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The use of chlorine-containing agents in the processing of spent blocks of uranium deposits

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ABSTRACT

The work is aimed at diversifying existing mines for the extraction and processing of natural uranium through additional processing of spent blocks of uranium deposits with chemical solutions using the method of in-situ well leaching (ISL) in order to extract associated useful components. A feature of this technology is the use of the existing production infrastructure for the extraction of associated useful components in existing uranium mines, without significant capital investments in production infrastructure and mining operations. The technology of underground borehole leaching has been reliably developed in uranium deposits for decades. The fundamental similarity of the technology for the extraction of uranium and a number of associated useful components (APC) - by the ISR method, allows the use of spent ore fields of uranium deposits for the extraction of PPC. The use of ready-made technological infrastructure (wells, pipeline network, pumping equipment, control units, etc.) allows, due to savings on infrastructure costs, to obtain profitability when mining ore-bearing blocks with a content of recoverable components from ≤ 1 g/t, up to 0.1 g/t. Taking into account the indirect savings of significant costs for the reclamation of spent blocks, it will be profitable to mine blocks with a content of recoverable components up to 0.01 g/t. In view of the foregoing, this technology has a good prospect for implementation in production.

Keywords: uranium mines, associated valuable metals, associated useful components, underground well leaching, productive solution, chlorine-containing solutions.

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Introduction

In industrial practice, in the hydrometallurgical method of gold mining, sodium cyanide solution is used as the main reagent.

Taking into account the global trend of the policy of strengthening the environmental friendliness of production, studies on the replacement of sodium cyanide with less toxic reagents for dissolving gold are relevant. One of the promising reagents for industrial use can be used solutions containing active chlorine, in the form of free halogen or in a compound in the form of hypochlorites. In this work, the studies were carried out on core samples obtained during exploratory drilling of a well at the Semizbay field.

Studies were carried out on the possibility of using the method of underground well leaching to extract associated useful components (gold, iron, aluminum, lanthanum, scandium, and others).

For gold leaching from underground horizons by the method of underground well leaching, the use of cyanides is practically excluded due to high environmental risks. As an alternative, among the leaching agents, chlorine-, iodine-, and bromine-containing reagents can be used, which are more environmentally friendly than cyanide reagents. In the case of using the method of underground well leaching, capital investments can be reduced by dozens of times, so the process of underground leaching can be economically efficient with a higher consumption of chlorine, with a duration of mining

of ore deposits of several months, as well as with the development of poor off-balance ores [1].

In [2], gold leaching was carried out without cyanides, using hydrochloride technology, which makes it possible to ensure the environmental safety of the work. As a solvent for the ores of the Ural deposits, potassium hypochlorite, sodium and chlorine are used.

When gold is leached [3] from crushed ore to a particle size of -12 + 0 mm using the addition of sodium acetate, it improves the kinetics of gold dissolution and increases gold recovery by ~ 4%.

In [4], sodium hypochlorite was used as a leaching agent in order to selectively extract gold and silver from copper concentrate. Hypochlorite leaching of copper concentrate was carried out with preliminary water oxidation under pressure. With direct hypochlorite leaching, the recovery of silver and gold was 45.0% and 42.7%. Using pressurized aqueous oxidation followed by hypochlorite leaching, a selective recovery of 92.5% silver and 90.0% gold from the copper concentrate was achieved.

The dissolution of gold using flat flakes and spheres in a chloride medium was studied in [5] by the method of obtaining chlorine. Based on thermodynamic and mass balance calculations performed in the study at 1.27×10-2 M Cl2 and 0.48 M Cl−, it was found that molecular chlorine (Cl2) is the most predominant species, followed by the Cl− ion at pH below 2.0. From pH 4.0 to 8.0 HClO and from pH 8.0 onwards, ClO− are reported to be more predominant.

In [6], tests on the leaching of a refractory mineral and tailings of a manganese-silver alloy with a low content of silver in manganese-silver minerals were carried out in two stages: extraction of manganese with sodium sulfite and sulfuric acid and leaching silver and gold with sodium hypochlorite and hydrochloric acid, yielding 97% silver recovery, over 80% gold recovery, and 98% manganese recovery. The concentration of hydrochloric acid is 200 g/l, and the concentration of sodium hypochlorite is 50 ml at a concentration of 3%. The tests were carried out at room temperature.

In [7], gold leaching was carried out with a solution of hydrochloric or sulfuric acid with the addition of sodium hypochlorite at a ratio of 0.1 N (HCl) and 0.1 N (NaClO). This solution is made immediately before injection into the ore.

In many sulfide refractory gold ores and concentrates, gold is often found as small inclusions in sulfide minerals, especially in pyrite [8].

In [9] proposed a hydroleaching method consisting of pre-treatment by pressure oxidation and chlorination for refractory gold concentrates with high sulfur content, and investigated the leaching by gold chlorination thermodynamically and experimentally.

It is advisable to maximize the concentration of chloride or reduce the concentration of $[AuCl4]^-$, taking into account the thermodynamics of the gold chlorination leaching reaction. The rational thermodynamic conditions for gold leaching are as follows: pH 3.5–7.8, redox potential over 0.9 V, chloride concentration over 1 mol/l, 10^{-5}–10^{-4} mol/l $AuCl4]^-$ concentration. The redox potential must be maintained above 1.0 V for 2 hours to obtain a high percentage leaching in the chlorination of the gold concentrate pre-treated by pressure oxidation.

If the redox drops below 1.0 V during leaching, the dissolved gold chloride precipitates again and the percentage of leached gold falls. The optimum conditions for chlorination are: pH 4, redox potential above 1.0 V, NaCl concentrations 75 g/l, reaction temperature 40 °C, liquid-solid ratio 3:1, and leaching time 2 hours. The percentage of gold leaching reaches 96.54%.

In studies on the processing of refractory ores and concentrates, the method of exposure to chlorine-containing solutions to extract gold is becoming more and more widely used [[10], [11], [12], [13], [14], [15], [16]].

In [17], the RSM-CCD statistical method was used to determine the optimal conditions for the experiment on leaching a gold ore sample with hypochlorite. SEM/EDX analysis showed that the gold particles are bonded to sulfide and silicate minerals. Moreover, the XRD spectrum showed that the main phases are silicate and muscovite. Elemental analysis by XRF confirmed the presence of Si, Al, Fe and K as major elements in the gold ore sample.

ANOVA analysis was applied to the results of leaching experiments, which showed that the amount of gold dissolved in the form of the gold chloride complex [AuCl4]- was highly dependent on the pH of the solution, followed by the concentration of calcium hypochlorite [Ca(OCl)2] and sodium chloride (NaCl) to facilitate the formation of [AuCl4]- ions. Optimization of leaching experiments at various pH values from 4 to 6,

Ca(OCl)2 molar amounts from 0.5 to 1.5 and NaCl mole amounts from 2.5 to 3.5 showed that the best theoretical conditions for hypochlorite leaching of gold-bearing ores the sample had a lower pH of 4.05 with NaCl and Ca(OCl)2 concentrations of 2.93 M at 1.08 M, respectively.

Verification experiments with optimized conditions showed that gold recovery rates were obtained from 78.99 to 82.46%. Characterization of the obtained residue from the verification experiment by XRD showed that quartz is the main phase. In addition, XRF analysis of the residue showed the presence of a high mass percentage of Ca and Fe compared with the original sample of gold ore.

In [18], the extraction of gold and silver was studied at a sodium chloride concentration in the range of 0.5–3 m, which increased the stability of gold chloride. However, within this range, more than 50% of the silver still remains in the solid AgCl form. Leaching of gold and silver using stirred reactors and static tests confirmed that the maximum recovery of gold and silver is controlled by the concentration of sodium chloride. However, recovery of gold and silver has reached 80% and 50% with reactor leaching.

If chlorinated sea water was used for leaching (approximately 0.5 M NaCl and pH 5.5 and Eh less than 1.00 V compared to the Ag–AgCl reference), both gold and silver could not be fully recovered due to the formation of hydroxide gold and silver chloride. It is best to keep the pH below 4 and keep the solution potential Eh above 1.00 V to keep the gold in solution. Static tests simulating tailings leaching show lower recoveries of both gold and silver reaching 70% and 30% respectively.

In [19], the studied leaching parameters were the S/L ratio, the type of oxidant, i.e. [Cu2+]/[Fe3+] and [Cl−]. The results showed that gold could be dissolved under exceptionally mild conditions when an appropriate adsorption/reduction site (activated carbon) was provided immediately after leaching. It has been found that the impurity metals iron and copper originating from gold ore (Fe 1.6% and Cu 0.05%) are the preferred self-initiating oxidants and 87% gold can be dissolved in pure calcium chloride solution (2.8 M).

The authors of [20] studied the oxidation of sulfide minerals and the leaching of gold from a gold-bearing sulfide concentrate using a chloride–hypochlorite solution. The effect of calcium hypochlorite concentration, sodium chloride concentration and initial pH of the leaching agent on changes in pH and Eh of the slurry was investigated. Then, taking into account the stability range of the gold complex (Eh ~ 1000 mV) and the formation of gaseous chlorine (pH<3.5), the optimal leaching parameters were determined.

Optimum conditions were obtained at 200 g/dm3 calcium hypochlorite, 200 g/dm3 sodium chloride and an initial pH of 11 (with a concentrate of 200 g/dm3, agitation speed of 600 rpm and a temperature of 25°C), at which about 82% gold.

In [21], the effect of various leaching conditions on the extraction of gold from sulfur-containing gold ore using an acidic sodium chlorate solution was studied. The content of gold and sulfur in the ore was 55.7 g/t and 11.67 wt. % respectively. The presence of sulfur has been found to significantly hinder gold recovery.

The optimum desulfurization temperature, desulfurization time, leaching temperature, leaching time, NaClO3 addition rate, HCl concentration, NaCl mass ratio to sample, stirring speed, and liquid leachate volume to solid sample weight ratio were 650°C, 2 h, 40°C, 45 min, 0.25 ml/min, 3M HCl, 0.2, 250 rpm and 10, respectively. The percent gold recovery and percent weight loss of the sample were 97% and 8.8%, respectively, under these optimum conditions. The leaching of gold by this method was quite fast and efficient, and the temperature was low. These characteristics make it possible to apply this method in continuous operation in industry.

Experimental part

Analyzing various literary sources based on the results of experimental studies of the leaching process of gold and other associated useful components, a leaching solution with the following contents of reagents was selected as the most optimal:

NaClO + HCl(c) (hydrochloric acid with sodium hypochlorite), pH=2.8-3.2.

For experimental work on leaching, samples of the core material of the Semizbai deposit were taken from three intervals - ore, supraore, and subore.

The mother liquors obtained as a result of agitation leaching were analyzed by the atomic absorption method to determine the concentrations of various elements (Table 1).

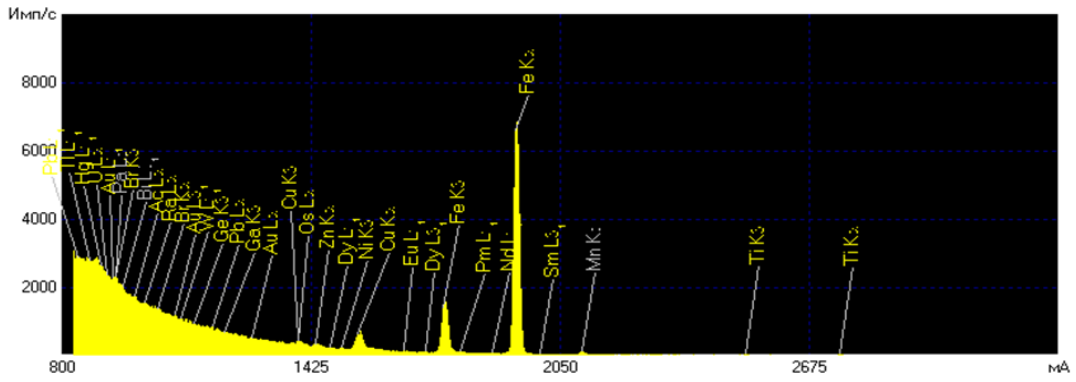
Leaching of gold (Au) from their technological samples. Works to determine the concentration of gold in technological samples were carried out by the laboratory "Technologies for the hydrocarbon and mining and metallurgical sectors and related service industries" of JSC "IMiO" in Almaty. Analyzes in solutions were carried out on an atomic absorption spectrometer. To determine the gold content in the combined samples of the core material, the assay method of analysis was used. The results of the analyzes are presented in Tables 2 and 3.

Table 1 - The concentration of related and useful components in technological solutions

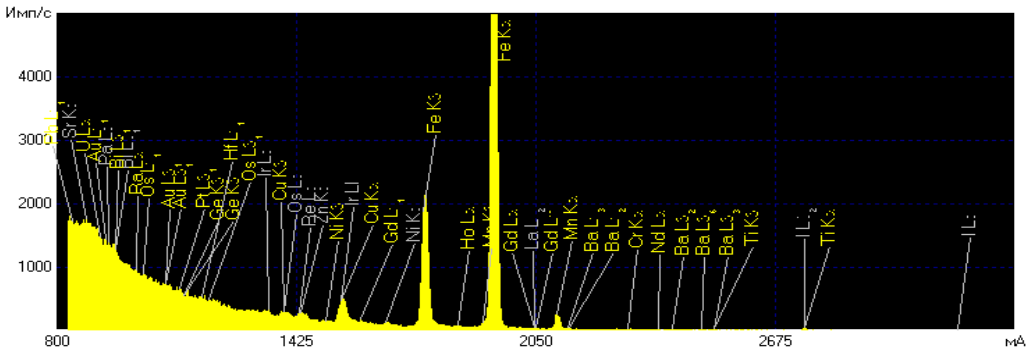
№	№ samples	Fe general, mg/dm³	Al, mg/dm³	La, mg/dm³	Sc, mg/dm³	Leaching solution concentration, %
1	P-4-1(H)	0.63	4.15	0.0053	0.00085	5
2	P-4-2(H)	22.96	15.09	0.0010	0.00027	
3	P-4-3(H)	0.56	10.28	0.014	0.0041	
4	P-5-1(H)	4.08	15.91	0.013	0.0048	8
5	P-5-2(H)	99.96	89.02	0.0043	0.056	
6	P-5-3(H)	2.10	28.13	0.087	0.0023	
7	P-6-1(H)	0.48	0.37	0.0088	0.00062	16
8	P-6-2(H)	136.03	14.43	0.26	0.0053	
9	P-6-3(H)	34.24	12.90	0.023	0.0087	

Table 2- Results of chemical analysis of pooled core samples using the assay method for gold

№	№ samples	Au g/l	Interval
1	Well 1	0.20	Suprarutal interval
2	Well 2	0.34	Ore interval
3	Well 3	0.34	Under-ore interval



Picture 1– Results of X-ray phase analysis of the core sample

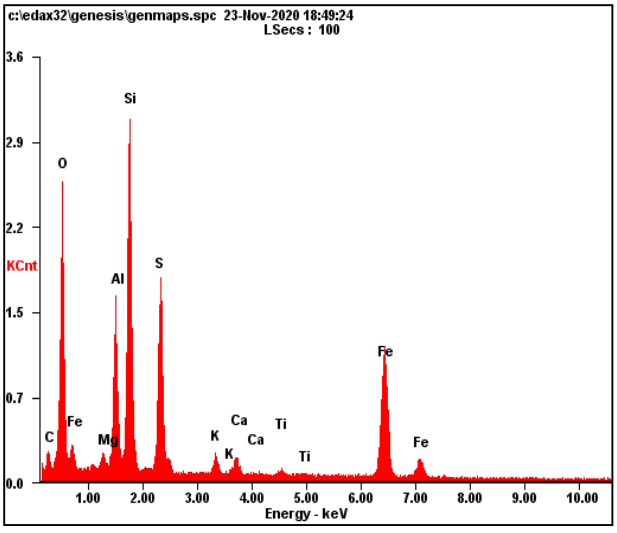


Picture 2 – Results of X-ray phase analysis of the core sample

The method of X-ray phase analysis was used to determine the chemical composition of the samples. The data of X-ray phase analysis are presented in Figures 1 and 2.

To study the local elemental composition of core samples, chemical analysis was carried out using a scanning electron microscope. Elemental analysis of core samples was carried out by energy-dispersive analysis on a Fei Quanta 3d 200i scanning electron microscope.

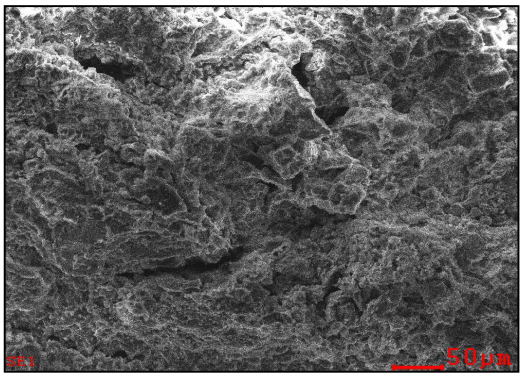
The resolution of the microscope is up to 1 nm at a voltage of 30 kV, the accelerating voltage is from 0.5 to 30 kV, the magnification is from x 10 to x 1,000,000, the beam current is up to 200 nA, the elemental analysis was carried out in vacuum for a number of elements C, O , Na, Mg, Al, Si, S, Cl, U, Ca, K and Fe. The characteristic X-ray spectrum for a number of elements and the general view of the micrograph of the core sample under study with the detected area are shown in Figure 3.



a) sample spectrum (first point)

Element	Wt%	At%
CK	17.44	27.23
OK	42.91	50.32
MgK	0.78	0.60
AlK	6.43	4.47
SiK	13.59	9.08
SK	6.95	4.07
KK	0.78	0.37
CaK	0.76	0.36
TiK	0.27	0.11
FeK	10.07	3.38
Matrix	Correction	ZAF

b) Elemental analysis of the obtained spectra in weight percent Wt %



c) micrograph 50 µm

Figure 3 - Characteristic X-ray spectrum

Mineralogical analysis of core samples for gold. To study the material composition of the core sample of the ore interval, a polished artificial briquette (size 0.25 mm) was made.

The sample material was studied under a microscope of the LEICA DM 2500 P brand in reflected light in order to diagnose and describe ore

minerals, in immersion preparations for the diagnosis of rock-forming minerals.

The result of the mineralogical analysis is shown in Figure 4.

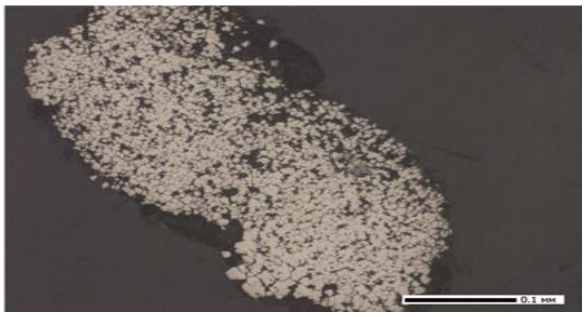


Figure 4 - Accumulation of globular pyrite.
Sample 1 (ore interval)
Polished briquette, led away. x 200

Table 3 - Comparison of the degree of recovery of recoverability of gold from ore samples at various concentrations

№	№ samples	Au mg/dm³	Leaching solution concentration, %
1	Well 1	0.033	5 (Surface interval)
2	Well 2	0.0171	8 (ore interval)
3	Well 3	0.057	16 (Under-ore interval)

More clearly, the degree of extraction of Au is shown in Figure 5.

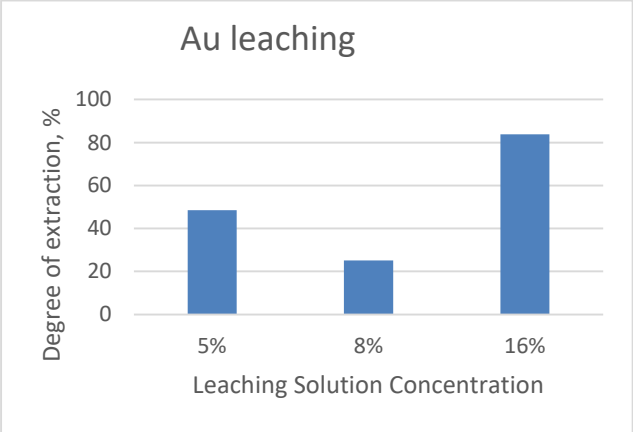


Figure 5 - The degree of extraction of Au during agitation leaching with NaClO solution with the introduction of oxidizing agents at various concentrations

The discussion of the results

Analyzing the output of associated and useful components in technological solutions (Table 1), it can be noted that the highest output of components in the solution was achieved when core material was leached from the uranium-bearing ore interval. Thus, the concentration of iron was 136.03 mg/dm^3, the concentration of aluminum - 89.02 mg/dm^3, scandium - 0.056 mg/dm^3, lanthanum - 0.26 mg/dm^3. X-ray phase analysis of the core sample (Figure 1 and Figure 2) in the range from 800 to 2050 nm in the decomposition spectrum shows fluctuations in the peaks of uranium (U), iron (Fe), copper (Cu), manganese (Mn), zinc (Zn). In addition to the above, the decomposition spectrum contains fluctuations of rare earth elements, such as osmium (Os), rhenium (Re), neodymium (Nd), actinium (Ac), scandium (Sc), cesium (Ce), as well as bismuth (Bi), etc. Note that the decomposition spectra show spectral lines of mercury (Hg) and unexpressed fluctuations of gold-Au.

The presence of mercury is interpreted by the content of mercury in sulfide minerals that are genetically related in chemical nature, which in turn are formed during the formation of uranium ores. During the amalgamation reaction, mercury forms compounds with gold. For this reason, the detection of lithophaneous reflections of gold and clear spectra of mercury indicate the possible presence of gold.

The characteristic X-ray spectrum (Figure 3) shows that the elements Si, O, S, Fe and Al are the main components of the sample, in which these elements have the following percentages: silicon (Si) - 13.59%, oxygen (O) - 42.91%, sulfur (S) - 6.95%, Iron (Fe) - 10.07% and aluminum (Al) - 6.43%.

Mineralogical analysis of a briquette of a core sample of the ore interval (Figure 4) shows the presence of pyrite, sphalerite and technogenic material in a small amount, resembling gold in color. Pyrite is found in the form of fine dissemination in non-metallic material and several free grains up to 0.3 mm in size. After testing the effect of HNO_3 acid on a grain resembling gold, it dissolved, thus its technogenic nature was established.

The result obtained from the data in Figure 5 characterizes a NaClO + HCl + H_2O solution with a concentration of 16% as more effective for Au than a solution with a concentration of 8% and 5%. The degree of extraction of Au for solutions with 16% leaching reagents reached 83.82%.

Conclusions

Based on the results of laboratory experiments on agitation leaching on samples of core material of ores from the Semizbay deposit of the supra-ore, ore and under-ore interval, it can be concluded that for the extraction of associated useful components by the ISR method from depleted uranium wells, a chlorine-containing solution of the composition $NaClO + HCl_{(c)} + H_2O$ (hydrochloric acid with sodium hypochlorite) at pH=2.8-3.2.

The highest extraction of gold (83.82%) into solution was observed during leaching of material from the sub-ore interval, while for associated useful components the highest degree of extraction was observed during leaching of material from the ore interval: iron - 136.03 mg/dm3, aluminum - 89.02 mg/dm3, scandium - 0.056 mg/dm3, lanthanum - 0.26 mg/dm3.

At the moment, the sodium hypochlorite leaching method together with hydrochloric acid is not used for gold mining on an industrial scale. In this work, the experiments were carried out directly on the ore material, which makes it possible to test this method in industry and the result was achieved, which makes it possible to apply this method on an industrial scale.

Thus, the use of the ISL method on spent ore uranium blocks for the extraction of valuable components is a breakthrough direction in research in the mining industry.

Conflicts of interest. On behalf of all authors, the author declares that there is no conflict of interest.

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Уран кен орындарының пайдаланылған блоктарын өңдеу кезінде құрамында хлор бар агенттерді пайдалану

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ТҮЙІНДЕМЕ

Жұмыс ілеспе пайдалы компоненттерді алу мақсатында жерасты ұңғымалық сілтісіздендіру әдісімен уран кен орындарының пайдаланылған блоктарын химиялық ерітінділермен қосымша өңдеу есебінен табиғи ураны өндіру және қайта өңдеу бойынша жұмыс істеп тұрған кеніштерді әртараптандыруға бағытталған. Бұл технологияның ерекшелігі (жүргізіліп жатқан жұмыс) қолданыстағы уран кеніштерінде ілеспе пайдалы компоненттерді өндіру үшін қолданыстағы өндірістік инфрақұрылымды өндірістік инфрақұрылым мен тау-кен жұмыстарына елеулі күрделі салымдарсыз пайдалану болып табылады. Жерасты ұңғымаларын шаймалау технологиясы уран кен орындарында ондаған жылдар бойы сенімді түрде пысықталды. Уран өндіру технологиясының және бірқатар ілеспе пайдалы компоненттердің түбегейлі ұқсастығы - жерасты ұңғымалық сілтісіздендіру әдісімен пайдалы компоненттерді өндіру үшін уран кен орындарының пайдаланылған кен өрістерін пайдалануға мүмкіндік береді. Дайын технологиялық инфрақұрылымды (ұңғымалар, құбырлар желісі, сорғы жабдықтары, басқару блоктары және т. б.) пайдалану инфрақұрылымдық шығындарды үнемдеу есебінен өндірілетін компоненттері ≤ 1 г/т-дан 0,1 г/т-ға дейінгі кенді блоктарды өңдеу кезінде рентабельділік алуға мүмкіндік береді. Пайдаланылған блоктарды қалпына келтіруге кететін айтарлықтай шығындарды жанама үнемдеуді ескере отырып, алынатын компоненттердің құрамы 0,01 г/Т дейін болатын блоктар тиімді жұмыс істейді, жоғарыда айтылғандарды ескере отырып, бұл технология өндіріске енгізу үшін жақсы перспективаға ие.

Түйін сөздер: уран кеніштері, ілеспе бағалы металдар, ілеспе пайдалы компоненттер, жер асты ұңғылап шаймалау, өнімді ерітінді, хлорлы ерітінділер.

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Использование хлорсодержащих агентов при обработке отработанных блоков урановых месторождений

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АННОТАЦИЯ

Работа нацелена на диверсификацию действующих рудников по добыче и переработки природного урана за счет дополнительной обработки отработанных блоков урановых месторождений химическими растворами методом подземного скважинного выщелачивания (ПСВ) с целью извлечения попутных полезных компонентов. Особенность (проводимой работы) этой технологии состоит в использовании существующей производственной инфраструктуры для добычи попутных полезных компонентов на действующих урановых рудниках, без значительных капитальных вложений в производственную инфраструктуру и горные работы. Технология подземного скважинного выщелачивания за десятки лет надежно отработана на урановых месторождениях. Принципиальная схожесть технологии добычи урана и ряда попутных полезных компонентов (ППК) - методом ПСВ, позволяет использовать отработанные рудные поля урановых месторождений для добычи ППК. Использование уже готовой технологической инфраструктуры (скважины, сеть трубопроводов, насосное оборудование, управляющие блоки и т.д.) позволяет за счет экономии на инфраструктурных затратах, получить рентабельность при отработке рудоносных блоков с содержанием извлекаемых компонентов от от ≤ 1 г/т, вплоть до 0,1 г/т. При учете косвенной экономии значительных затрат на рекультивацию отработанных блоков, то рентабельно будет отрабатывать блоки с содержанием извлекаемых компонентов вплоть до 0,01 г/т. С учетом вышесказанного данная технология имеет хорошую песпективу для внедрения в производство.

Ключевые слова: урановые рудники, попутные ценные металлы, попутные полезные компоненты, подземное скважинное выщелачивание, хлорсодержащие растворы.

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