – rivers; 19 – railway; 20 – automobile roads, 21 – geological monuments, 22 – the studied quarries: 5 – quarry "Kerbadiel" (samples Nos. Kk/2m, Kk/4c), 6 – quarry "Lesnik" (sample No. Kl/133); 7 – quarry "Yarega" (samples Nos.Ky/6n, Ky/7n, Ky/8c, Ky/9n, Ky/10n, Ky/ 11n); 8 – quarry "Vetlasyan" (sample No. Kv/7v); 23 – the studied wells: 1 – well T–19 (samples Nos. 19/37, 19/49, 19/61, 19/63); 2 – well K-15(samples Nos. 15/2, 15/6); 3 – well Ch-1004 (sample No. 152); 4 – well 1–CK (oil well-2b sample No. 35a); 24 – natural outcrops of devonian formations.

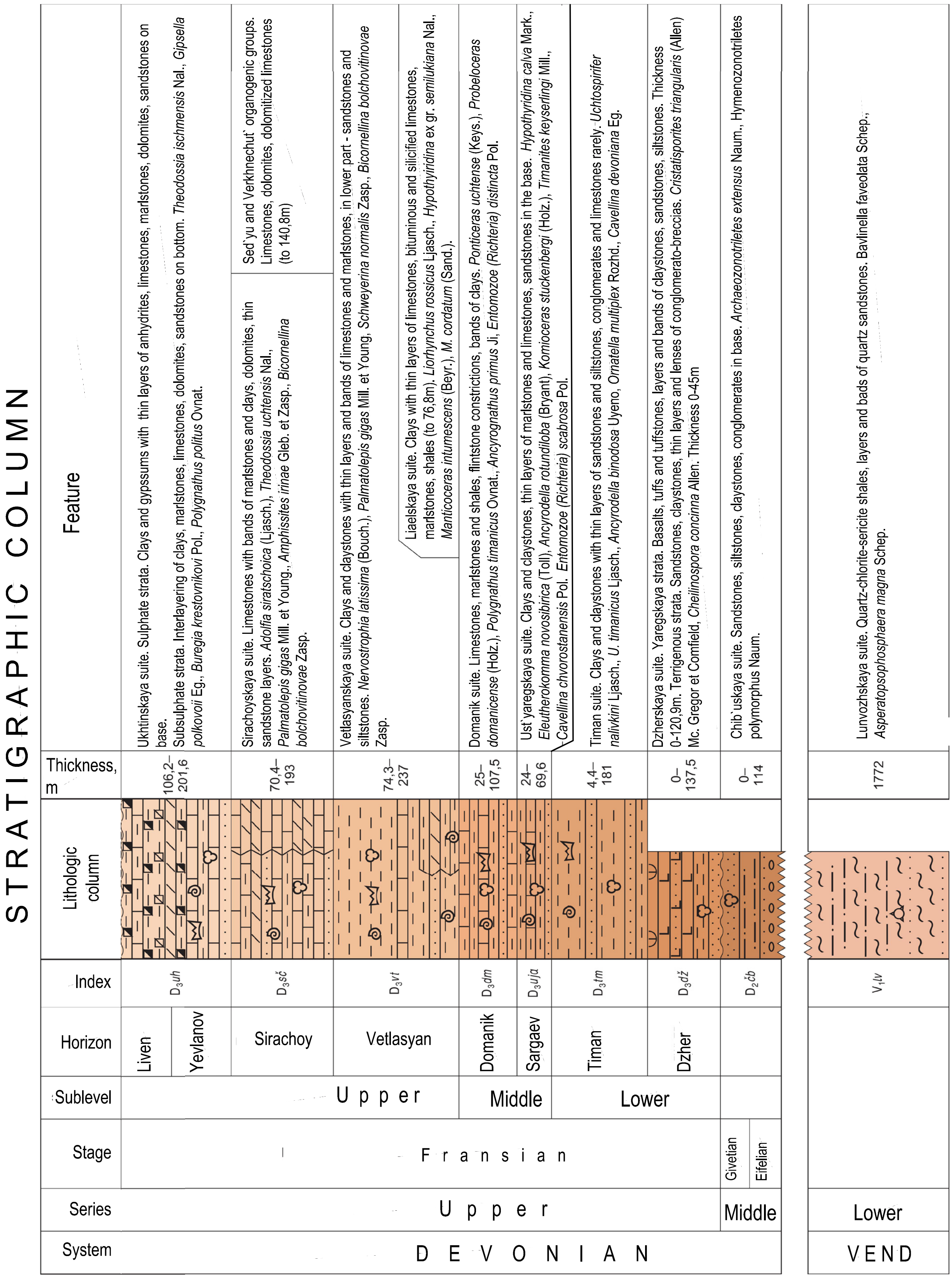

Fig. 2. Stratigraphic column of Vendian – Upper Devonian section within the Ukhta anticline fold (Used cartographic materials of F.L. Yumanov, N.S. Sivash, N.F. Ivanov and others on a scale of 1:200 000, sheet P-39-VI) (Yumanov et al., 2013).

to minus 600–700 m). In a number of places located on the geological outcrops of rivers Domanic, Chut, Ukhta, Devonian sediments, including the domanic suite, which is distinguished as a regional stratotype, come to the earth's surface. This fact contributes to the creation of accessible objects of investigation in the form of natural geological monuments.

Performing complex complementary investigations using the proposed physical methods (gamma-spectroscopic method and IR-spectroscopic method) is an actual task. This is due to the fact that, despite a long geological study of this region, even genesis of titanium ores from the Yaregskoye deposit remains largely discussion. Currently there are three models of deposit formation (Parmuzin et al., 2016).

The purpose of our investigations is to offer, on the basis of express physical methods, a number of complementary indicators for defining processes, which control the formation of ore and hydrocarbon accumulations. In our estimation of the relation of thorium to uranium calculated from the data of activity of these elements using the gamma-spectroscopic method, as well as the estimation of the degree of transformation of organic matter and the estimation of the degree of perfection of the crystal structure of kaolinite minerals within the Ukhta geosynclinal fold, expand the understanding of local processes within the Ukhta fold.

This article presents the characteristics of two new indicators that were initially established using gamma-ray spectroscopy data and tested on results of the neutron activation method and on data of the chromatography-mass-spectrometry method with inductively coupled plasma (ICP MS). Article also highlights the question of estimating changes in the uranium content as a result of its redistribution in shale rock strata. It is known that the processes of the introduction-removal of the uranium can be the result of metamorphism, metasomatosis, influence on geological rocks, as well as the result of redeposition of weathering crusts and acid leaching of rocks by hydrothermal waters (Yurichev, 2015). It is shown that the data of IR-spectroscopy method serve as an additional control of the appearance of certain processes in sedimentary rocks.

Review of literature

Studies of Devonian sediments of the Ukhta anticline fold have been conducted by many researchers both in terms of their ore content and oil content. Generalities of these results has been published in a number of works (Zavyalov, 1966; Zakharov, Kozulin, 1979; Neruchev, Rogozina, 1986; Sivash, Berg, 2010; Berg, Sivash, Bogdanov, 2012; Yentsov, 2013; Yumanov et al., 2013; Parmuzin et al., 2016).

The main features of the geological composition and structure of Devonian sediments include their inconsistent occurrence on metamorphosed shale rocks of the Late Riphean-Early Vendian basement and the complete absence of Lower Devonian sediments, the presence of signs of active volcanism in the marine basin during the Devonian period, which is associated with the formation of tuffites and tuff layers in the sediments of Djersky horizon, as well as the formation of high-carbon-content siliceous rocks, which allocated in a domanic suite (Zavyalov, 1966; Yumanov et al., 2013; Fig. 2).

In accordance with the local working nomenclature for the Ukhta fold, prospective objects include the following: basic sandstones of Middle-Upper Devonian or layer III, which is titanium- productive at the Yaregskoye deposit; fine-grained and small-grained up to grained and gravelitic sandstones of the Upper Devonian – in the upper part of the Djerskaya suite (oil-bearing layers II and II B) and sand rocks in the upper part of the timanskaya suite (layers A and I), this suite differs from the underlying sediments by a more clayey composition (Zakharov, Kozulin, 1979; Yentsov, 2013).

Radiogeochemical methods are part of the search-and-estimation and exploration work for hydrocarbons, ore bodies and diamonds. In the practice of radiogeochemical and geochemical research, calculation indicators are widely used, which are determined based on the relations of values of a particular value. According to the literature data for sedimentary rocks, the following values for the relation of thorium to uranium (*Th/U*) were established: siliceous rocks – 0.7, carbonate rocks – 1.1, clays and clayey shale – 2.2, sandstones – 3.4 (Grigoriev, 2003). In general, the highest *Th/U* value (>7) is found in sedimentary sediments of coastal-marine facies and coarse-grained oxidized rocks, while clayey and carbonate sediments are characterized by low values of the indicator *Th/U* (<1–3). Minimum values of *Th/U* indicator are set in the volcanogenic formations of ocean depressions (<2), and its maximum values are set in the rare-metal granites and some types of alkaline rocks (from 5 to 10 and higher). In metamorphic rocks, *Th/U* values depend on the degree of metamorphism. In geological rocks of eclogite and granulite facies, *Th/U* indicators vary in the range from 1 to 3, in the rocks of epidote-amphibolite facies – from 3 to 5 (Geological dictionary, 1973).

However, there are limitations to the wider application of radiogeochemical methods in the search geology. They are related both to the peculiarities of the geochemistry of uranium (the formation of organometallic compounds, variable valence, etc.), and to the large labor costs in the study of isotopes of radioactive elements.

The established correlation between uranium and organic matter observed in shale and domanic facies (Neruchev, 1982) provides a key to estimating a number of geological processes for changes in the content of these components in the formation.

Results of the study of dependence of the uranium content (Sukhanov et al., 2014) on the content of organic matter in dictionem shale of Leningrad Region of different maturity have led to the following conclusions: uranium-238 is found mainly in the organic component of dictionem shale; the content of thorium-232 is less than 10 % of the content of uranium-238, which is the main source of radioactivity in dictionem shale; an inverse correlation was observed between the content of uranium-238 and the maturity of organic matter in the studied samples of dictionem shale, i.e. the more mature the organic matter, the less it contains uranium-238 and its decay products. Based on the established inverse correlation between the content of uranium-238 and the maturity of organic matter, it is assumed that radioactivity could not be the main factor of more intensive maturation of organic matter in individual layers of dictionem shale. The maturity of individual shale layers at 1.5–2.0 gradation of catagenesis higher (compared to other shale strata) was explained by the influence of hydrothermal solutions with silica dissolved in them. The presence of silica was traced along an intense band with two maxima – at values of 800 and 780 cm^{-1}, which is caused by fluctuations in Si-O-bonds in silica. At the same time, the relation of the intensities of the absorption bands of silica to the intensities of the absorption bands of organic matter was constant in the different samples. This is possible only when silica is embedded in the structure of organic matter of graptolites under hydrothermal influence. Similar data for the spasmodic change in the degree of maturity of organic matter were traced in the section of the domanic horizon within the southern part of Kolvinsky megashaft of Timan-Pechora oil and gas province (Prishchepa et al., 2014).

This paper is a continuation of the study of shale of different ages by two methods: gamma-spectroscopic method and IR-spectroscopic method. It is well known that sediments saturated by range of metals are associated with the beginning of uranium accumulation cycles (Neruchev, 1982). In the domanic sediments of Ukhta Region, positive correlation between the uranium content and vanadium content is traced, as well as positive correlation between the content of thorium and the content of rare earth elements is traced too (Laptev et al., 2017).

To improve the effectiveness of the results of research of geological objects, the authors of this paper in the earlier works (Makarova et al., 2015; Makarova, 2017) proposed a complex of geological-geophysical and geochemical methods. Gamma-spectroscopy method is included in this complex as one of the priority express methods. Given the importance of the issue, we will focus more on the results of an earlier publication by authors of this article. It provides an approbation of the application of *AU/ATh* indicator (the relation of uranium activity to thorium activity) on the example of studying domanic deposits from a deep well (Table 1).

It should be noted some of the benefits of *AU/ATh* indicator. This indicator differs from the thorium-uranium relation (*Th/U*) considered above by a much larger range of changes in values – from 0.4 to 75.0, also this indicator describes the processes associated with the inflow and accumulation of uranium. This allows us to distinguish levels with an increased content of uranium by the distribution of the values of *AU/ATh* indicator across the section. Together with gamma-ray logging data, these levels are the reference points for five cycles of uranium accumulation in the domanic sea basin. They are confidently compared with five stages of increased intensity of uranium accumulation in the domanic stratotypic section (Ukhta Region) (Neruchev, 1982; Makarova et al., 2015).

No.	Depth of occurrence, m	Type of the geological rocks	IRR, %	GRL/AU	*AU/ATh*	$U(10^{-4}\%)/C_o(\%)$	LAU	Cycles of uranium inflow
1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
1	3228.3	marl	20.98	0.25	**6.2**	**2.6**	**5**	**V**
2	3230.1	claystone	72.11	n.d.	n.d.	1.7	**4***	**IV**
3	3232.2	carbonate-siliceous	67.32	4.88	1.5	0.5		**III**
4	3234.1	carbonate-siliceous	69.72	0.49	1.15	1.1		
5	3235.5	silicite	87.9	0.81	2.3	3.2		
6	3237.4	claystone	n.d.	0.34	3.5	n.d.		
7	3238.1	carbonate-siliceous-clayey	58.28	0.45	**6.0**	**1.7**	**3-a**	
8	3239.9	claystone	70.05	0.21	0.6	1.5		
9	3240.7	claystone	75.02	0.19	3.9	1.9	**3**	
10	3242.0	carbonate-clayey	77.74	0.57	**9.2**	**0.7**		
11	3243.9	claystone	73.45	0.51	0.7	0.2		**II**
12	3246.3	marl	42.31	1.80	12.0	0.2		
13	3246.9	carbonate-clayey	53.49	0.29	**75.0**	**0.9**	**2**	
14	3248.4	silicite	75.16	0.40	1.2	0.7		**I**
15	3249.5	silicite	80.21	1.32	1.0	0.4		
16	3250.0	marl	12.79	1.16	0.4	3.4		
17	3250.2	carbonate-clayey	52.26	0.06	**2.0**	**4.3**	**1**	

*Table 1. Results of the study of radioactivity of domanic horizon rocks based on gamma-ray logging data, uranium and thorium activities in the core (Makarova et al., 2015, with additions). * – level is determined by the values of radioactive logging, 3-a – sublevel of the inflow of uranium in the III cycle, n.d. – indicates that there is no data. IRR – insoluble residue of rock, GRL – gamma ray logging.*

Determining the ways in which metals enter sediments and determining the nature of their distribution in high-carbon-content strata is a highly relevant task when searching for deposits of hydrocarbons and ore bodies. This problem is partially solved when determining metals in the fluids of fractured zones (Gorobets et al., 2018). As a result of migration and discharge of fluids along faults and in areas of increased fracturing, uranium, rare and scattered metals are accumulate. In this regard, we note the importance of works of N.S. Sivash with co-authors (Sivash, Berg, 2010; Berg, Sivash, Bogdanov, 2012; Sivash, Makarova, Muravieva, 2016) with convincing geological evidence of migration of deep ore-bearing solutions and hydrocarbons through the same channels. In these works, using a model of geofluidic system (for example, in the Ukhta Region), it is shown that the presence of fluid-conducting channels is one of the determining factors for the formation of oil accumulations and ore occurrences.

Materials and methods

The materials presented by authors contain the results of a comprehensive study of Devonian sediments and domanic deposits from four outcrops and four wells, which are given on the map of pre-Neogene formations of Ukhta fold (Fig.1).

Benchmark samples of shale from the reference collection of authors of this article become the basis for development of new indicators using the gamma-spectroscopic method. Samples were experimentally studied by several methods and from them were obtained complementary gamma-ray spectroscopic, IR-spectroscopic, geochemical data. Materials and samples from old wells drilled in the 60s of the last century were kindly provided by N.S. Sivash for investigation. It should be noted that the most ancient rocks (metamorphosed shale and overlying sand and clay rocks) in the downhole material do not have a clear faunistic characteristic. In this regard, they are attributed to the Upper Riphean – Lower Vendian and to the Middle – Upper Devonian, respectively, by their position in the section. Samples of domanic suite located above in the section are most widely represented. We selected samples from different quarries, such as “Lesnik”, “Yarega”, “Kerbadiel” and “Vetlasyan” together with N. S. Sivash in 2015–2016. Thus, this article compares new data on the single preserved samples from wells and more representative collection of the samples of domanic formation from quarries.

Gamma-ray spectroscopy method. Methodological part of this article presents the characteristics of 7 benchmark samples of shale. Based on the characteristics, the analysis of the thorium-uranium relation values by activity values was carried out and two new indicators proposed:

- indicator of the relation of uranium activity to thorium activity, converted to g/t;
- indicator of uranium-thorium accumulation.

Measurements of rock samples (weight not less than 50 g) are performed on a gamma-spectrometer with a semiconductor HPGe-detector with a sensitive volume of 51 cm^3. The previously tested indicator of the relation of uranium activity to thorium activity (*AU/ATh*) is determined by the intensity of gamma-lines in the decay of daughter nuclides in the chains of thorium and uranium decay.

The proposed methodological approach for applying new indicators consists in converting the values of uranium and thorium activities (Bq) into the values generally accepted for geochemical research – in weight indicators of the content of uranium and thorium (in g/t).

Gamma-spectroscopic data for determining the weight content of uranium, thorium and potassium (in g/t) in relation to the intensities of gamma-lines are shown in Table 2.

Calculated values of the considered indicators for benchmark samples of shale from the different stratigraphic levels, as well as their interpretation, are shown in Table 3.

U/Th indicator. In the sedimentary rocks saturated by organic matter, the average value of uranium content relates to the average value of thorium content as one in three (1:3) (Makarova, Grokhotov, 2017).

This relationship is true for certain “equilibrium” conditions. This relationship is violated when the significant amount of syngenetic organic matter and the associated authigenous uranium (U_a) is accumulated in the sediment. The accumulation of authigenous uranium is controlled by the values of indicator *U/Th* greater than 0.3 (from 0.4 to 14.6).

Values of *U/Th* indicator close to 0,3 reflect the balance between content of uranium and content of thorium entering the sedimentation zone.

Values of *U/Th* indicator less than 0.3 indicate the removal of uranium. Such values are not established among the benchmark samples of shale rocks, saturated by organic matter, but characterize rocks underlying the domanic sediments.

Nuclide	E_γ, keV	$T_{1/2}$, 10^{17}, sec	P, %	ε, %
^{238}U	63.29	1.4	4.8	1.0
^{232}Th	911.21	4.4	2.58	0.5
^{40}K	1460.82	0.94	10.66	0.4

Table 2. Gamma-spectroscopic data for determining weight content of uranium, thorium and potassium (g/t). This table included: E_γ – energy of gamma-lines (in keV), $T_{1/2}$ – half-life (in ten to seventeenth power, seconds?), P – a number of gamma quanta per disintegration (in percentage), ε – absolute efficiency (in percentage).

No. of sample	U, g/t	Th, g/t	*Th/U*	The interpretation of *Th/U* values	*U/Th*	The interpretation of *U/Th* values	PN_{U-Th}	The interpretation of PN_{U-Th} values
1	2	3	4	5	6	7	8	9
3r	**7**	24	**3.4**	The exceeding of values in clarks, sandstones, the increasing of alkalinity, metamorphism	**0.3**	U- and Th- enrichment of rocks, common source of mineralization	**-1.0**	The intensity of uranium accumulation lags behind that of thorium
1r	**4**	11	**2.8**	claystones	**0.4**	U- and Th- enrichment of rocks	**0.4**	The sorption of U_a
55r	**3**	4	**1.3**	Carbonate rocks	**0.7**	U-enrichment of rocks	**1.7**	The enrichment of Ua
7r	**7**	9	**1.3**	Carbonate rocks	**0.8**	U- and Th- enrichment of rocks	**4.0**	The enrichment of U_a
13r	**26**	16	**0.6**	Siliceous rocks	**1.6**	endogenous source of U- and Th- inflow (cracks)	**20.7**	The enrichment of U_a Cc>10
6r	**8**	3	**0.4**	Siliceous rocks	**2.6**	endogenous source of U-inflow (cracks)	**7.0**	The enrichment of U_a
3cr	**44***	3	**<0.1**	Siliceous rocks	**14.6**	endogenous source of U-inflow (fault)	**43.0**	The enrichment of U_a Cc>20

1r,3r,6r,7r,13r – shale rocks of Riphean; 55r – domanic rocks, 3cr – Upper Jurassic, Bazhenovskaya suite; PN_{U-Th} - indicator of the relational uranium-thorium accumulation; U_a – authigenous uranium; Cc – coefficient of uranium concentration relative to clarke in the earth's crust.

Table 3. Content of uranium and thorium in the benchmark samples of high-carbon-content rocks according to gamma-spectroscopic data

Thus, *U/Th* indicator (aka the uranium-thorium relation) is used to determine levels with high uranium content associated with uranium accumulation cycles.

PN_{U-Th} *indicator* (indicator of the relational uranium-thorium accumulation). This indicator is used to estimate the uranium content relative to thorium content in catagenetically and metamorphically transformed shale rocks.

Under natural conditions, total amount of uranium depends on various factors and includes uranium of different origin. Content of total uranium in the geological rock consists of content of mineralized uranium and content of authigenous uranium. The amount of authigenous uranium consists of content of dissolved uranium deposited from sea waters and uranium in the organic remains of dying biota.

Calculation of total uranium content is based on formula (1) (Makarova, Grokhotov, 2017):

$$U_t = U_a + 1/3 \cdot Th, \quad (1)$$

where U_t is total content of uranium, U_a is authigenous content of uranium.

In general, the processes of relational increasing or decreasing of uranium content (relatively to thorium) are estimated by the values of PN_{U-Th} indicator, which is determined by the formula 2:

$$PN_{U-Th} = U_t - 1/3 \cdot Th, \quad (2),$$

where U_t is total content of uranium.

Values of PN_{U-Th} indicator vary in a wide range from negative to positive values:

- positive values of PN_{U-Th} indicator characterize the processes of uranium accumulation relatively to thorium;
- negative values of PN_{U-Th} indicator characterize the processes of decreasing of uranium content relatively to thorium content.

Negative values of PN_{U-Th} indicator illustrate how much the uranium content lags behind the thorium content with an average value of uranium content relates to value of thorium content as one in three (1:3). The reason for relatively low uranium content may be related with different processes. Main processes are:

- the transition of linked uranium to soluble forms and its removal from rocks by hydrothermal solutions;
- the creation of conditions unfavorable for uranium deposition in comparison with thorium;
- processes of thorium accumulation as a result of the destruction of weathering crusts relative to uranium content.

Additionally, to test the proposed indicators, namely *U/Th*, PN_{U-Th}, calculated values of different methods were compared. We used for comparison data of energy-dispersion analysis (SPECTROSCAN MAKC-GV), chromatography-mass-spectrometry data with inductively coupled plasma (ICP MS) and data from the neutron activation method; all this data was obtained by us earlier (Gorobets et al., 2018).

The IR-spectroscopy method. The IR-spectroscopy method is used to clarify the question: what exactly is the process associated with negative values of PN_{U-Th} indicator; also, the IR-spectroscopy method is used to estimate the degree of conversion of organic matter in shale of different ages.

Measurements of rock samples were carried out on a Fourier IR-spectrometer FSM-2202 using a standard method with preparation of a special pressed tablet consisting of rock powder mixed with KBr. In the mixture, content of rocks powder is 1 mg and relates to content of KBr as 1:300.

The interpretation of IR-spectrum graphs was performed with their preliminary normalization on the spectrum of KBr to exclude the influence of impurity components in the air. IR-spectra are measured in the range of wave numbers from 4000 to 400 cm^{-1}.

When interpreting IR-spectra on the graph, two ranges of wave numbers are considered. The first range of wave numbers located in the range of wave numbers from 3100 cm^{-1} to 2800 cm^{-1}. The appearance of absorption bands in these ranges indicates the presence of connections -CH_2 and -CH groups in the composition of organic substances (Tarasevich, 2012).

The definition of the degree of metamorphism and quality of coals is based on measuring the intensity of optical absorption of wave numbers in the region of 4000 cm^{-1}, 3040 cm^{-1}, 2920 cm^{-1}, 2860 cm^{-1}, 2000 cm^{-1} (Ivanov, 2016). We also allow the possibility of using one of the simplest metamorphism indicators for a comparative estimation of the degree of organic matter transformation of samples from domanic suite and from Riphean shale without their preliminary demineralization. From several indicators of coal quality, we selected an indicator that "reflects the relation of number of unsaturated CHnn and aliphatic CH_2 bonds in the molecular structure of shale" (Ivanov, 2016). By analogy with the definition of metamorphism in coals, the degree of transformation of shale is determined by the relation of wave number intensities of two absorption bands 3040 cm^{-1} and 2920 cm^{-1}. This indicator is indicated as $PP_{3040/2920}$ and it estimates in conventional units (units).

The second range of wave numbers located in the range of wave numbers from 3700 cm^{-1} to 3600 cm^{-1} and it corresponds to hydroxyl groups (OH) of clayey minerals of kaolinite group.

The intensive absorption bands with maximum located in the range of wave numbers 3696 cm^{-1} and 3620 cm^{-1} are real signs of kaolinite presence in the samples (Plyusnina, 1967; Plyusnina, 1977; Rumi et al., 2018). Our spectra show a shift of the maximum 3696 cm^{-1} by 2 cm^{-1}. Absorption bands in the range of wave numbers from 3670 cm^{-1} to 3652 cm^{-1} are considered by some experts as the most sensitive to perturbations of hydrogen bonds due to the influence of various factors, including hydrothermal ones (Sergeeva, 2018; Dyatlova, Bobkova, Sergievich, 2019).

To estimate changes in the clay component of rocks as a result of hydrothermal processing, calculated parameter of the relation of intensities of absorption bands in the range of wave numbers 3696 cm^{-1} and 3620 cm^{-1} is used. This indicator, similar to Hinckley radiographic index, allows us to identify patterns of changes in the degree of ordering of the crystal structure of kaolinite minerals (Plyusnina, 1967; Dyatlova et al., 2019). According to experimental data, the higher the value of this relation, the more perfect the structure of kaolinite minerals (Plyusnina, 1967; Dyatlova et al., 2019). Our materials display a shift of wave number maximum by 2 cm^{-1}. The indicator we apply is denoted as $PK_{3698/3620}$. It also estimates in conventional units.

The results of the study

On the example of 19 samples of rocks from Devonian deposits and undifferentiated deposits of Upper Riphean – Lower Vendian of the Ukhta Region, the application of new indicators and their complex analysis with IR-spectroscopy data were tested.

According to data of the distribution of uranium and thorium given in Table 4, it is established that the samples of shale of Upper Riphean – Lower Vendian (R_3-V_1), boundary sediments of Middle Devonian – Upper Devonian (D_2-D_3dž) of Djerskaya suite (D_3dž) differ significantly from the overlying strata of domanic suite (D_3 dm). They also differ in values of the following indicators: *Th/U, U/Th* and $PN_{U\text{-}Th}$.

Based on analysis of IR-spectra in a number of studied samples the presence of kaolinite clays was found (Table 4, column 4). As will be shown below, kaolinite clays and their structural features are important indicators of a number of geological and geochemical processes.

The graph of the IR-spectrum of kaolinite clay sample No. 19/37 (Fig. 3, a) shows the absorption bands of wave numbers in the region of 3698 cm^{-1}, 3670 cm^{-1}, 3652 cm^{-1}, 3620 cm^{-1}. Such values of wave numbers are caused by fluctuations in the bonds of hydroxyl groups (Fig. 3, b).

According to IR-spectroscopy data, column 10 of Table 4 shows the values of the indicator of transformation of the organic matter ($PP_{3040/2920}$ indicator). Column 11 of table 4 shows values of the degree of crystallinity of kaolinite clays ($PK_{3698/3620}$ indicator). Changes in these indicators do not clearly depend on the composition and the age of samples.

The discussion of the results of study

In Table 4 (column 9), according to the distribution of the values of the $PN_{U\text{-}Th}$ indicator, the following can be traced by the section: this section is divided into two parts. In the lower part of the section (R_3-V_1lv, D_2-D_3dž, D_3dž), negative values of the $PN_{U\text{-}Th}$ indicator (from -4.3 to -0.2) are predominant. In the upper part of the section (with a few exceptions), positive values of this indicator are predominant (1.6–5.2). This means that in the lower part of the section, thorium predominates, and in its upper part, uranium predominates. It is well

Suite, index	No. of sample	Geological formation	Kaolinite clay *	U, g/t	Th, g/t	*Th/U*	*U/Th*	*PN_{U-Th}*, g/t	*$PP_{3040/2920}$* in units	*$PK_{3698/3620}$* in units
1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**
D_3vt	Kv/7v	siliceous-clayey-carbonate	KC	n.d.	n.d.	n.d.	n.d.	n.d.	5,0	0,8
D_3vt	15/2	siliceous-clayey	-	4,2	1,4	0,3	3,0	3,7	1,2	0,7
D_3dm	15/6	carbonate- siliceous	KC	4,5	20,4	4,5	0,2	-2,3	8,2	1,1
D_3dm	Kk/2m	siliceous-clayey	-	5,7	1,4	0,2	4,0	5,2	1,8	0,3
D_3dm	Kk/4c	clayey- siliceous	KC	4,5	0,7	0,1	6,4	4,3	1,9	0,7
D_3dm	Kl/133	clayey- carbonate-siliceous	KC	n.d.	n.d.	n.d.	n.d.	n.d.	3,6	1,2
D_3dm	Ky/6n	clayey- carbonate-siliceous	KC	2,8	0,2	0,1	14,0	2,7	0,1	0,7
D_3dm	Ky/7n	limestone	-	2,6	0,7	0,3	3,5	2,5	0,3	n.d.
D_3dm	Ky/8c	carbonate- siliceous	-	n.d.	n.d.	n.d.	n.d.	n.d.	0,3	n.d.
D_3dm	Ky/8n	clayey- siliceous-carbonate	-	2,7	3,2	1,2	0, 8	1,6	0,1	n.d.
D_3dm	Ky/9n	clayey- siliceous-carbonate	KC	2,2	7,1	3,2	0,3	-0,1	0,3	0,7
D_3dm	Ky/10n	siliceous- carbonate	-	4,9	3,6	0,7	1,2	3,7	0,2	n.d.
D_3dm	Ky/ 11	clay	KC	n.d.	n.d.	n.d.	n.d.	n.d.	0,3	*0,3*
D_3dž	1004	sandstone	-	3,7	11,8	3,1	0,3	- 0,2	n.d.	n.d.
D_3dž	1-NS-2b/35a	clay (cement)	KC	n.d.	n.d.	n.d.	n.d.	n.d.	1,0	0,8
D_2 -D_3dž	19/37	clay	KC	2,4	19,0	7,9	0,1	-3,9	10,5	1,1
D_2 -D_3dž	19/49	clay	KC	1,5	17,5	11,2	0,1	-4,3	4,0	1,1
R_3-V_1lv	19/61	shale	-	2,7	21,0	7,1	0,1	-4,3	6,8	n.d.
R_3-V_1lv	19/63	shale	-	2,2	8,7	3,9	0,3	-0,7	0,6	n.d.

Table 4. Characteristic of high-carbon-content samples of rocks and accommodating sediments according to such methods: IR-spectroscopy method, neutron activation method, method of ICP MS. KC – the presence of kaolinite clay; "-" – the absence of attribute; n.d. – no definitions; PN_{U-Th} indicator is indicator of the relational uranium-thorium accumulation in g/t; $PP_{3040/2920}$ indicator is indicator of transformation of the organic matter; $PK_{3698/3620}$ indicator is indicator of the degree of crystallinity of kaolinite clays. Numbers of wells, numbers and names of quarries are given in the legend to Fig. 1.

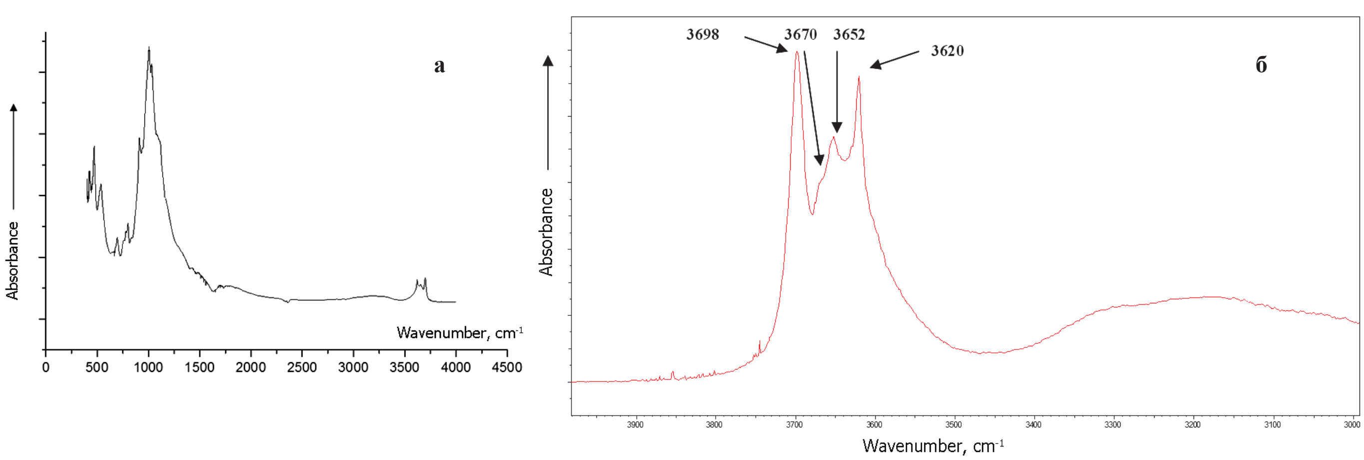

Fig. 3. IR-spectrum of the absorption of kaolinite clay: a – overall look of IR-spectrum of the absorption of kaolinite clay in the range of wave numbers from 4000 cm^{-1} to 400 cm^{-1}; b – part of IR-spectrum in the range of wave numbers from 4000 cm^{-1} to 3000 cm^{-1} with maxima of absorption bands in the region of such wave numbers: 3698 cm^{-1}, 3670 cm^{-1}, 3652 cm^{-1}, 3620 cm^{-1}, such values of wave numbers are specific to fluctuations in the bonds of OH-groups of kaolinite clays (sample No. 19/37).

known that thorium, like uranium, accumulates in acidic igneous rocks, but in clays and shales the thorium content is close to Clarke content. And what, then, can be the reason for the differences in the content of thorium and the content of uranium within the studied stratigraphic range? In all likelihood, the increased or decreased content of these elements is controlled by acid-base parameters and redox conditions of the environment. Thorium belongs to the group of sedentary elements-complexing agents and hydrolysates (namely Ti, Cr, Th, Ga, Sc and others). These elements partially migrate in strongly acidic and strongly alkaline waters. In addition, thorium in the earth's crust is involved in post-magmatic high-temperature hydrothermal processes.

Uranium belongs to the group of anionogenous elements (namely V, U, Mo, Au etc.) that are mobile in an oxidizing environment and inert in a reducing one (Perelman, Kasimov, 1999).

That is, if the inflow of thorium and uranium in the rocks is associated with acid magmatism and subsequent hydrothermal processes, then their increased or decreased content is controlled by acid-base parameters and redox conditions of the environment. In confirmation of this, we can note that in the adjacent territory with the area of investigation in the lower part of the D_2-D_3dž section with an increased content of thorium, not only a titanium deposit was found, but also increased concentrations of Cr, Ga, Sc and other elements in the rocks were found. In the overlying domanic suite, the rocks, along with increased values of uranium, characterized by increased contents of V, Mo and Au (Zavyalov, 1966; Yumanov et al., 2013). In particular, native gold was determined in the bottom samples of the Nizhnyaya Chut River, and a preliminary assessment of vanadium resources was carried out for the Ukhta Fold area (Yumanov et al., 2013).

The oldest formations are characterized by a high content of thorium, varying from 8.7 to 21.0 g/t. Values of the relation of thorium to uranium is also high (from 3.9 to 11.2). As it was shown above, values greater than 7 correspond to oxidized formations and formations formed in coastal-marine facies, values from 3 to 5 correspond to metamorphosed formations.

According to *U/Th* indicator (the relation of uranium to thorium), the considered interval of section has the lowest values – (0.1–0.3), which, together with the data on the thorium content, indicates a predominant accumulation of thorium.

Here, $PN_{U\text{-}Th}$ indicator is characterized by low negative values, which vary from -4.3 to -0.7 g/t. Negative values demonstrate a slower accumulation of uranium in comparison with thorium accumulation, when there is a "lag" of uranium accumulation from thorium accumulation, taking into account the average relation of these elements in the formation as one in three (1:3).

This also confirms the predominant accumulation of thorium in comparison with accumulation of uranium.

In the samples from domanic suite, thorium content varies from 0.2 g/t to 7.1 g/t; uranium content varies from 2.2 g/t to 5.7 g/t.

According to the relation of thorium to uranium, the most samples of domanic suite and one sample of vetlasyanskaya suite are characterized by very low values (less than 1), this fact reflects the presence of silica in the samples. In three samples, values of the relation of thorium to uranium (*Th/U* indicator) are greater than 1. At the same time, value of *Th/U* indicator equal to 1.2 characterizes clayey-siliceous-carbonate formations, while the values equal to 3.2 and 4.5, determined in samples of clayey-siliceous-carbonate and carbonate-siliceous formations, for some reason do not fully correspond to sedimentary geological rocks of this composition.

Negative values of $PN_{U\text{-}Th}$ indicator (-2.3 and -0.1) are determined in two samples (sample No. 15/6, sample No. Ky/9n). Such values are considered as a quantitative characteristic of the "lag" of uranium accumulation in relation to thorium accumulation taking into account the average relation of these elements in the formation as one in three (1:3).

Indicator of transformation of the organic matter ($PP_{3040/2920}$). Values of this indicator vary from 0.1 to 10.5.

Samples from quarry "Yarega" (namely Nos. Ky/6n, Ky/7n, Ky/8n, Ky/8c, Ky/9n, Ky/10n, Ky/11n) are characterized by low values of indicator of transformation, which vary from 0.1 to 0.3.

In the domanic suite, relatively high values (1.8–3.6) are determined in single samples from the following quarries: quarry "Vetlasyan" (sample No. Kv/7v), quarry "Lesnik" (sample No. Kl/133), quarry "Kerbadiel" (samples Nos. Kk/2m, Kk/4c).

The highest values of this indicator, 8.2 and 10.5, were determined in the IR-spectra from the samples of kaolinite clays of the domanic horizon and from the clays of the Djersky horizon.

It should be noted that sample of kaolinite clay from the deposits of vetlasyanskaya suite of Upper Devonian (No. Kv/7v) has a high value of indicator of transformation of the organic matter – 5.0. This value is comparable to the value of indicator of organic matter transformation in metamorphic rock (shale) (6.8). We propose that the presence of kaolinite clays may be a sign of the influence of hydrotherms on formations. As a result of the influence of hydrotherms, organic matter of Upper Devonian of vetlasyansky horizon is more strongly transformed than in the underlying deposits of domanic age. In formations unchanged by hydrotherms, values of indicator of organic matter transformation depend on the composition of geological rocks of the same age: more low values of the indicator are found in limestones and clays, and more high values are found in siliceous formations.

Characteristic of kaolinite clays by indicator of the degree of crystallinity ($PK_{3698/3620}$ indicator). Values, which characterize kaolinite clays by the degree of their structural features and the degree of perfection of their crystal structure, vary from 0.3 to 1.2. The lowest values of such indicator, equal to 0.3, are determined in mainly clayey formations of domanic suite (sample No. Kk/2m). The highest values of indicator 1.1 and 1.2 are found in

D_2-D_3dž clays and in the samples of clayey-siliceous-carbonate sediments of domanic suite.

From the data presented in Table 4, it follows that values of 0.7–0.8 are distributed throughout the Devonian section, i.e. they do not depend on the age and lithological composition of formations. In our opinion, this type of distribution of given indicator by the studied samples may be related with superimposed hydrothermal processes.

The formation of kaolinite clays is usually associated with the destruction of weathering crusts and redeposition of clayey minerals. However, according to the latest data, a new formation of kaolinite also happens in the case of modern hydrothermal processes (Chernov et al., 2019). In addition, it has been experimentally shown that for "all kaolins" a kaolinite crystallinity index (Hinckley radiographic index) increases as a result of hydrothermal action (Yevtushenko et al., 2012).

The influence of low-temperature hydrotherms in the domanic suite is clearly shown in the data obtained from "Kerbadiel" quarry. So, in the sample of domanic suite No. Kk/2m, value of indicator of the degree of crystallinity is 0.3, and in the sample No. Kk/4c value of this indicator is 0.7, which indicates a higher degree of crystallinity of the second sample. These samples also differ visually: the first sample shows signs of flexibility and traces of bitumoids. These samples also differ by their lithological composition: the first sample is more clayey, the second is more siliceous (Table 4). Value of the indicator of transformation of the organic matter in the second sample (1.9) is slightly higher than in the first sample (1.8). Value of the indicator of the relational uranium-thorium accumulation (in g/t) in the second sample No. Kk/4c (4.3) is lower than in the first sample No. Kk/2m (5.2). In our opinion, such changes in the samples located in the same section are associated with a mixed result of hydrothermal processing of mainly clayey and mainly siliceous formations.

Summarizing the results of investigations and data presented in Table 4, we note the following: kaolinite clays of hydrothermal nature are characterized by relatively high values of two indicators: indicator of the degree of crystallinity and indicator of transformation of the organic matter. When comparing all these indicators with the content of thorium and uranium, there are differences in their content depending on the composition of geological rocks. In terrigenous sediments, negative values of indicator of the relational uranium-thorium accumulation ($PN_{U\text{-}Th}$) are traced, while in carbonate sediments, on the contrary, its mostly positive values are established. The presence of a source of thorium admission in Devonian carbonate rocks is estimated by negative values of $PN_{U\text{-}Th}$ and by high values of Th/U indicator (aka the thorium-uranium relation). As for the comparison of indicators in the Table 4, such as KC, $PK_{3698/3620}$, $PP_{3040/2920}$, $PN_{U\text{-}Th}$, it is possible to identify a certain relationship between them. There is a direct relationship between the presence of kaolinite clays and the lowest (negative) values of the uranium-thorium accumulation indicator $PN_{U\text{-}Th}$ (from -4.3 to -2.3); at the same time, there is a high content of thorium in the rock (up to 17.5–21.0 g/t), which is generally characteristic of high-temperature hydrothermal processes. Simultaneously with the lowest negative values of $PN_{U\text{-}Th}$ indicator, the rock samples are characterized by high values (from 3.6 to 10) of the organic matter conversion indicator $PP_{3040/2920}$, as well as relatively high values (1.1–1.2) of the crystallinity indicator of kaolinite clays $PK_{3698/3620}$. Taken together, these values may indicate the thermal transformation of the rock mineral matrix and organic matter as a result of hydrothermal action. This transformation leads to the leaching of a number of elements and the formation of a void space, i.e., to the improvement of the reservoir properties of the rock. This can be directly related to the presence of reservoirs in the D_2-D_3dž sediments filled with hydrocarbon components.

Comparing our materials with the results of other researchers, it is necessary to provide data on the nature of the transformation of clays under the influence of acidic fluid of hydrothermal clays, situated in a modern thermoanomaly called Nizhne-Koshelevskaya. Thus, in the near-surface conditions of hydrothermal clays, "kaolinite is often formed, characterized by high structural defects, poor crystallization of particles and their high dispersion..." (Chernov et al., 2019). It is established that layers of such clays have rather high hydrophilic properties and flexibility in comparison with well-crystallized kaolinite in the lower part of section. At the same time, flexible clay layers with hydrophilic properties of clays are considered as an anti-filtration screen and geochemical barrier. They prevent rapid removal of deep fluid and create favorable conditions for mineral formation and ore formation in the modern environments (Chernov et al., 2019).

Thus, the important ore-forming role of kaolinite clays in the superimposed hydrothermal processes and in the similar ancient environments is not excluded.

On the other hand, in the practice of oil-and-gas exploration within the Tatar arch in the Tatarstan Republic, the influence of superimposed hydrothermal processes was revealed, which "manifested itself in the formation of a specific complex of clayey minerals". In particular, it was found that "increased porosity values are associated with sections and intervals of geological rocks with a predominance of kaolinite component" in the weathering crusts of crystalline basement (Sidorova, Sitdikova, 2013).

In this regard, we consider another important role of hydrothermal kaolinite clays with high crystallinity, namely, as a sign of an increase in the effective porosity in shale, which is actual when identifying secondary reservoirs. In the basement shale in the area of Ukhta fold, as well as within the Tatar arch, there are faults and numerous cracks. In addition, they are significantly elevated compared to the adjacent oil-and-gas-bearing Izhma-Pechora depression, which also suggests the possibility of migration of hydrocarbons through fluid-conducting zones and faults. In this regard, it is possible that ancient metamorphosed shale may become perspective oil-and-gas exploration objects.

Conclusions

This paper justifies the use of gamma-spectroscopic method and a number of indicators, including two new ones, as a reliable express tool for identifying factors of regional and local accumulation of uranium in domanic sediments and other high-carbon-content sediments.

1. Methodological part of the work shows wide possibilities of using gamma-spectroscopic and IR-spectroscopic data to identify searching criteria in the oil geology and in the ore geology. New indicators are proposed that, in contrast to the thorium-uranium relation, allow us to determine the processes of preferential accumulation of uranium or thorium, which allows us to more reasonably identify the sources of inflow of these elements.

2. On the example of sediments from Upper Riphean – Lower Vendian and Middle – Upper Devonian of the Ukhta fold a testing of all these indicators in combination with IR- spectroscopic data was carried out. Different information content of the indicator of thorium-uranium relation, the indicator of uranium-thorium relation, as well as the indicator of the relational uranium-thorium accumulation for carbonate and terrigenous geological rocks is shown. The last two indicators (out of the three listed) are the most informative for Upper Devonian carbonate rocks, while the indicator of thorium-uranium relation is an informative indicator mainly for terrigenous rocks.

3. The presence of kaolinite clays was determined by the IR-spectroscopic method data in different parts of the section from Upper Riphean – Lower Vendian to Upper Devonian (vetlasyansky horizon). For the samples from formations, the characteristics of indicator of transformation of the organic matter ($PP_{3040/2920}$) and indicator of the degree of crystallinity of kaolinite clays ($PK_{3698/3620}$) were obtained.

4. In our materials two ways of formation of kaolinite clays are noted: these clays can be formed as a result of destruction of the weathering crust and under influence of the hydrothermal processes, so it is important to distinguish between these processes. When analyzing the actual materials from wells and outcrops on the basis of IR-spectroscopic method data, it was found that high values of indicator of transformation of the organic matter in some cases do not depend on the age and composition of formations. This fact is considered by us as a sign of hydrothermal influence on the geological rocks of Middle Devonian and Upper Devonian in this region.

5. Features of kaolinite clays identified by us, due to hydrothermal influence, are characterized by relatively high values of the degree of crystallinity of kaolinite clays. Values of indicator of transformation of the organic matter for a number of samples is also increased. At the same time, negative values of indicator of the relational uranium-thorium accumulation (PN_{U-Th}) are observed in terrigenous sediments, while in carbonate sediments, on the contrary, it's mostly positive values are established.

6. If the upper Devonian carbonate rocks have negative values of indicator PN_{U-Th}, and indicator of thorium-uranium relation is high, then we can assume that there is a source of thorium admission, including in connection with erosion and redeposition of more ancient rocks.

In total, the estimation of uranium and thorium content by non-destructive gamma-spectroscopy method in conjunction with data of IR-spectroscopy method opens up a wide range of possibilities for detecting correlations between the composition of rocks, radioactive elements and metals determined by other methods.

This is especially important because the accumulation of uranium in the Riphean and Devonian high-carbon-content strata is accompanied by the accumulation of a number of elements in the ore-bearing and on-board concentrations (Laptev et al., 2017; Gorobets et a., 2018).

We plan to present for more details the characteristics of associations of metals with ore-bearing content and on-board content in the sections of Ukhta Region in subsequent publications.

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