

# EXPERIENCE AND ISSUES OF REPROCESSING LIQUID RADIOACTIVE WASTE OF COMPLEX CHEMICAL COMPOSITION AT FSUE “MCC”

Gavrilov P. M., Merkulov I. A., Matselya V. I., Druz D. V.

FSUE “Mining and Chemical Combine”, Zheleznogorsk, Krasnoyarsk Region, Russia

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*The article overviews FSUE “MCC” experience in managing liquid radioactive waste generated as the result of uranium-graphite reactor fuel reprocessing .*

**Keywords:** *liquid radioactive waste, radioactive solution processing, LRW underground injection, acceptance criteria, alkaline concentration, TBP, hexachlorobutadiene.*

## Introduction

Mining Chemical Combine being an enterprise run by the Ministry of Secondary Engineering, was established with the aim of producing and separating weapons-grade plutonium. From the early 1960's, the key units involved in this process until 2013 were the reactor (RP), radiochemical and isotope-chemical plants of the enterprise. Development of production facilities and changes in environmental regulations prompted the improvement and upgrading of relevant technological processes and equipment. Thus, at the first stage, until 1967, all liquid radioactive waste (LRW) generated during radiochemical reprocessing of nuclear fuel irradiated in RP reactors were held in special storage tanks with a capacity of some 3,000 m<sup>3</sup>. This was due to the unavailability of LRW underground injection facility (Severnii site). Further on, following the Severnii site commissioning, requirements on LRW solutions injected into its disposal horizons were established. Comprehensive reconstruction of the entire technological flow chart at the radiochemical

plant in the late 1970's when precipitation processes were replaced by extraction ones, fundamentally upgraded the production method involving separation and purification of valuable components providing for an enhanced level of safety. It also enabled to increase uranium and plutonium recovery from LRW prior to its disposal.

## Sources of LRW generation

According to Article 3 of the Federal Law of November 21, 1995 № 170-FZ On Atomic Energy Use, radioactive waste involves in particular materials and substances considered as not being subject to any future use. Operation of reactor and radiochemical plants results in the generation of different LRW types that can be divided into (1) process discharges generated directly as the result of a technological process and (2) non-process waste discharges generated due to some supplementary operations. This article considers the management

of process LRW only. Figure 1 shows a schematic flow chart presenting the management of process LRW generated by the enterprise.

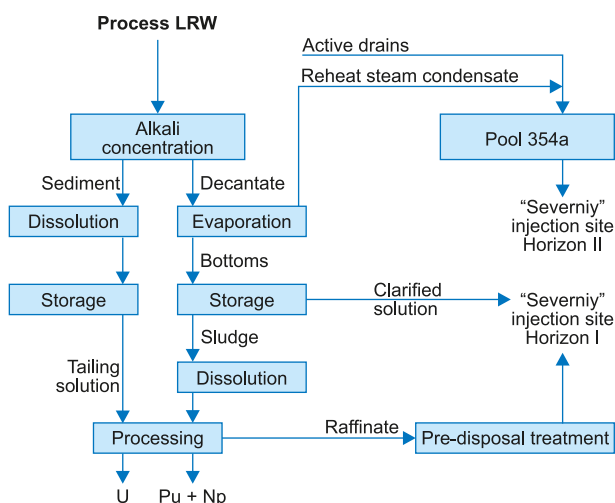


Figure 1. Schematic flow chart presenting the management of process LRW

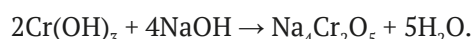
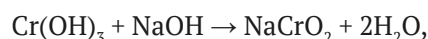
## Process LRW

Liquid process waste generated from the reprocessing of standard uranium lumps (ISUL) irradiated in production reactors were formed from a big number of products with the radiochemical production ones considered as the primary generation source. The first LRW management stage involved alkali concentration of target components, such as uranium, plutonium and neptunium, with further evaporation of generated alkali decantate and solution of the resulting sediments by nitric acid to finalize the extraction process. The following liquid waste streams were handed over for uranium, plutonium and neptunium alkali concentration:

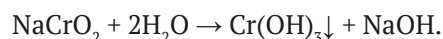
- oxalate desorbates and rinse water from sorption unit used to treat nitric acid solutions from dissolution of ISULs;
- raffinate from extraction cycles, washing solutions from extractant washing;
- sorbates and washing solutions from plutonium and neptunium sorption refining, oxalate filtrates and decantates from precipitation of plutonium and neptunium oxalic acid;
- washing nitric acid solutions from equipment and sludge washing, solutions from pulp processing;
- drainage solutions, condensate from gas purification equipment.

Alkali sedimentation of plutonium and neptunium in presence of accessory heavy metals Fe, Cr, Ni, Mn results in sparingly soluble hydroxides or basic salts  $\text{Pu}(\text{OH})_4$ ;  $\text{Pu}(\text{OH})_{4-x}\cdot\text{H}_2\text{O}$ . Decrease in the solubility of plutonium compounds in alkaline

medium is prompted by a chemical reaction with atmospheric oxygen and plutonium present in the solid phase: the former one oxidizes to a hexavalent state subsequently forming mixed crystals with sodium uranium salts during recrystallization. Due to this process, in the presence of uranium salts, solubility of plutonium compounds in an alkaline medium seems to be very low amounting to some tens of micrograms per liter of solution. Hydroxides and salts of heavy metals precipitating in an alkaline medium are considered as good sorbents for the micro amounts of plutonium and neptunium and for the majority of fission products. Therefore, along with concentration of uranium due to coprecipitation, alkali sedimentation also involves concentration of plutonium, neptunium and hydrolysable radionuclides. The extent to which valuable components and fission products are concentrated during alkali sedimentation amounts to 30–40. Concentration of valuable components and fission products was performed via alkali redeposition method at a temperature of  $(90 \pm 5)^\circ\text{C}$  with constant stirring of the reaction volume with bubbler air. Alkali redeposition method enables to generate dense and fast-settling precipitate. Compact precipitates during reverse deposition process are formed due to a small amount of heavy metals: Fe, Cr, Ni, U contained in the initial solution are fed into the sodium hydroxide solution per unit time. Thus, a small number of crystallization centers is formed with large individual units growing in their size. Reduced supersaturation favors the formation of large particles of sodium diuranate. During reverse deposition, polymerization of hydroxide molecules also contributes to formation of crystalline hydroxide precipitates that accelerates with the increase in the alkalinity of the solution. One should note the differences in the behavior of chromium during direct and reverse deposition. Direct deposition results in the formation of an amorphous precipitate of chromium hydroxide being prone to long-term aging. In case of reverse deposition with chromium salts being discharged into hot concentrated alkali, chromium hydroxide precipitates immediately dissolve with chromic iron generated following the reactions given below:



Further on, with decreasing alkalinity of the medium, hydrolysis of chromic iron occurs resulting in chromium hydroxide formation:



For this reason, during reverse deposition, chromium practically precipitates at high supersaturation

rates resulting in the formation of amorphous precipitates. Moreover, if diuranates and iron hydroxide precipitate simultaneously, chromium hydroxide precipitates later than these forming a separate self-sufficient precipitate.

Under the reverse alkaline deposition, compact precipitates are commonly formed by iron. However, under increased iron concentration, not crystalline but amorphous precipitates are formed, thus, pulp compaction is not observed. Presence of manganese (II) salts admixtures in the initial solution do not affect the sludge compaction rate, since manganese (II) hydroxide precipitates. However, upon the contact with air oxygen supplied for mixing, manganese (II) oxidizes to manganese (IV) forming the following compound:  $MnO_2 \cdot nH_2O$ . Manganese dioxide is an amorphous substance having a developed surface that captures the suspended matter, settles on the walls and fixtures of the device and forms colloids. Therefore, the sedimentation of solutions with a high concentration of manganese occurs very slow. To increase the settling rate for these solutions, the concentration of uranium in the initial solution had to be increased.

The reprocessed uranium lumps involved an aluminum shell. Thus, a certain amount of aluminum was contained in the raffinates from the first extraction cycle. During alkaline precipitation with an excess of NaOH, a significant part of the aluminum remained in the decantate in the form of sodium aluminate. Sedimentation of aluminum hydroxide resulted in unconsolidated loose precipitate. To allow more ample transfer of aluminum into decantate, the precipitate was treated with a hot solution of sodium hydroxide (alkaline decompression operation).

Precipitates of heavy metal hydroxides Fe, Cr, Ni and sodium diuranate formed during alkaline deposition are characterized with high adsorption capacity and capture individual fission-product elements from the solution. Capture capacity depends on the pH of the medium, salt concentration in the solution and the nature of the fission-product element. For cesium, its sorption capacity on the hydroxide precipitate is low. Therefore, during alkaline concentration, cesium almost entirely remains in the solution. Sorption capacity for strontium depends on the medium alkalinity (increased alkalinity of the medium results in more intense sorption of strontium by hydroxide precipitates) accounting for at least 95%.

Zirconium, due to its chemical properties, coprecipitates well in an alkaline environment with precipitates of heavy metal hydroxides. In the range of pH = 7–9, niobium is also quantitatively captured by hydroxide precipitates. However, an increase

in the alkalinity of the solution causes formation of soluble niobate and release of niobium into decantate.

Rare-earth elements ( $^{141}Ce$ ,  $^{144}Ce$ ,  $^{147}Pm$ ,  $^{144}Pr$ , etc.) accounted for a significant fraction of radionuclides in the solutions supplied to alkaline deposition with their sorption capacity on hydroxide precipitates differing significantly. However, at pH=9.5 and the temperature of the medium being higher than 90 °C, rare-earth elements are almost completely captured by precipitates of chromium hydroxides, iron and sodium diuranate. To increase the deposition rate during the processing of solutions with a low content of valuable components and impurities, an additional dosage of uranium solution was carried out.

Concentration of tributyl phosphate (TBP), hexachlorobutadiene (HCBd) and their decomposition products in aqueous solutions fed to alkaline deposition was determined based on their solubility rate. However, the content of some organic products was found to be exceeding the solubility limit due to microemulsions or mechanical entrainment of the organic phase upon deviations from process flow chart parameters. Presence of an excess amount of the organic phase somewhat complicated the alkaline concentration process. During deposition, slight foaming and a decrease in the sedimentation rate due to the “hanging” of extractant droplets along the height of the installation was observed. The amount of hydroxide precipitate in the presence of 1% (vol) extractant increased by an average of 1.2 times. Due to the interaction of plutonium with dibutyl phosphoric acid (DBPA) and formation of stable emulsions in the presence of hydroxide precipitates, the concentration of TBP in alkaline decantate reached 100 mg/l, the concentration of plutonium in decantate amounted to 0.2 mg/l. Presence of extractant in the initial solutions did not affect uranium content in the decantate.

Following the sedimentation of solutions saturated with the extractant, more than 90% of TBP remained in the decantate. Whereas, sedimentation of solutions containing mechanically entrained extractant having a concentration of 0.2–1% (vol), 60–90% of TBP was captured by the precipitate. HCBd content in the decantate depended on the content of the extractant in the initial solution. If the content of TBP in the initial solution did not exceed the solubility limit, HCBd was basically removed with decantate. With an increase in the amount of extractant content in the initial solution to 1% (vol), HCBd content in the decantate decreased to 70%. Under alkaline precipitation, monobutylphosphoric acid (MBPhA) could be released into decantate by 80–95%. Under alkaline

concentration at 95–100 °C, hexachlorobutadiene could be distilled off by 90–95 % with a vapor-gas phase.

Processing of solutions containing extractant within the solubility limit did not cause any difficulties. In this case, the sediment volume and the settling time did not increase: TBP could be almost completely removed to the decant with no separate phase formed upon precipitate dissolution. The presence of significant amounts of HCBD resulted in an increased content of plutonium in alkaline decantate.

A technological method was used to increase the coalescence rate for organic microemulsions: alkaline concentration operation was preceded by hydrogen peroxide injection into the initial solutions. Along with other technological methods applied to reduce TBP and HCBD concentrations, the initial solutions were heated to 85–95 °C with subsequent minimum 3-hour long exposure under stirring. In this case, HCBD was almost completely distilled off, whereas TBP — by about 60 %.

Among other fission products, ruthenium ( $^{105}\text{Ru}$ ,  $^{106}\text{Ru}$ ) is considered of a particular importance as it's the only element subject to all possible oxidation states and may resemble to the metals of all groups. Ruthenium capture by precipitating iron, chromium and sodium diuranate hydroxides is highly dependent on the redox potential, pH of the medium and temperature. Usually the amount of ruthenium captured by precipitation of metal hydroxides varies from 10 to 100 % of its initial content in the solution. Most optimal conditions that can be provided by the technological process to enable most complete capturing of ruthenium are as follows: precipitation of iron, chromium and sodium diuranate hydroxides at a temperature of about 100 °C,  $\text{pH} < 9.5\text{--}10$  with no excess amounts of oxidizing agents.

ISUL processing with short exposure time resulted in some small amounts of radioactive iodine present during alkaline concentration operation. Slightly improved sorption of the radioiodine was attained by decreasing the pH of the medium, bringing it up to 10 % at best compared to its content in the initial solution.

Evaporation of alkaline decantates was carried out to reduce the volume of liquid ILW subject to underground disposal up to four times, while the concentration of radioisotopes and sodium nitrate in decantates increased. To avoid contamination of the secondary steam condensate, it was extremely important to maintain the technological evaporation modes. Decontamination factor ( $K_{dec}$ ) for the secondary steam condensate was significantly reduced due to the following factors: drop

entrainment, foaming, as well as volatility of radionuclides. Entrainment amount mainly depended on the rate of secondary steam removal from the boiling zone and the size of the drops. The decrease in the rate of secondary steam removal was ensured by equipment designs — the evaporator was equipped with a separator. However, entrainment could be increased significantly due to a dramatic increase in the vaporization rate. Such an increase is associated with exhausting processes occurring in the evaporator when a solution with a temperature being above the boiling point is poured into the separator or due to high boiling rate of the solution, which was not allowed, in particular, due to the automation means applied.

Foaming caused by the presence of organic substances, suspensions or dissolved gases can significantly reduce  $K_{dec}$  by increasing the level of liquid in the evaporator due to reduced volume of separation and formation of stable bubbles carried along with the vapor stream. The key method proposed to avoid foaming was to reduce the content of extractant in the solutions subject to processing. This was achieved by a sufficient number of technological and preventive organizational measures. The volatility of iodine, ruthenium, iodine organic compounds present in solutions during evaporation could potentially increase the release of radionuclides into the environment. Dramatic decrease in the volatility of ruthenium and iodine was ensured by maintaining the redox potential of the medium in the evaporator. During evaporation of alkaline decantates containing trace amounts of extractant, TBP and HCBD were completely distilled off with a vapor-gas phase. At the same time, formation of amorphous easily settling precipitates was observed in the bottom residues (in the volume of 0.15–0.20 % as compared to the volume of the evaporated solution). These basically accounted for silicon dioxide, aluminum compounds, as well as compounds of evaporation equipment corrosion products with HCBD and MBPhA.

Alkaline precipitates were transferred to the solution by means of their dissolution in nitric acid. Hexavalent uranium, plutonium and neptunium formed relevant compounds:  $\text{UO}_2(\text{NO}_3)_2$ ,  $\text{PuO}_2(\text{NO}_3)_2$ ,  $\text{NpO}_2(\text{NO}_3)_2$ , tetravalent plutonium —  $\text{Pu}(\text{NO}_3)_4$ , quinquivalent neptunium —  $\text{NpO}_2\text{NO}_3$  respectively. Most of the elements formed nitrate salts, while iron, nickel, chromium, manganese (IV) formed metal oxides, and silicon formed silicic acid gel used as a basis for polymer complex compound formation. Nitrate solution resulting from the dissolution of precipitate was sent for preliminary storage followed by subsequent extraction processing.

Dissolution of the precipitate containing TBP and HCBD in nitric acid, phase separation occurred with the bulk part of the extractant collected at the bottom of the installation and partially on the walls (drops). Up to 50% of uranium and plutonium was captured by the extractant, which caused some difficulties associated with subsequent processing and storage of the obtained concentrates. The fact that under ionizing radiation exposure HCBD undergoes radiation-chemical oxidation releasing atomic chlorine and hydrochloric acid had to be accounted for in the designs of the process equipment, thus, requiring the application of a number of technological methods and limitations in order to avoid equipment corrosion.

### Sludge processing

Hydroxide sludge is formed during the storage of evaporated alkaline solutions in storage tanks prior to their underground disposal. The sludge has a complex composition and mainly consists of iron, aluminum, chromium, nickel, calcium hydroxides, manganese oxide and silicic acid. During the operation of storage tanks, radionuclides are concentrated due to hydrolysis and sorption processes. The Mining and Chemical Combine is now addressing the challenge of emptying the storage tanks with a design capacity of up to 3,200 m<sup>3</sup> involving radioactive sludge retrieval and its processing. Sludge processing involves their staged dissolution in nitric acid and sodium hydroxide in presence of reducing and complexing agents. The main objective of such processing is seen to reduce the sludge volume, to extract the bulk amounts of uranium, plutonium, <sup>90</sup>Sr, <sup>144</sup>Ce, <sup>137</sup>Cs and to decontaminate the insoluble residues. The use of various reducing and complexing reagents upon heating ensures effective dissolution of partially dehydrated metal hydroxides and polymerized silicic acid. Clarified nitric acid solution resulting from sludge processing was sent to the extraction processing unit along with the tailing solutions.

**Table 1. Averaged composition of nitric acid solution from the processing of sludge contained in storage tanks (excluding uranium and plutonium content)**

Component	Unit	Value
Neptunium	mg/l	1–5
Americium-241	mg/l	0.020–0.035
Thorium	mg/l	2–5
Nitric acid	g/l	50–60
Specific beta-activity	Ci/l	0.7–1.5
Strontium-90	Ci/l	0.3–0.6

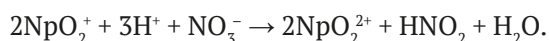
### Extraction processing of “tailing” solutions

Nitric acid solution from dissolution of alkaline precipitate was kept in tanks with a capacity of 300 m<sup>3</sup> before its further processing. After that, uranium, neptunium and plutonium were extracted from the solutions, and the resulting raffinates were brought into compliance with the acceptance criteria and sent for underground disposal. The processing flow chart involved the following stages:

- sorption purification of the initial solution to remove radioactive zirconium and niobium by filtration via a silica gel filter;
- extraction recovery of uranium, neptunium and plutonium;
- sedimentation of the first extraction cycle's raffinate to separate the organic phase, distillation of HCBD with water vapor;
- final treatment of the solution ensuring its compliance with the disposal criteria established by neutralizing nitric acid with sodium hydroxide and dilution;
- injection of the resulting conditioned product to LRW disposal wells.

Processing of tailings was usually carried out on a yearly basis. In doing so a number of activities was carried out to upgrade the flow chart and allow for additional pre-treatment of technological products. During the pre-treatment of products before the extraction processing, it was necessary to take into account the specific aspects associated with the behavior of uranium, plutonium and neptunium present in different valence forms. For example, the fact that the extractability of these elements in the hexavalent state decreases in the following range  $U > Np > Pu$ , in the tetravalent state it decreases in another order  $Pu > Np > U$ . Whereas, the extractability of uranium of different valences increases in the following range —  $U^{(IV)} < U^{(VI)}$ ; for plutonium —  $Pu^{(III)} \ll Pu^{(VI)} < Pu^{(IV)}$ ; for neptunium —  $Np^{(V)} \ll Np^{(IV)} < Np^{(VI)}$ . For this reason, conversion of plutonium and neptunium (in case of separate flows) to most extractable forms of  $Pu^{(IV)}$  and  $Np^{(VI)}$  is seen as the main task in adjusting the valence state of U, Pu and Np in an aqueous solution before the extraction occurs.

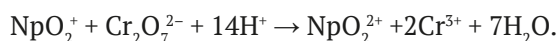
In nitric acid solutions, neptunium may be present in various valence forms:  $Np^{(IV)}$ ,  $Np^{(V)}$ ,  $Np^{(VI)}$ , the ratio between which depends on a number of factors. Neptunium was converted from the most stable pentavalent state to the most extractable form of  $Np^{(VI)}$  using sodium dichromate and nitric acid. Oxidation of  $Np^{(V)}$  with nitric acid in presence of trace amounts of nitrous acid formed due to nitric acid radiolysis, proceeds according to the following reaction:



Since the reaction is reversible, the process of converting  $\text{Np}^{(\text{V})}$  to  $\text{Np}^{(\text{VI})}$  was carried out in a two-phase system. In this case, due to  $\text{Np}^{(\text{VI})}$  transition into TBP phase, the reaction equilibrium shifts to the right side. To avoid a reverse reaction in the extraction and washing zones, it is necessary to maintain a concentration of nitric acid of at least 3 mol/l. In the case of a two-zone extract washing (strongly acid and weakly acid), neptunium accumulates in the aqueous phase in the weakly acid washing zone. As for single-zone washing at a concentration of nitric acid of at least 3 mol/l, neptunium accumulation in the first unit does not occur. Disproportionation reaction of pentavalent neptunium is also seen as a common one for nitric acid solutions:



Moreover, the degree of disproportionation and the reaction rate was found to be increasing with acidity growth. Sodium bichromate converts  $\text{Np}^{(\text{V})}$  to  $\text{Np}^{(\text{VI})}$  quickly and completely following the reaction presented below:



In this case, the shift of equilibrium towards  $\text{Np}^{(\text{VI})}$  formation was promoted by an increase in the acidity of the medium, as well as by the oxidation process in the two-phase system, due to  $\text{Np}^{(\text{VI})}$  transition into TBP phase. Sodium bichromate solution can be introduced into the initial aqueous solution. However, higher neptunium recovery rates were achieved by introducing the oxidizing agent directly into the extraction zone or into the strongly acidic washing zone. Nitric acid present in the aqueous solution was extracted using tributyl phosphate with the following compounds being formed:



The latter reaction occurs when the concentration of nitric acid in the aqueous phase exceeds 5 mol/l. As the concentration of nitric acid increases to 6 mol/l, the distribution coefficients for uranium, plutonium and neptunium increase due to an increase in the total concentration of nitrate ion. When the concentration of nitric acid in the aqueous phase reaches over 6 mol/l, the distribution coefficients for uranium, plutonium and neptunium decrease, since under these conditions the extractability of nitric acid increases. Application of the abovementioned chemical properties of the

**Table 2. Results of processing "tailing" solutions**

Evaluated component	Unit	Before extraction processing	After processing
Plutonium	mg/l	40–80	0.03
Uranium	g/l	35–70	0.01
Nitric acid	g/l	77–130	7.4
Neptunium	mg/l	10–20	0.7
Thorium	mg/l	7.5–16.0	1.5
Aluminum	g/l	0.05–1.40	0.02
Nickel (II)	g/l	1.35–3.00	0.136
Specific $\beta$ -activity	Bq/kg	$3.8 \cdot 10^{11}$ – $1.8 \cdot 10^{12}$	$1.9 \cdot 10^{10}$
Silicon dioxide	mg/l	Up to 250	40.9
Total phosphorus	mg/l	10–95	2.6
Iron (III)	g/l	1.5–3.0	0.16
Manganese (II)	g/l	0.6–1.4	0.07
Chrome (III)	g/l	0.005–0.340	0.04
Strontium-90	Bq/kg	$1.1 \cdot 10^{11}$ – $2.1 \cdot 10^{11}$	$5.3 \cdot 10^9$
Americium-241	mg/l	0.007–0.38	0.016
Tributyl phosphate	mg/l	$\leq 17$	9.7
Chloride ion	mg/l	115–200	17.1

extracted components, as well as the use of a number of technological methods during further operations, enabled to obtain solutions meeting the requirements established for the safe disposal.

## Conclusion

The paper briefly overviews the experience accumulated by FSUE MCC on managing solutions of complex chemical composition generated as the result of reprocessing ISULs from production uranium-graphite reactors. A number of challenging issues associated with the behavior of various chemical elements at the main technological stages of such reprocessing process were discussed. Based on statistical processing of the measurements, the main data on the compositions of both intermediate and final reprocessing products was provided.

Application of the discussed engineering methods at FSUE MCC to manage LRW with different activity levels, enabled to avoid the exceedance of maximum allowable limits for polluting components concentration in allowed sources of discharges into an open hydrographic network. These methods also ensured the compliance with waste acceptance criteria set for solutions subject to underground disposal.

### Information about authors

*Gavrilov Petr Mikhailovich*, Doctor of Engineering Science, Director General, Federal State Unitary Enterprise “Mining and Chemical Combine” (53, Lenin Str., Zheleznogorsk, Krasnoyarsk Region, 662972, Russia), e-mail: [atomlink@mcc.krasnoyarsk.su](mailto:atomlink@mcc.krasnoyarsk.su).

*Merculov Igor Aleksandrovich*, Chief Engineer of an enterprise, Federal State Unitary Enterprise “Mining and Chemical Combine” (53, Lenin Str., Zheleznogorsk, Krasnoyarsk Region, 662972, Russia), e-mail: [atomlink@mcc.krasnoyarsk.su](mailto:atomlink@mcc.krasnoyarsk.su).

*Matselya Vladimir Ivanovich*, Head of the Office, Technical Management Department, Federal State Unitary Enterprise “Mining and Chemical Combine” (53, Lenin Str., Zheleznogorsk, Krasnoyarsk Region, 662972, Russia), e-mail: [atomlink@mcc.krasnoyarsk.su](mailto:atomlink@mcc.krasnoyarsk.su).

*Druz Dmitry Vitalyevich*, Senior Specialist, Department for Education and Business Development, Federal State Unitary Enterprise “Mining and Chemical Combine” (53, Lenin Str., Zheleznogorsk, Krasnoyarsk Region, 662972, Russia), e-mail: [atomlink@mcc.krasnoyarsk.su](mailto:atomlink@mcc.krasnoyarsk.su).

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