SILVER RECOVERY FROM RADIOACTIVE WASTE GENERATED DUE TO THE DISPOSITION OF SILVER-CONTAINING PRODUCTS

Kuznetsova N. A., Sakhnenko O. A., Kharlova A. G., Lyzlova Ye. V., Zimin P. Yu.

FSUE PA Mayak, Ozyorsk, Chelyabinsk Region, Russia

The studies performed resulted in the development of a method enabling silver recovery from radioactively contaminated silver-containing products. This method includes the following stages: preliminary decontamination of products, dissolution of silver product fragments in a nitric acid solution, production of finely dispersed silver by silver metal displacement with copper, submerged melting of finely dispersed silver at a temperature of at least 960 °C. To reduce the operating costs, evaluated was the feasibility of silver recovery from its compounds (chloride, sulphate and oxide).

Keywords: *radioactive waste, decontamination, silver, radioactive contamination, precipitation, silver recovery, niello, remelting, ingots.*

Introduction

Radioactive silver-containing waste is currently generated at some nuclear enterprises: during their operation, some parts and elements of the equipment constantly interacts with radioactive substances and, in particular, with plutonium. Therefore, their non-removable contamination level with respect to α-activity can amount to 10^5 α-part./(cm²·min), whereas the removable contamination can reach up to 10^4 α -part./(cm²·min).

To recycle this silver, it should be decontaminated to a total contamination level that would not exceed the values established by the sanitary rules of radiation safety (hereinafter, OSPORB-99/2010) [1]. According to OSPORB-99/2010, the radioactive contamination of metals used in the national economy should not exceed 1.0 α-part./(cm²·min). In this case, any removable contamination should be absent, the dose rate of gamma-neutron radiation should not exceed 0.2 µSv/h and the specific activity of plutonium in the metal should be less than 0.1 Bq/g.

This study focuses on the development of a method providing effective treatment of precious metals from plutonium and cooccurring chemical elements, as well as minimal losses of silver during the treatment process and reduced costs associated with secondary waste processing.

The first stage of the study involved a series of decontamination experiments focused on the immersion of items with silver containing fragments into a mixture of concentrated phosphoric and sulfuric acids at a volume ratio of 2 : 1 with citric acid added to a mass concentration of 50 g/dm³ at temperatures of 60 and 90 °C for 1 hour. Table 1 presents the results obtained.

As seen from Table 1, high performance was demonstrated by this decontamination option.

Table 1. Decontamination of items with silver fragments and its results

Reference product number	Ultrasonic treatment	Temperature of the decontamination solution, °C	Initial removable contamination, part./(cm²·min)	Residual removable contamination, part./(cm²·min))	Decontamination factor, K_d
1	Not applied	60	43 600	32	1 362
2			240	16	15
3			74 800	224	334
4	Applied	90	1 400	20	70
5			1 000	background	over 500
6			1 000	12	83
7			1 760	28	63
8			1 960	36	54
9			4 600	736	60

Silver fragments of the items were dissolved directly in a nitric acid with a mass concentration of 450 g/dm3 at a temperature of 60 °C using ultrasonic treatment of the solution until complete dissolution of silver for 40–90 min. Upon its completion, the process was assessed visually. Table 2 presents the compositions of the solutions.

Table 2. Compositions of nitric acid solutions after silver dissolution

Reference number of the item	Volume of nitric acid solution, cm³	Mass concentration, mg/dm³						
		Ag	Ti	Fe	Ni	Cr	Al	Pu
1−3	100	161.4·10³	7	110	308	8	240	35·10⁻³
4−9	200	181.6·10³	26	60	790	10	1022	9·10⁻³

Solutions resulted from the dissolution of silver fragments pertaining to items No. 1—3 and No. 4—9 were handed over, respectively, for sorption treatment and for silver reduction using metallic copper.

Concentration of nitric acid in the solution supplied to the sorption treatment stage was adjusted to provide most effective sorption of plutonium: it was extracted from the solutions in a dynamic mode at a solution filtration rate of 1 c. v./h. Sorbent volume in the column amounted to 5 cm³ (height to

diameter ratio H : D was equal to 5 : 1) with the following ion-exchange materials involved:
• anion exchanger VP-3Ap;
• cation exchanger Purolite S957;
• impregnated sorbent AXION MND 40T.

Plutonium was desorbed from the anionite phase using a 0.5 mol/dm³ nitric acid, whereas the desorption from the cationite phase was done based on a reducing solution with:
• ascorbic acid mass concentration of 10 g/dm³;
• hydrazine molar concentration of 0.2 mol/dm³;
• nitric acid molar concentration of 3.5 mol/dm³.

Plutonium was desorbed from the impregnated sorbent phase using a DTPA solution with a mass concentration of 10 g/dm³ at pH = (5.5 ± 0.5).

At the desorption stage, the solution — sorbent contact time amounted to 60 min. Table 3 summarizes the results of experiments on plutonium extraction from solutions after the dissolution of silver fragments from items No. 1—3.

The study has shown that thanks to the sorption process involving various materials, the activity associated with Pu available in the solution can be dropped to the background levels.

However, it should be noted that sorption treatment results not only in plutonium extraction, but also in significant losses of silver due to its sorption (up to 35%).

Table 3. Results of experiments on the extraction of plutonium from solutions after silver fragment dissolution

Sorbent	Composition of the initial solution			Volume of the supplied solution, c. v.	Mass concentration in the filtrate		Amount of sorbed silver, % of the initial vol.
	Pu, µg/dm³	Ag, g/dm³	HNO₃, mol/dm³		Pu, µg/dm³	Ag, g/dm³	
VP-3Ap	27	110.0	6.5 ± 0.5	4	background	80.5	26.8
Purolite S 957	35	161.0	4.5 ± 0.5	2	background	104	35.4
AXION MND 40T	16	76.8	2.5 ± 0.5	1	background	52.3	31.9
	35	159.3	4.5 ± 0.5	1	background	112.4	29.4
	27	137.5	6.5 ± 0.5	1	background	90.3	34.3

This process taking place on anion exchanger is due to the fact that in a nitric acid solution with a molar concentration of (6.5 ± 0.5) mol/dm^3, silver is available in the form of an anionic complex $[Ag(NO_3)_2]^-$. Sorption of silver on cation exchanger from a solution with a (4.5 ± 0.5) mol/dm^3 molar concentration of nitric acid is due to its presence in the solution in the form of an Ag^+ cation. It was demonstrated that the Purolite S957 cation exchanger also provides solution treatment from such impurities as aluminum, nickel and titanium. The sorption degree of these components amounts to 78, 34, and 86 %, respectively. The partial reduction of silver on the impregnated AXION MND 40T sorbent is apparently associated with the formation of a complex in which silver is bound to the diglycolamide molecule via ether oxygen.

It was demonstrated that 4 volumes of desorbing solutions provide the extraction of 10 to 25 % of plutonium and silver from the impregnated sorbent phase. Relevant extraction levels amount to 65 % and 100 % in case of a cationite and anionite phase respectively.

Considering large silver losses (up to 35 %) during the sorption treatment of nitric acid solution from Pu, it was decided not to introduce this stage in the processing method for silver-containing RW.

After the silver fragments from items No. 4—9 were dissolved, the mixture was supplied to the next stage of treatment to condition the silver to a fine powder by its metallic copper-based reduction. Mass concentration of Pu in the solution was equal to 9 µg/dm^3. The process was implemented at a room temperature until the exothermic reaction accompanied by NO_2 release due to the decomposition of silver nitrate (reaction (1)) was completed. The required copper consumption was evaluated stoichiometrically according to the below chemical reaction (2).

$$2AgNO_3 = 2Ag + 2NO_2 + O_2, \qquad (1)$$

$$AgNO_3 + Cu = 2Ag + Cu(NO_3)_2. \qquad (2)$$

Resulting silver powder (niello) was repeatedly washed with distilled water, then treated on a paper filter and air-dried for one day. The loss of silver during filtration did not exceed 1.5 %.

At the next stage, a weighed portion of the resulting powder was dissolved in nitric acid with a mass concentration of 450 g/dm^3. The impurities content and the mass concentration of silver in the obtained sample were evaluated.

Based on the evaluated samples, the mass fraction of impurities in the silver powder was found to be ranging from 10 to 14 %. This number of impurities can be explained by incomplete washing of the niello from accompanying metal salts and their possible further hydrolysis. The amount of unreduced silver in the nitric acid mother liquor was no more than 0.005 %. The Pu content in the niello was found to be equal to 675 Bq/kg. According to the rules [1], the specific activity of Pu in the metal should not exceed 0.1 Bq/g.

To achieve the required purity of the precious metal, the next stage involved niello fusing under a flux at a temperature of 970 — 1 100 °C. The exposure time accounted for 10—15 minutes. A mixture of sodium tetraborate and sodium carbonate with a mass ratio of 3 : 2 was used as a flux.

The radiation purity of the precious metal upon fusion treatment stage completion was evaluated by radiometric method: the flux density of α-particles escaping the ingot surface was measured. It was found that relevant levels did not exceed the background values of the UIM-2D radiometric unit (the background level was assumed as being equal to 0.6 α-part/(cm^2·min)). The analysis carried out according to the method [2] showed that technogenic α-emitters were absent in the silver scrap. In terms of radioactive contamination, the metal complied with the standards [1].

An insignificant part of silver (about 0.2 %) remained in the slag and on the walls of the crucible. No actual losses of precious metal were identified at the remelting stage.

As a result, a processing technology was recommended for silver-containing metal RW treatment. It involved the following stages [3]:

1. Preliminary decontamination suggesting that the items are immersed in a mixture of concentrated phosphoric and sulfuric acids with the addition of citric acid at a temperature of 90 °C and an exposure time of 1 hour.

2. Dissolution of silver-containing fragments in a nitric acid solution with a mass concentration of 450 g/dm^3 at a temperature of 60 °C involving ultrasonic treatment.

3. Generation of finely dispersed silver in the form of niello by reducing metallic silver with copper from a nitric acid solution.

4. Repeated washing of the resulting silver powder with distilled water, treatment using a paper filter and air-drying for one day.

5. Finely dispersed silver alloying under a flux in corundum crucibles at temperatures ranging from 970 to 1 100 °C and an exposure time of 10—15 minutes. A mixture of sodium tetraborate with sodium carbonate at a mass ratio of 3 : 2 was used as a flux.

6. Removal of slag from the surface of the ingots, first mechanically, and then by their etching in a sulfuric acid solution with a mass concentration of 100 g/dm^3.

Thanks to the developed method the resulting residual metal contamination did not exceed the background levels assumed for the radiometric installation being equal to 0.6 α-part/ (cm^2·min). The performed α-spectrometric analysis of the facings sampled from all the resulting ingots has demonstrated the absence of technogenic α-emitters in the silver scrap.

Loss of precious metal during all the above operations amounted to some 10 %.

To optimize the loss of precious metal, to reduce the number of silver separation operations, and, consequently, the cost of the process, a series of experiments were implemented to extract silver from its compounds: chloride, sulfate and oxide.

This study evaluated the fundamental possibility of producing silver ingots from sulfate and oxide and also explored the precious metal separation methods via silver niello reduction from chloride. The tests involved silver ingots with no radioactive contamination.

Development of a technology for metallic silver production from silver chloride

A method was tested to treat silver from α-emitters [4]. It involved the following operations:
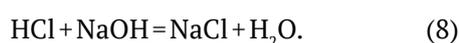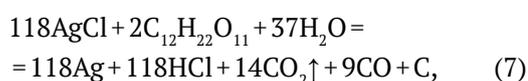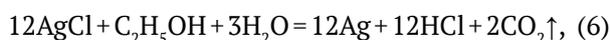- dissolution of silver in nitric acid with a mass concentration of 450 g/dm^3 at a temperature of 60 °C using ultrasonic treatment. The reaction proceeded according to the following equations (3), (4):

$$3Ag + 4HNO_3 = 3AgNO_3 + NO\uparrow + 2H_2O, \quad (3)$$

$$Ag + HNO_3 = AgNO_3 + NO_2\uparrow + H_2O; \quad (4)$$
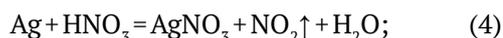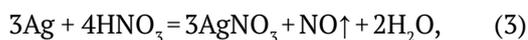
- silver chloride precipitation via concentrated hydrochloric acid addition to a nitric acid solution that proceeded according to the following expression (5):

$$AgNO_3 + HCl = AgCl + HNO_3; \quad (5)$$
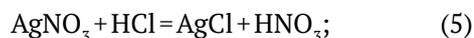
- decantation of sediment;
- reduction of silver from chloride in the form of niello with an aqueous solution of ethyl alcohol and sucrose at a temperature of 50 to 70 °C until the CO_2 gas emission stopped (2—3 h) along with the neutralization of excess hydrochloric acid with sodium hydroxide. The reduction reactions proceeded according to the following expressions (6)—(8):

$$12AgCl + C_2H_5OH + 3H_2O = 12Ag + 12HCl + 2CO_2\uparrow, \quad (6)$$

$$118AgCl + 2C_{12}H_{22}O_{11} + 37H_2O =$$
$$= 118Ag + 118HCl + 14CO_2\uparrow + 9CO + C, \quad (7)$$

$$HCl + NaOH = NaCl + H_2O. \quad (8)$$

At atmospheric pressure and moderate temperatures, the activation energy required for the reduction of silver from its chloride with an aqueous solution of alcohol appeared to be quite high, but practically the process did not proceed. Therefore, it proceeded at high rates, when the mixture was heated from 50 to 70 °C and above. In an alcohol-containing solution, the curdled precipitate of silver chloride disintegrated into tiny particles; this, in turn, contributed to an increased rate and completeness of silver recovery from such a precipitate. In addition, the reduction potential of the sugar-alcohol system was higher than in case when only one of the solutions was involved;
- fine silver sediment treatment on a paper filter;
- air drying of the precipitate at a room temperature;
- fine silver remelting in corundum crucibles under a flux at temperatures from 1 000 to 1 100 ° C for no more than 30 minutes (the silver precipitate was fused together with a filter to reduce the precious metal loss).

Table 4 presents the results, conditions and modes of the experiments.

Table 4. Results of operations designed to generate metallic silver from chloride (conditions and modes)

AgCl dissolution and production mode	Ag reduction from AgCll	Ingot weight, g	Silver loss, %
Ag — 13,23 g, 450 g/dm^3 HNO$_3$ — 100 cm^3, concentrated HCl — 13 cm^3	AgCl* — 16.27 g, NaOH — 20 g, NH$_4$OH — 1.5 g, 20 % C$_2$H$_5$OH — 75 cm^3, 85 % C$_{12}$H$_{22}$O$_{11}$ — 50 g	11.94	9.7
Ag — 12,73 g, 450 g/dm^3 HNO$_3$ — 100 cm^3, concentrated HCl — 30 cm^3	AgCl* — 16.9 g, NaOH — 15 g, 20 % C$_2$H$_5$OH — 60 cm^3, 85 % C$_{12}$H$_{22}$O$_{11}$ — 17 g	12.72	0
Ag — 9,4 g 450 g/ dm$_3$ HNO$_3$ — 100 cm^3, concentrated HCl — 10 cm^3	AgCl* — 12.5 g, NaOH — 19.7 g, 20 % C$_2$H$_5$OH — 90 cm^3, 85 % C$_{12}$H$_{22}$O$_{11}$ — 18.65 г	9.02	4.2
Ag — 6,18 g, 450 g/dm^3 HNO$_3$ — 40 cm^3, concentrated HCl — 7 cm^3	AgCl* — 9.76 g, NaOH — 10.0 g, 20 % C$_2$H$_5$OH — 60 cm^3, 85 % C$_{12}$H$_{22}$O$_{11}$ — 13.2 g	5.96	3.5

*calculated quantity

As seen from Table 4, at different ratios of chemicals, the loss of precious metal did not exceed 10 %. The minimum losses were observed in the second experiment.

According to the authors, the tested method allows to remove the radionuclide impurities in a sizeable way at the precipitation stage, whereas the reduction stage results in niello with required thresholds of residual contamination. Niello remelting into ingots during the refinement of the technology based on silver-containing waste, may not be required, which significantly reduces the cost of the process. In addition, after the silver chloride precipitation, the mother liquor can be recycled to dissolve the feedstock — reaction (5), and after silver reduction, chloride can be precipitated from a new portion of the nitric acid solution — reactions (6) and (7), which, in turn, reduces the generated LRW inventory.

It should be taken into account that the presence of chloride ion in the circulating nitric acid solution is considered unacceptable. To prevent this, recycled hydrochloric acid solution should be added during silver deposition when the residual silver content is checked. The experiments have shown that when the mass concentration of silver in the mother liquor falls in the range of $1-3$ g/dm^3, chloride ion in the liquid phase is practically absent. This silver content in the solution corresponds to a 98% chloride precipitation. The chloridions present in the nitric acid solution intended for the dissolution of silver in a concentration of $2-5$ mg/dm^3 do not produce any negative effect on the dissolution process.

As seen from Table 4, the mass fraction of alcohol in the silver chloride reducing solution amounted to 20%. This alcohol content in the reduction solution is considered optimal. In case of lower alcohol content, it only minorly affects the effectiveness of the process, whereas, according to the literature data, at a higher content, intensive evaporation of alcohol should be observed at reduction temperatures, while the rate of the process stays the same.

To reduce silver in the form of niello, sugar syrup with a 15% mass fraction of water was used. Higher syrup concentration would require solution overheating which was considered undesirable, since it would result in intensive evaporation of alcohol. Application of a more diluted syrup would result in increased volumes of circulating solutions also decreasing the reduction rate.

The process temperature should be maintained within a specified range of 50 to 70 °C. If the temperature rises, alcohol evaporates intensively, the rate of the process and gas evolution becomes excessive (solutions can be thrown out or splashed from the containers used under the reduction process).
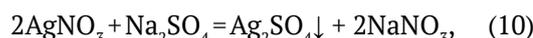
Development of a technology for metallic silver production from silver sulfate

Two ways were considered to produce silver sulfate:
1. Dissolution of metallic silver in hot concentrated sulfuric acid according to the following reaction (9):

$$2Ag + 2H_2SO_4 = Ag_2SO_4\downarrow + SO_2\uparrow + 2H_2O. \qquad (9)$$
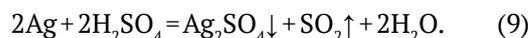
2. Precipitation from solutions under ion exchange reactions according to the following expressions (10), (11):

$$2AgNO_3 + Na_2SO_4 = Ag_2SO_4\downarrow + 2NaNO_3, \qquad (10)$$

$$2AgNO_3 + H_2SO_4 = Ag_2SO_4\downarrow + 2HNO_3. \qquad (11)$$

The resulting precipitate was treated on a paper filter and dried in air at a room temperature, then fused together with the filter at a temperature of 1 200 °C for no more than 3 h.

It is known that silver sulfate forms colorless crystals that melt at 660 °C. At temperatures from 750 to 1 000° C and above, the crystals thermally decompose releasing metallic silver [5] according to the following reaction (12):
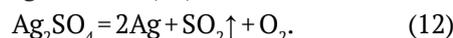
$$Ag_2SO_4 = 2Ag + SO_2\uparrow + O_2. \qquad (12)$$

Table 5 presents the experimental results.

Table 5. Experimental results, conditions and modes for silver sulfate production

Ag dissolution and Ag_2SO_4 production mode	Ingot weight, g	Silver loss, %
Ag − 14.43 g, 450 g/dm^3 HNO_3 − 100 cm^3, concentrated H_2SO_4 − 30 cm^3	13.05	9.5
Ag −13.05 g, 450 g/dm^3 HNO_3 − 100 cm^3, 450 g/l H_2SO_4 − 135 cm^3	10.16	22.0
Ag − 13.35 g, 450 g/dm^3 HNO_3 − 100 cm^3, 190 g/dm^3 H_2SO_4 − 60 cm^3	9.26	30.6
Ag − 7.61 g, 450 g/dm^3 HNO_3 − 23 cm^3, concentrated H_2SO_4 − 4 cm^3	6.83	10.2
Ag − 6.83 g, 450 g/dm^3 HNO_3 − 20 cm^3, 820 g/dm^3 H_2SO_4 − 4 cm^3.	5.66	17.0
Ag − 5.35 g, 450 g/dm^3 HNO_3 − 20 cm^3, concentrated H_2SO_4 − 7 cm^3	4.30	19.6
Ag − 5.34 g, 450 g/dm^3 HNO_3 − 40 cm^3, hot H_2O − 120 cm^3, $Na_2SO_4 \cdot 10H_2O$ − 40.0 g	3.62	32.2
Ag − 9.98 g, 450 g/dm^3 HNO_3 − 40 cm^3, hot H_2O − 60 cm^3, $Na_2SO_4 \cdot 10H_2O$ − 25.6 g	7.40	25.8
Ag − 5.96 g, 450 g/dm^3 HNO_3 − 40 cm^3, hot H_2O − 150 cm^3, Na_2SO_4 − 45.0 g	4.65	21.9

Based on the presented results, one can conclude that the completeness of silver sulfate precipitation based on dilute sulfuric acid and sodium sulfate solution is two to three times less as compared to the application of concentrated sulfuric acid.

However, the use of concentrated acid also does not allow 100 % precipitation. This is explained by the fact that the product of silver ions and sulfate ions concentration in the solution ($2.5 \cdot 10^{-5}$) slightly exceeds the product of the silver sulfate solubility ($1.2 \cdot 10^{-5}$). In addition, in case of a sulfuric acid excess, reactions associated with the formation of poorly dissociated complex ions $AgSO_4^-$ and $Ag(SO_4)_2^{3-}$ soluble in the same sulfuric acid proceed simultaneously with the silver sulfate formation [5].

As seen from Table 5, the minimum loss of precious metal will account for at least 10 % only if the silver sulfate is formed due to the interaction of silver nitrate with concentrated sulfuric acid.

An attempt to dissolve metallic silver in hot concentrated sulfuric acid failed. This reaction can proceed only if silver is present in a powder form [5].

This study has demonstrated the principal possibility of silver sulfate remelting.

Development of a technology for metallic silver production from silver oxide

Silver oxide was generated from its nitric acid solution via its interaction with sodium hydroxide according to the following reaction (13):

$$2AgNO_3 + 2NaOH = Ag_2O\downarrow + 2NaNO_3 + H_2O. \quad (13)$$

Dry alkali was sprinkled to the nitric acid solution in an amount required to neutralize the acid and form silver oxide. Then the precipitate was treated on a paper filter and washed with distilled water until neutral pH level was reached. After drying, the precipitate was fused.

Table 6 presents relevant experimental results.

Table 6. Experimental results, conditions and modes for silver oxide generation

Ag dissolution and Ag₂O production mode	Ingot weight, g	Loss, %
Ag − 5.43 g, 450 g/dm³ HNO₃ − 30 cm³, NaOH − 10.6 g	5.24	3.5
Ag − 5.24 g, 450 g/ dm³ HNO₃ − 30 cm³, NaOH − 10.5 g	5.19	0.9
Ag − 5.17 g, 450 g/dm³ HNO₃ − 30 cm³, NaOH − 10.5 g	5.06	2.3

Based on the data from Table 6, one can see that in case of metallic silver generation from precipitated silver oxide, the loss of precious metal did not exceed 3.5 %.

This technology reduces the number of operations, as well as the cost of the process and the loss of precious metal during the silver fragment treatment.

Conclusion

A flowchart enabling the processing of radioactive silver-containing waste was developed and tested. It involves the dissolution of silver-containing fragments of products, precipitation (reduction) of silver from a nitric acid solution with metallic copper and dry residue remelted into ingots.

Under production conditions, to optimize the loss of precious metal, to reduce the number of silver separation operations, and, consequently, to reduce the cost of the process during the processing of silver-containing items, a technology has been proposed allowing the separation of silver from its oxide and salts: chloride and sulfate.

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Information about the authors

Kuznetsova Natalya Anatolyevna, Head of the Group on Decontamination of Equipment and Special Products, Central Plant Laboratory, FSUE Mayak PA (18, Yermolayev st., Ozyorsk, Chelyabinsk Region, 456780, Russia), e-mail: poniku@mail.ru.

Sakhnenko Olga Anatolyevna, Process Engineer, Central Plant Laboratory, FSUE Mayak PA (18, Yermolayev st., Ozyorsk, Chelyabinsk Region, 456780, Russia), e-mail: sahnenko08@rambler.ru.

Kharlova Alexandra Georgiyevna, Process Engineer, Central Plant Laboratory, FSUE Mayak PA (18, Yermolayev st., Ozyorsk, Chelyabinsk Region, 456780, Russia), e-mail: alex.kharl@gmail.com.

Lyzlova Yevgeniya Viktorovna, Candidate of Technical Sciences, Head of the Group, Central Plant Laboratory, FSUE Mayak PA (18, Yermolayev st., Ozyorsk, Chelyabinsk Region, 456780, Russia), e-mail: e-lyzlova@mail.ru.

Zimin Pavel Yuryevich, Process Engineer, Chemical Metallurgical Plant, FSUE Mayak PA (31, Lenin Ave., Ozyorsk, Chelyabinsk Region, 456784, Russia), e-mail: Zimin_pyu@mail.ru.