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Satbayev University

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
Satbayev University

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Amangeldykyzy A.*, Kopobayeva A.N., Bakyt A., Ozhigin D.S., Blyalova G.G.

Karaganda Technical University, Karaganda, Kazakhstan.
E-mail: amangeldykyzy@inbox.ru

MINERALOGY AND GEOCHEMISTRY OF THE SHUBARKOL DEPOSIT JURASSIC COALS

Abstract. This article studies mineralogy of Jurassic coals of the Shubarkol deposit using the method of scanning electron microscopy (SEM-EDX) Hitachi S-3400N that was used at the Uranium Geology International Innovation-scientific Education Center of the Tomsk Polytechnic University Department of Geoecology and Geochemistry. In the course of study, maps of the metal distribution of metals were built in the QGIS program, the content of which was obtained by instrumental neutron activation analysis (INAA) at the nuclear geochemical laboratory of the Department of Geoecology and Geochemistry of National Research Tomsk Polytechnic University (TPU) (analyst A.F. Sudyko). The choice of this object of study was determined by the tasks of research that included studying the regularities of the abnormal concentrations of metals accumulation, the geological environment various factors effect on the levels of their accumulation in coals, as well as the conditions of their concentration and forms in coals. The samples for the study were taken by the channel sampling in the Central and Western sections of the field, as well as by the core method in the Eastern section. According to the results of scanning microscopy, the only scandium-containing mineral particle of complex Si-Al-Ca-Zr-Sc-Ti-O composition (~ 0.66 % Sc) was established. Mineral inclusions of complex composition (Al-Si-Zr-K-Ca-Sc-Ti) with the presence of its analogue Zr in coals were identified, as well as the presence of small grains of zircon and titanium oxides that could be considered as the main concentrators of zirconium and hafnium. The correlation analysis of Nb and Ta in the coals and coal-bearing rocks of the Shubarkol deposit showed a significant correlation between the elements, the highest significant correlation was found with Zr, Hf, Th, Sc and rare earth elements. Celestine was identified as the main mineral form of strontium in the coals of the Shubarkol deposit; Sr-barite was also found that was probably formed during the coals oxidation. Minerals are predominantly of authigenic origin.

Key words: coal, mineralogy, geochemistry, Shubarkol, scandium, hafnium, niobium, coal seams.

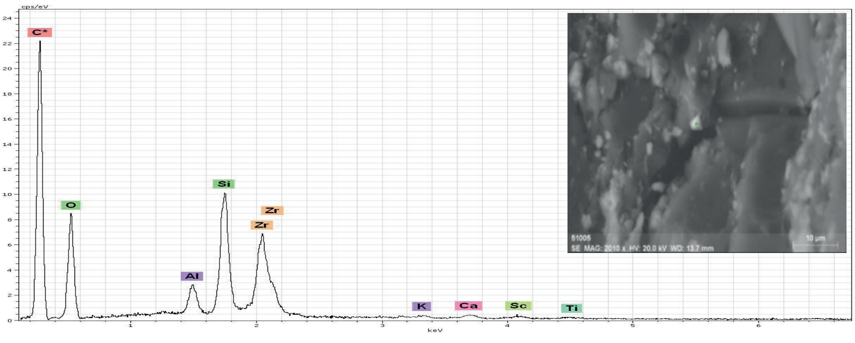
Introduction. Mass-scale studies of trace elements in coal deposits and basins of all the continents carried out over the past few decades have shown [1-3] that coals are the concentrators of many valuable metals including rare and scattered ones; this has led to the fact that coal is used not only as a fuel and energy raw material but also as a promising source of a large group of rare, scattered and noble metals [2-5]. The most complete sets of elements are characteristic, which are found in the coals of almost every coal basin, of any genetic type and age, brown and black ones [1-7]. The differences are mainly determined by the tectonic position of the coal basin or deposit in a particular metallogenic province, the concentration of the local geochemical background [8]. One of such striking examples is the Shubarkol deposit consists in that the composition of coals is diverse, they contain various impurity elements and rare metals. Studies have shown [1] that the deposit contains significant contents of Ba, Sr, U, Th, Rb, Fe, Co, Ce, Zr, Zn and Sc. Studying the distribution of trace elements in coals and the forms of their occurrence can provide important information for understanding in what environment of coal formation they appeared, as well as what syngenetic and epigenetic processes they are associated with [9]. Minerals in coals are usually formed due to the input of terrigenous material, ground and sea water, fallout of volcanic ash, and intrusion of igneous rocks [9, 10]. Some researchers point out [2, 11, 12] that igneous rocks and hydrothermal fluids are among the main geological factors that can cause increasing the content of REE and trace elements in coals. Most studies have shown that carbonate minerals are often present in thermally exposed coals and are possibly formed of volcanic hydrothermal solutions that have entered the coal and deposited in cracks [11, 12, 13].

Characteristic of the object of study. The object of study is the Shubarkol coal deposit. It was formed in the inherited depression of folded areas formed on the Sarysu-Teniz uplift. The basin of the field developed over ancient sedimentary complexes that are part of the structural framework of the Caledonian accretionary-folded areas confined to the central part of the Sarysu-Teniz uplift or fault-shear zone, a large tectonic structure of the Western part of the Central Asian orogenic belt (CAOB). The deposit is a gently sloping asymmetric sub-latitudinal trough filled with Lower Jurassic continental deposits, in the section of which there are three coal horizons (Upper, Middle and Lower) of complex structure with the total working thickness of 5.0 to 47.0 m. The deposits are represented by fine and coarse-grained sandstones, siltstones, mudstones, loamy rocks and coals. Mudstones, siltstones and coals predominate over all the rocks.

Methods of study. In total, 25 samples of Jurassic coals and mudstone were taken from the Shubarkol deposit using the channel and core sampling methods. Samples of the Central and Western sections were taken by the gross method, the sampling interval was kept within 15-35 m. The rocks of the western wing of the Central section were sampled by the point method. The rocks of the Eastern section were sampled by the core method, the sampling interval was kept within 5-20 m. Using the primary samples, 1 cm*1 cm briquettes were made for scanning electron microscopy, which were used to study and to determine the morphology and the composition of minerals. The forms of occurrence of minerals in coal samples were investigated using SEM-EDX. The geochemical content of coals was determined by the method of instrumental neutron activation analysis (INAA) at the nuclear geochemical laboratory of the Department of Geoecology and Geochemistry of National Research Tomsk Polytechnic University (TPU) (analyst A.F. Sudyko).

Results of study and their discussion. Based on the results of the scanning microscopic analysis, various mineral impurities were found in the composition of the Shubarkol deposit coals. These are mainly aluminosilicates, sulfides and sulfates with inclusions of microparticles of rare and rare earth elements, and the results of instrumental neutron activation analysis revealed anomalous concentrations of Sc, Ta, Nb, Hf, Zr, Ba, Sr, Ce and REE. The forms of occurrence of minerals and geochemistry of some metals abnormally contained in coals were studied. Scandium. Sc-rich minerals are rarely found in abnormally Sc-containing coals [14]. This study revealed unidentified minerals of complex Si-Al-Ca-Zr-Sc-Ti-O composition (~ 0.66 % Sc) (Figure 1). These materials are isolated authigenic mineral formations, but their contribution is rather small and does not contain the total amount of Sc determined in the samples, especially in the abnormally Sc-containing coals. Figure 2 shows the map of the Sc distribution in the coals of the Central and Western sections of the Shubarkol deposit. When constructing the map for the correct assessment of the nature of the element distribution, anomalous contents of the element in the Eastern section were excluded.

Due to the fact that in the coals subjected to weathering at the Shubarkol deposit, according to the results of laboratory studies, high concentrations of impurity elements have been revealed, it makes sense to consider the behavior of scandium during the organic matter oxidation. This phenomenon can be observed on the example of the Eastern section of the Shubarkol field.



Increasing the content of scandium in coal within the oxidation zone was found in the studied local area of the field along wells No. 1, 2, 3 (Figure 3a).

Figure 1. Unidentified complex (Si–Al–Ca–Zr–Sc–Ti–O) composition minerals.

From the graph it follows that the Sc content is higher in oxidized coals, where the mass fraction of organic matter is lower. Possibly, under oxidation conditions, scandium was released from the organic matter of coal followed by its secondary concentrating.

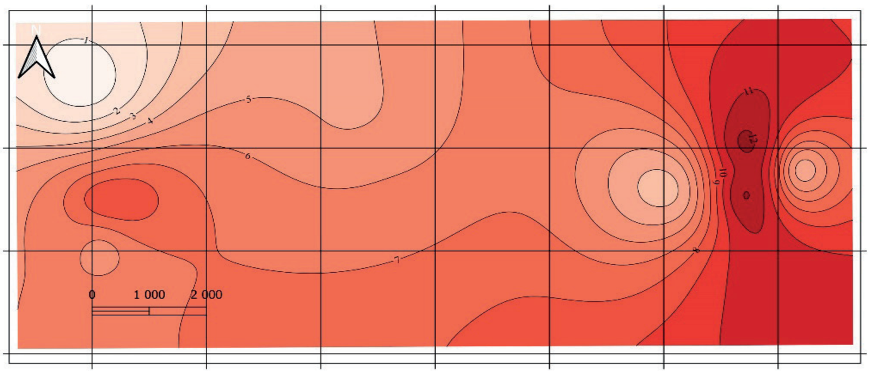


Figure 2. The map of Sc distribution in the coals of Central and Western sections of the Shubarkol deposit

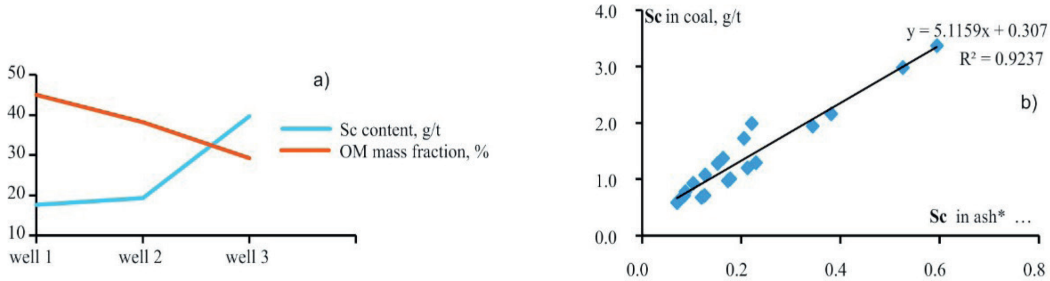


Figure 3. The graph of Sc content, organic mass (OM) fraction in oxidized coals of the Eastern section (a); correlation graph of the Sc content in coal, in coal ash (obtained by recalculation) (b)

The conditions for accumulation of high concentrations of scandium in coals are poorly studied, and at present there are no clear ideas on this issue. The available information is very fragmentary, often contradictory and does not give a clear picture of the factors responsible for anomalous concentrations accumulation. There is no clear idea of the forms of scandium concentration. The average content of scandium in terms of ash from the Shubarkol deposit is 90 g/t, and in coal-bearing rocks 2226 g/t. Accumulation of such high metal concentrations is hardly possible only due to the clastogene matter. It is obvious that other processes of element concentration also took place there.

The syngenetic accumulation of scandium in the composition of the clastogene matter has been established; there is no doubt of the mineral substance of coals role in the concentration of scandium. This is evidenced by the direct correlation dependence of its content in coal on its content in coal ash (Figure 3b).

In high-ash but scandium-poor coals the fraction of the clastogene component is higher, and in metal-rich low-ash coals it is lower. Some part of mineral impurities in the process of peat formation in the aggressive environment of the peat bog is destroyed, new minerals are formed, mainly carbonates, sulfides and kaolinite, but in general, the role of clastic material in scandium the accumulation is quite significant. In the case when coals with low levels of metal accumulation characterized by the normal or increased ash content are studied, as clastogene scandium predominates a rule. In those cases when coal enriched with it is considered, the proportion of the clastogene part is significantly reduced and the role of scandium associated with the organic part of the coal increases.

Epigenetic accumulation of scandium in the coals of the Shubarkol deposit is possible but it is distributed only in the Eastern section of the deposit, since oxidized weathered coals are predominantly distributed in this area. An example of epigenetic accumulation is the data of the scandium behavior during weathering and oxidation of coal seams (Table 1).

Table 1. Sc average content in the Shubarkol deposit coals, g/t

Element	In unoxidized coals	In oxidized coals	In oxidized weathered coals	Clarke for coal *	In rock interlayers	Clarke for sedimentary rocks**
Sc	1.37	8.3	25.5	4	289.49	9.6

*- acc. to Ya.R. Yudovich, M.P. Ketris, 2005; **- acc. to N.A. Grigoryev, 2003

Hafnium and zirconium. Hafnium and zirconium have very similar chemical properties. Hafnium does not have its own minerals; however, it is a constant companion of zirconium in all its minerals, since it has a close ionic radius (Zr 0.74, Hf 0.75). Hafnium is a geochemical analogue of zirconium; therefore, it is assumed that zircon and baddeleyite are also carriers of hafnium [14, 15].

The factors affecting the zirconium concentration in coal seams are not yet fully known. However, according to [15], the main supplier of zirconium to the coal seam is considered to be terrigenous and volcanogenic solid material rather than aqueous solutions. As for zirconium, the factor of Hf accumulation in coals can be the erosion of alkaline rocks in the sources of drift. An important factor in Hf accumulation in coals could be pyroclastics and the discharge of carbon dioxide thermal waters with an increased content of zirconium and hafnium into the peat bog (or into coal seams). If the Zr/Hf ratio in coals is lower than their clarke in sedimentary rocks, then it can be argued that hafnium could accumulate in coals rather than its geochemical analogue Zr [15].

Figure 4 shows the nature of the relationship between the levels of zirconium and hafnium accumulation in coals and coal-bearing rocks. On the Zr/Hf diagram of the ratio for coals (Figure 4a), there is a weak correlation, which indicates different levels of accumulation and sources of these elements input in coals, however, on the Zr/Hf diagram, the ratios for coal-bearing rocks (Figure 4b) there is a high correlation between the elements accumulation, which is explained by the sorption capacity of the mudstones containing Shubarkol coals [16]. According to [16], where there were studied samples of coal and carbonaceous rocks, it was revealed that carbonaceous rocks had the highest sorption capacity, which exceeded that of coals.

In the Shubarkol deposit coals hafnium is distributed unevenly and has an average content below the clarke. The average content in unoxidized coals of the Central and Western sections is 0.362 g/t. However, its maximum concentrations in the coals of the deposit are confined to the Central section of the deposit (Figure 5).

When studying the Shubarkol deposit coals on a scanning electron microscope, no mineral phases with the presence of Hf have been found, however, there are mineral inclusions of a complex composition (Al-Si-Zr-K-Ca-Sc-Ti) with the presence of its analogue Zr in coals (Figure 6a), as well as the presence of small grains of zircon and titanium oxides (Figure 6b), which can be considered as the main concentrators of zirconium and hafnium. A high content of zirconium in the coals and a low ash content of the coals of the Shubarkol deposit suggest the authigenic nature of these minerals [2].

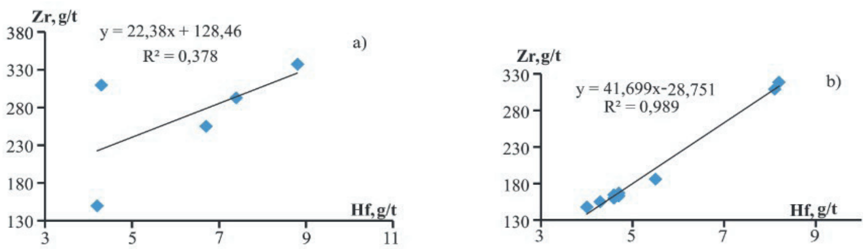


Figure 4. Zirconium to hafnium ratio in coals (a) and coal-bearing rocks (b) of the Shubarkol deposit

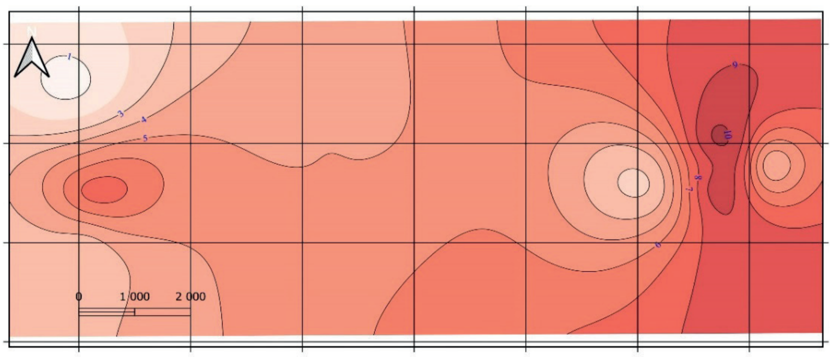


Figure 5. The map of the Hf distribution in the coals of Central and Western sections of the Shubarkol deposit

Electron microscopic studies of coal and coal-bearing rocks have shown the presence of small grains of zircon and titanium oxides, which can be considered as the main concentrators of zirconium and hafnium.

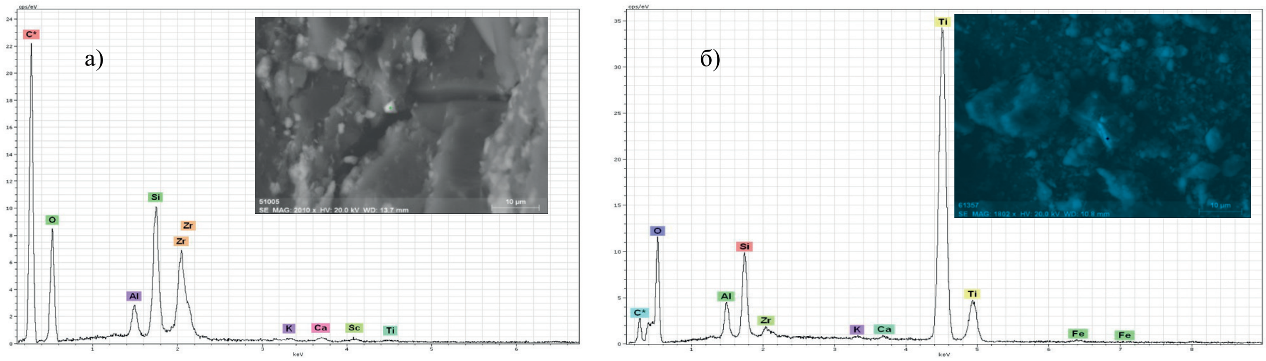


Figure 6. Mineral inclusions of complex (Al-Si-Zr-K-Ca-Sc-Ti) composition (a); titanium oxide with Zr inclusions (b)

The correlation analysis indicates a high significant positive relationship in Hf coals with La, Sc, Ta, Nd, Cs, and Tb and other lithophylic rare metals. This allows assuming accumulation of a significant part of hafnium not due to the clastogene substance but its sorption concentration from aqueous solutions and possibly, biogenic accumulation.

Niobium and tantalum. Niobium and tantalum in endogenous formations are considered together because they are a geochemically related pair of elements. According to [15], the average niobium content in coals in the world is 4.0 g/t, in coal ash 22 g/t.

Tantalum is a geochemical analogue of niobium but with an order of magnitude lower clarke. Its poor knowledge is caused by analytical methods of determination, nevertheless, due to the introduction of INAA and mass spectrometry into practice, more data of tantalum have been obtained at present. Its average content in coal in the world is 0.30 g/t, in coal ash 2.0 g/t, according to [15].

The average tantalum content in unoxidized coals of the Shubarkol deposit estimated according to INAA data is 0.06 g/t.

The distribution of tantalum in coal-bearing deposits is very uneven. The correlation analysis of Nb and Ta in the coals and coal-bearing rocks of the Shubarkol deposit (Figure 7) showed a significant correlation between the elements (0.6 and 0.7, respectively). The highest significant relationship was also found with Zr, Hf, Th, Sc and rare earth elements. The correlation analysis indicates the accumulation of the bulk of elements in heavy accessory minerals.

When studying the Shubarkol deposit coals using an electron microscope, the micromineral forms of Nb and Ta were not found, which is probably due to the limited capabilities of the method used.

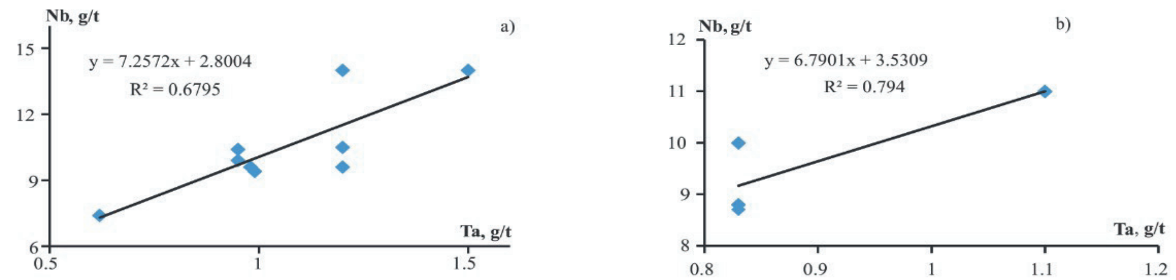


Figure 7. Niobium to tantalum ratio in coals (a) and coal-bearing rocks (b) of the Shubarkol deposit

Thus, the studies carried out and the literature analysis show that the concentration of tantalum and niobium in coals is due to both their accumulation in the clastogene matter and their concentration in the organic mass of coal. Abnormal concentrations are of the chemogenic-sorption nature. Tantalum and niobium concentrated in organic matter can be present in it both in sorbed form and in the form of finely dispersed minerals phases.

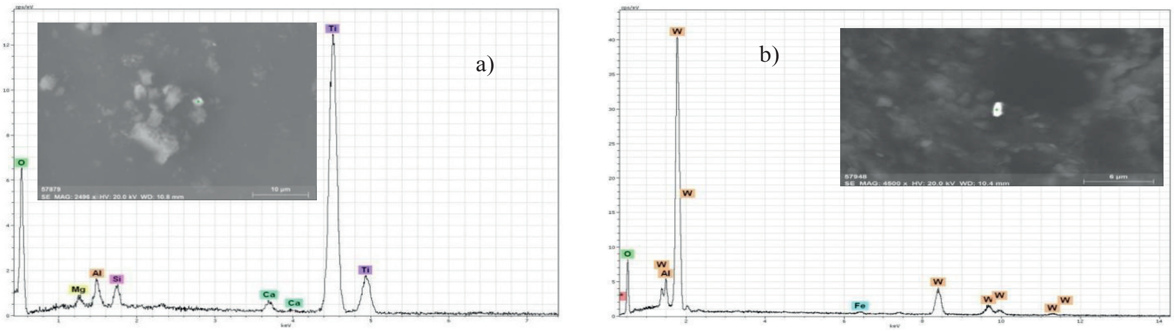


Figure 8. Titanite (sphene) particle (a); native tungsten particle (b)

The condition for sorption accumulation in coals is the introduction of metals by ground or surface waters. Coal enrichment with tantalum and niobium is possible due to their partial leaching from coal-bearing rocks.

Other impurity elements and their mineral forms. It should also be noted that there are other mineral phases in the coals of the Shubarkol deposit discovered during the studies. For example, Figure 8a shows titanite (CaO-TiO_2-SiO_2). In the samples of oxidized coal, particles of native tungsten were revealed; in general, all the grains have an irregular shape (Figure 8b). Together with strontium sulfate in the sample, barite particles are most often encountered (Figure 9a). Barite crystals have a tabular appearance. The barium content is several times higher (1173.7 g/t) than the clarke value, which in turn is 150±20 g/t [15].

According to [15], the average strontium content for bituminous coals is estimated at 120±10 g/t. In the studied coals, the strontium content is above the limit (689.3 g/t) of detection by neutron activation analysis, and electron microscopic studies have revealed several mineral phases containing strontium.

It was mainly found in the composition of strontium sulfates (Sr-S-O, presumably celestine) and iron oxides (Figure 9b).

Celestine grains were found in weathered coal seams, but the concentration in mudstones was several times higher than in coals.

The form of the precipitates is diverse, from irregular microinclusions (~ 2 μm) to well-faceted crystals ~ 6 μm in size. The average selenium content in coal in the world is 1.0±0.15 g/t, in the weathered coals of the Shubarkol deposit the selenium concentration shows a near-clarke value. Most often it occurs together with copper and iron sulfides or aluminosilicate accumulations (Figure 10a, b). Tymmanite is the mineral of selenium accumulation in the form of small grains.

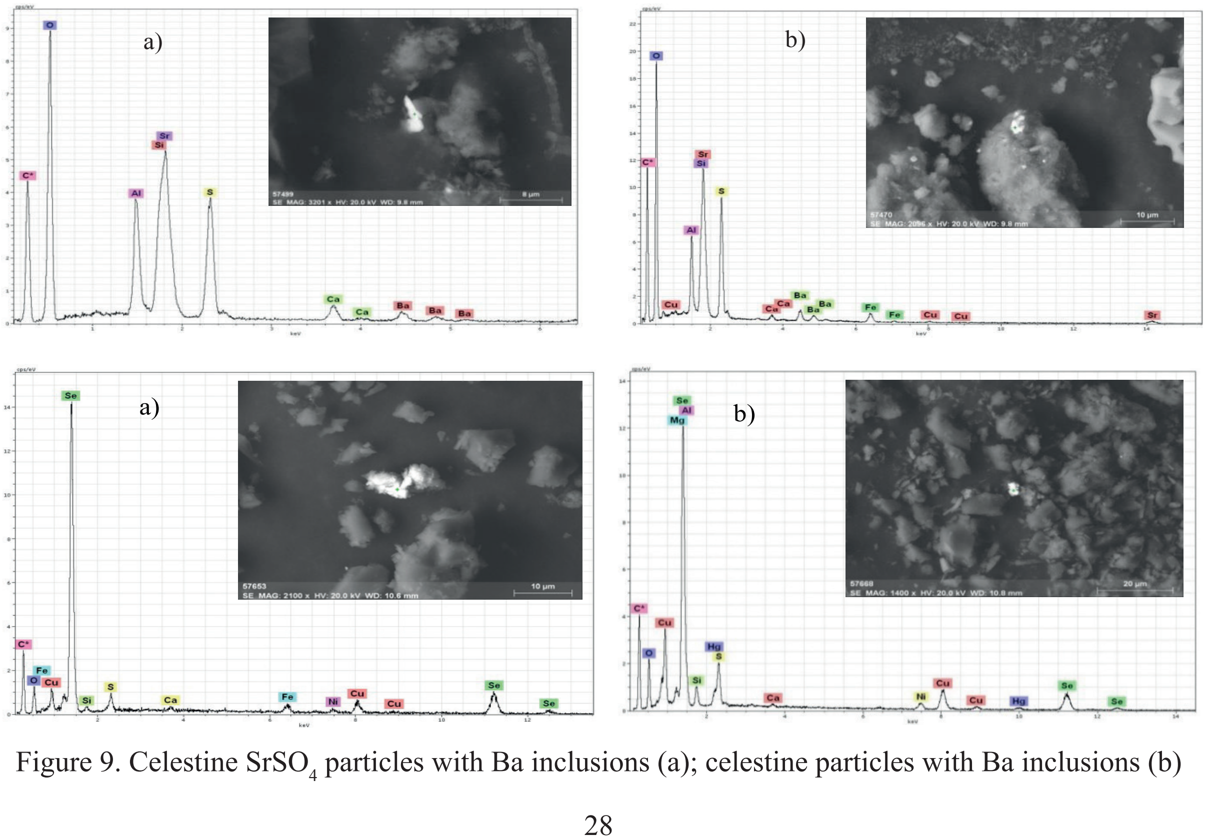


Figure 9. Celestine $SrSO_4$ particles with Ba inclusions (a); celestine particles with Ba inclusions (b)

Conclusion. The results obtained indicate that the coal mineralogy of the Shubarkol deposit is diverse. Scandium is not characterized by its own mineral forms. The only scandium-containing mineral particle of complex composition Si-Al-Ca-Zr-Sc-Ti-O (~ 0.66% Sc) was identified. The irregular shape and complex composition of the mineral particle suggests its authigenic formation.

When studying the Shubarkol coals on a scanning electron microscope, no mineral phases with the presence of Hf were found, however, there were mineral inclusions of a complex (Al-Si-Zr-K-Ca-Sc-Ti) composition with the presence of its analogue Zr in coals, as well as the presence of small grains of zircon and titanium oxides that which could be considered as the main concentrators of zirconium and hafnium. A high content of zirconium in

Figure 10. Copper sulfides with Se inclusion (a); aluminosilicates with tymmanite inclusion HgSe (b) the coals and a low ash content of the coals of the Shubarkol deposit suggest the authigenic nature of these minerals.

The correlation analysis of Nb and Ta in coals and coal-bearing rocks of the Shubarkol deposit showed a significant correlation of elements (0.6 and 0.7, respectively), the highest significant correlation was found with Zr, Hf, Th, Sc and rare earth elements. The correlation analysis indicated accumulation of the bulk of elements in heavy accessory minerals. An impurity of zirconium was found in the Sc-containing grain of complex composition.

Celestine was identified as the main mineral form of strontium in the coals of the Shubarkol deposit; Sr-barite was also found. It was probably formed during the coal oxidation. Both minerals are authigenic in origin. Studies of this kind are of very important practical interest, since various methods are used in the world practice aimed at extracting valuable elements of impurities.

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Амангелдіқызы А.*, Копобаева А.Н., Бақыт Ә., Ожигин Д.С., Блялова Г.Г.

Қарағанды техникалық университеті, Қарағанды, Қазақстан.
E-mail: amangeldykyzy@inbox.ru

ШҰБАРКӨЛ КЕН ОРНЫНДАҒЫ ЮРА КӨМІРІНІҢ МИНЕРАЛОГИЯСЫ ЖӘНЕ ГЕОХИМИЯСЫ

Аннотация. Бұл мақалада Шұбаркөл кен орнындағы юра кезеңінің көмірлерінің минералогиясына, ТПУ-дағы Геоэкология және геохимия кафедрасына қарасты, «Уран геологиясы» ХЗҒБО-да жүргізілген сканерлеуші электронды микроскопия (SEM-EDX) Hitachi S-3400N әдісін пайдалана отырып зерттеу жүргізілді. Зерттеу барысында QGIS бағдарламасы арқылы металдардың таралу карталары жасалды (талдаушы А.Ф. Судыко). Ондағы құрамдық ақпараттар Томск ұлттық зерттеу политехникалық университетінің (ТПУ) геоэкология және геохимия кафедрасының ядролық-геохимиялық зертханасында аспаптық нейтрондық-активациялық талдау (ИНАА) әдісімен алынған. Зерттеу объектісін таңдау, олардағы металдардың қалыптан тыс концентрациясының жинақталу заңдылықтарын, геологиялық ортаның әртүрлі факторларының көмірде жинақталу деңгейіне әсерін, сондай-ақ оның шоғырлану шарттары мен көмірде болу формаларын зерттеу міндеттерімен анықталды. Зерттеу үшін сынамалар кен орнының Орталық және Батыс учаскелерінде бороздалық сынамалау әдісімен, сондай-ақ Шығыс учаскелерінде керндік тәсілмен алынды. Сканерлеу микроскопиясының нәтижелері бойынша, күрделі құрамды Si-Al-Ca-Zr-Sc-Ti-O (~0,66% Sc) құрамында скандий бар жалғыз минералды бөлшек анықталды. Көмірде тағы бір күрделі құрамды минералды қосындыда (Al-Si-Zr-K-Ca-Sc-Ti) Zr аналогы анықталды, сонымен қатар цирконий мен гафнийдің негізгі концентраторлары ретінде қарастыруға болатын ұсақ циркон түйірлері мен титан оксидтерінің кездесуі байқалды. Шұбаркөл кен орнының көмірі мен көмір сыйыстырушы жыныстарындағы Nb және Ta корреляциялық талдауы элементтердің маңызды корреляциялық байланысын көрсетті, Zr, Hf, Th, Sc және сирекжер элементтерімен ең жоғары маңызды байланыс орнатылды. Шұбаркөл кен орнының көмірінде стронцийдің негізгі минералды түрі ретінде – целестин, сондай-ақ көмірдің тотығу процесінде пайда болған Sr-барит табылды. Минералдардың жаралуы негізінен аутигенді болып табылады.

Түйінді сөздер: көмір, минералогия, геохимия, Шұбаркөл, скандий, гафний, ниобий, көмір қабаттары.

Амангелдіқызы А.* , Копобаева А.Н., Бакыт А., Ожигин Д.С., Блялова Г.Г.

Карагандинский техничеcкий унивеситет, Караганда, Казахстан.
E-mail: amangeldykyzy@inbox.ru

МИНЕРАЛОГИЯ И ГЕОХИМИЯ ЮРСКИХ УГЛЕЙ МЕСТОРОЖДЕНИЯ ШУБАРКОЛЬ

Аннотация. В данной статье изучается минералогия юрских углей месторождения Шубарколь с использованием метода сканирующей электронной микроскопии (SEM-EDX) Hitachi S-3400N, которая проводилась в МИНОЦ «Урановая геология» при кафедре геоэкологии и геохимии ТПУ. В ходе исследования построены карты распределения металлов в программе QGIS, содержания которых получено методом инструментального нейтронно-активационного анализа (ИНАА) в ядерно-геохимической лаборатории кафедры геоэкологии и геохимии Национального исследовательского Томского политехнического университета (ТПУ) (аналитик А.Ф. Судыко). Выбор данного объекта изучения определялся задачами исследований, включающими изучение закономерностей накопления в них аномальных концентраций металлов, влияния различных факторов геологической среды на уровни накопления их в углях, а также условий его концентрирования и форм нахождения в углях. Пробы для исследования были взяты бороздовым способом опробования на Центральных и Западных участках месторождения, а также керновым способом на Восточном. По полученным результатам сканирующей микроскопии установлена единственная скандийсодержащая минеральная частица сложного состава Si-Al-Ca-Zr-Sc-Ti-O (~0,66 % Sc). Выделены минеральные включения сложного состава (Al-Si-Zr-K-Ca-Sc-Ti) с присутствием его аналога Zr в углях, а также обнаружено наличие мелких зерен циркона и оксидов титана, которые могут рассматриваться в качестве основных концентраторов циркония и гафния. Корреляционный анализ Nb и Ta в углях и углевмещающих породах Шубаркольского месторождения показал значимую корреляционную связь элементов, наиболее высокая значимая связь установлена с Zr, Hf, Th, Sc и редкоземельными элементами. В качестве главной минеральной формы стронция в углях месторождения Шубарколь установлен целестин, также обнаружен Sr-барит, образовавшийся, вероятно, в процессе окисления углей. Минералы преимущественно имеют аутигенное происхождение.

Ключевые слова: уголь, минералогия, геохимия, Шубарколь, скандий, гафний, ниобий, угольные пласты.

Information of the authors:

Amangeldykyzy Altynai – PhD student of Karaganda Technical University, Master in Science of Engineering, specialty Geology and Exploration of Mineral Resources, Karaganda, Republic of Kazakhstan; amangeldykyzy@inbox.ru; *https://orcid.org/0000-0002-6665-8804;*

Kopobayeva Aiman Nygmetovna – PhD, senior lecturer of the Department of Geology and Exploration of Mineral Resources, Karaganda Technical University, Karaganda, Republic of Kazakhstan; *aiman_25.87@mail.ru*; *https://orcid.org/0000-0002-0601-9365;*

Bakyt Aigerim – master student of the Department of Geology and Exploration of Mineral Resources, Karaganda Technical University, Karaganda, Republic of Kazakhstan; *b_aigerim_98@mail.ru*; *https://orcid.org/0000-0003-0661-310X;*

Ozhigin Dmitry Sergeyevich – PhD, senior lecturer of the Department of Mine Surveying and Geodesy, Karaganda Technical University, Karaganda, Republic of Kazakhstan; *ozhigin.dima@mail.ru*; https://orcid.org/0000-0002-2443-3068;

Blyalova Gulim Galymzhanovna – Master in Science of Engineering, specialty Geology and Exploration of Mineral Resources, Karaganda Technical University, Karaganda, Republic of Kazakhstan; krg_gulim@mail.ru; *https://orcid.org/0000-0001-8801-8683.*

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