

The Influence of Deviations in Process Parameters on the Purification of Uranium from Different Radionuclides

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Abstract—In the present study, the effect of deviations in the process has been determined during the process of the extraction processing of irradiated nuclear fuel (INF) VVER-440 on RT-1 plant on the purification of uranium from ⁹⁹Tc, ¹²⁵Sb, ¹⁰⁶Ru, ¹³⁷Cs, ²²⁸Th, etc. The coefficients of the purification of uranium from these nuclides for both cycles of the Purex scheme of plant are calculated. It has been shown that, upon deviations in the process (the short-term shutdown of process, fluctuations of hydrodynamic mode, deposition of elements in the extractors, etc.), an increase in the coefficients of uranium purification from most unwanted nuclides is observed, except for technetium. However, it has been found that, at an increased inflow of Tc to the second extraction cycle, the purification of the final uranium product from this element may be insufficient.

Keywords: processing of INF, liquid extraction, uranyl nitrate, uranium fission products, coefficients of purification

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INTRODUCTION

The main product of the plant when processing INF is recovered uranium, which after reenrichment can be returned back to the nuclear fuel cycle. In the Russian RT-1 radiochemical plant (FSUE Mayak Industrial Group), regenerated uranium obtained from the extraction processing of INF VVER-440, is available in the form of a melt of uranyl nitrate hexahydrate, then sent to reenrichment on ²³⁵U by the gas centrifuge method. Strict requirements are applied to a uranium melt on the content of a number of fission products (FPs), such as ⁹⁹Tc, ¹²⁵Sb, ¹⁰⁶Ru, and ¹³⁷Cs, as well as daughter products of the radioactive decay of components of INF, such as ²³³Pa (parent nuclide ²³⁷Np), ²³⁴Th (parent nuclide ²³⁸U), and ²²⁸Th (parent nuclide ²³²U) [1, 2].

During the extraction processing of INF, different deviations of the process can be observed, such as failures of the hydrodynamic extraction mode, the appearance of sediments and contaminants in the recycled solutions, the occurrence of undesirable oxidation processes and the deposition (the so-called “tab”) of various elements in the extractors. These phenomena may lead to a reduction of the coefficients of uranium purification ($K_{\text{pur}}\text{U}$) achieved at various stages of the extraction process from the above nuclides.

In the present study, the effect of these deviations on the delivery of unwanted radionuclide contaminants includes uranium products of the scheme and the purification of uranium at two extraction cycles of the RT-1 plant is considered; furthermore, the average $K_{\text{pur}}\text{U}$ achieved during the extraction process are determined.

EXPERIMENTAL

Extraction processing of INF at RT-1 is carried out via a dicyclic Purex scheme (Fig. 1) All operations in two cycles are performed in multistage extractors of the mixer–settler type.

The first step in the framework of first extraction cycle is joint extraction of uranium and plutonium into tributyl phosphate (TBP), with that Tc and Np are also extracted [3]. Most of the uranium fission products, and the products of the radioactive decay of INF components in this operation comes out to highly active raffinate, but trace amounts of certain undesirable elements are capable of moving on the Purex scheme and can be detected in different uranium products. This operation is performed in the main extractor 1.

Extractant with extracted components is fed into the machine of reductive reextraction Pu 7 (Fig. 1), where the latter, together with Tc and Np under the influence of the produced in the apparatus of U(IV)

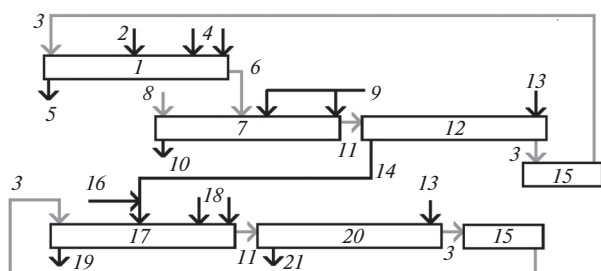


Fig. 1. Schematic diagram of the Purex process of RT-1 plant: 1. head extractor (joint extraction of U and Pu); 2. initial solution of INF; 3. back extractant; 4. nitric wash solutions; 5. highly active raffinate; 6. extractant with the extracted components; 7. device of reducing Pu reextraction; 8. additional flow of back extractant; 9. reducing and complexing reagents; 10. Pu reextract; 11. extractant with U; 12. device of reextraction of U of the first cycle; 13. reextracting solution; 14. uranium reextract flow of the first cycle; 15. regeneration device of extractant; 16. solutions of previously processed uranium; 17. device of uranium extraction of the second cycle; 18. nitric wash solutions with complexing agents; 19. low-active raffinate; 20. reextraction device of U of the second cycle; 21. flow of uranium reextract of the second cycle.

and washing solution, which contains hydrazine, as well as complexing reagents, is removed to the aqueous stream and directed to the separate extraction cycle (is not shown in the diagram). After removing Pu, Tc, Np, and extractant flow with uranium is directed to the reextraction operation to corresponding unit 12, which received uranium reextract enters the second extraction cycle, and extractant after regeneration returns into main extractor.

Second extraction cycle structurally repeats first (except for the operation of reducing reextraction of Pu) and also supposes the extraction of uranium into TBP with washing extract 17, wherein the residual nuclide pollutants are removed to raffinate of the second cycle. Next, the reextraction of uranium 20 is performed and reextract is directed to obtain a melt of uranyl nitrate hexahydrate.

To determine the effect of technological deviations in uranium purification, an operating period of the extraction scheme with a duration of 24 days was considered. The sampling and analysis of uranium products was produced regularly throughout the considered period.

K_{pur} of uranium from different nuclides for each extraction cycle were found as the ratio of the specific

activity or content of these components in the initial solution, which enters the cycle, to their specific activity in the uranium reextract, which exits the cycle.

For all samples, the specific activity of different β - and γ -emitting nuclides on the spectrometer of the gamma radiation energy was measured using a SEG-01-SCD semiconductor detector in the geometry of Marinelli vessel and SEG-05-SCD. The specific content of Tc in the studied solutions was determined using mass spectrometers with inductively coupled plasma (ICP MS) Agilent-7500 CX and Perkin Elmer Elan PRC II.

It should be noted that, in order to determine $K_{\text{pur}} \text{U}$ in the first cycle, uranium reextract was taken directly from extractor apparatus 12 because a significant flow of the uranium solution 16 recycled earlier is subsequently joined with this product in the technological scheme. Despite that an analysis of the combined solution cannot be used to calculate $K_{\text{pur}} \text{U}$ on the first cycle, a continuous analysis of the samples of this product enabled the tracking of fluctuations in the distribution of radionuclides, i.e., pollutants at process deviations on the first extraction cycle, since the specific activity of nuclides, which are pollutants in the previously recycled uranium solutions during the observation period, did not change.

Moreover, for highly active products, which enter the first extraction cycle directly from the dissolving operation of INF, it is often impossible to determine the specific activity of a nuclide like ^{228}Th and ^{234}Pa , or the specific content of Tc; as a consequence, these parameters were determined by calculations based on data on the burn-up range and the exposure of recycled INF. The average burn-up of INF VVER-440 processed for the observation period was 40 GW day/t of U.

RESULTS AND DISCUSSION

At the first stage of the work, it was identified how abnormalities of the process affect the purification of uranium in the first extraction cycle. The averaged data on the specific activity of some of the most important nuclides, FPs, and the specific power of the exposure dose (PED) in the initial solution of INF are given in Table 1.

Specific activity of INF solution is caused mainly by ^{137}Cs , (86% of the total specific activity), while the activity proportions of radioisotopes of ruthenium,

Table 1. Specific activity of certain of fission products in initial solution of irradiated nuclear fuel

| PED, $\mu\text{R/s g U}$ | Specific activity of the nuclide, Bq/gU | | | | |
|--------------------------|---|--------------------|--------------------|--------------------|--------------------|
| | ^{125}Sb | ^{106}Ru | ^{137}Cs | ^{144}Ce | ^{154}Eu |
| 14.24 | 2.75×10^6 | 1.84×10^8 | 3.15×10^9 | 1.74×10^8 | 1.47×10^8 |

Table 2. Specific activity of a number of nuclides in uranium reextract of the first cycle and $K_{\text{pur}}\text{U}$ from these nuclides

| Observation day | PED, $\mu\text{R/s g U}$ | Specific activity of nuclide, Bq/g U | | | | |
|---|-----------------------------|--------------------------------------|--------------------|--------------------|--------------------|--------------------|
| | | ^{125}Sb | ^{106}Ru | ^{137}Cs | ^{144}Ce | ^{154}Eu |
| 1 | 4.4×10^{-5} | 427 | 4.40×10^4 | 130 | 54 | 2 |
| 4 | 4.1×10^{-5} | 501 | 3.65×10^4 | 555 | 57 | 13 |
| 8 | 1.0×10^{-4} | 745 | 1.05×10^5 | 121 | 56 | 32 |
| 10 | 1.0×10^{-4} | 654 | 1.09×10^5 | 164 | 49 | 2 |
| Average | 7.3×10^{-5} | 582 | 7.36×10^4 | 242 | 54 | 12 |
| $K_{\text{pur}}\text{U}$ from the component | 1.95×10^5 | 4.72×10^3 | 2.51×10^3 | 1.30×10^7 | 3.23×10^6 | 1.21×10^7 |

Table 3. Specific content of Tc and specific activity of certain radioactive products of decay in the initial solution of irradiated nuclear fuel and in uranium reextract of the first cycle, achieved $K_{\text{pur}}\text{U}$ from Tc, and number of radioactive decay products

| Nuclide | Specific activity of nuclide, Bq/g U | | $K_{\text{pur}}\text{U}$ from the component in the first extraction cycle |
|---|--------------------------------------|--------------------------------------|--|
| | in the initial solution of INF | in reextract of U of the first cycle | |
| ^{228}Th | 1439 | <3 | >537 |
| ^{233}Pa | 1.9×10^4 | 5350 | 11 |
| $^{234}\text{Th} + ^{234\text{m}}\text{Pa}$ | 2.14×10^4 | 1068 | 32 |
| $^{99}\text{Tc}^*$ | 946.8 | 2.35 | 406 |

* Given in $\mu\text{g/g}$.

cerium, and europium were about the same and in total were 14% of the total specific activity.

The data on the specific activity of the most important γ -emitting nuclides fission products in the uranium reextract directly from the reextractor apparatus and average $K_{\text{pur}}\text{U}$ from these nuclides achieved during the first extraction cycle are given in Table 2.

Thus, the total purification from gamma-active impurities was approximately 2×10^5 , purification from cesium was the highest, i.e., about 10^7 , which is explained by the practical nonextractability of cesium in TBP [4]. The lowest was purification of uranium from ruthenium, i.e., about 10^3 , which is due to the existence of quite high extractable compounds of this element in nitric medium [5, 6].

The measured values of the specific activity of ^{234}Th , along with the subsidiary $^{234\text{m}}\text{Pa}$, ^{233}Pa , and the specific content of Tc in the uranium reextract of the first extraction cycle were correlated with the calculated values of these parameters for the original product to be processed, which allowed the coefficients of the purification of uranium from these components in the first extraction cycle to be obtained.

The specific content of Tc per 1 g of U that enters the first extraction cycle was calculated based on the data in [7] on the content of Tc data in INF from VVER-440 with different burn-up and was in average $947 \mu\text{g/g}$ of U for recycled INF. The specific activity of ^{233}Pa and ^{234}Th in the initial solution was calculated

based on the data on [8]. The specific activity of ^{228}Th was calculated according to the data on the exposure time of INF and the values of the mass fraction of ^{232}U in each of the processed operations, the decay product of which is ^{228}Th . The results of the calculations and $K_{\text{pur}}\text{U}$ from the corresponding components are shown in Table 3.

$K_{\text{pur}}\text{U}$ from Tc varied in the range of 340–450 and $K_{\text{pur}}\text{U}$ from ^{228}Th exceeded 500, which allowed us to obtain a uranium reextract of the first cycle that was almost free of this nuclide.

The lowest value was $K_{\text{pur}}\text{U}$ from nuclides from its own chain of the radioactive decay, Σ (^{234}Th and $^{234\text{m}}\text{Pa}$), as well as from ^{233}Pa , which was the product of the radioactive decay of ^{237}Np .

Figure 2 shows the values of the specific activity and the specific content of a number of important nuclides pollutants in the combined flow of uranium reextract of the first extraction cycle and previously processed uranium (hereinafter referred to as the combined reextract of uranium of the first cycle). The total content of γ -emitting nuclides in this uranium product is given according to the PED of analyzed solutions.

Two regions can be identified on graphs I and II, in which there was a marked increase in the specific activity of a number of nuclides in the joint reextract, as well as region III, where this effect was less pronounced. The behavior of thorium should be specially

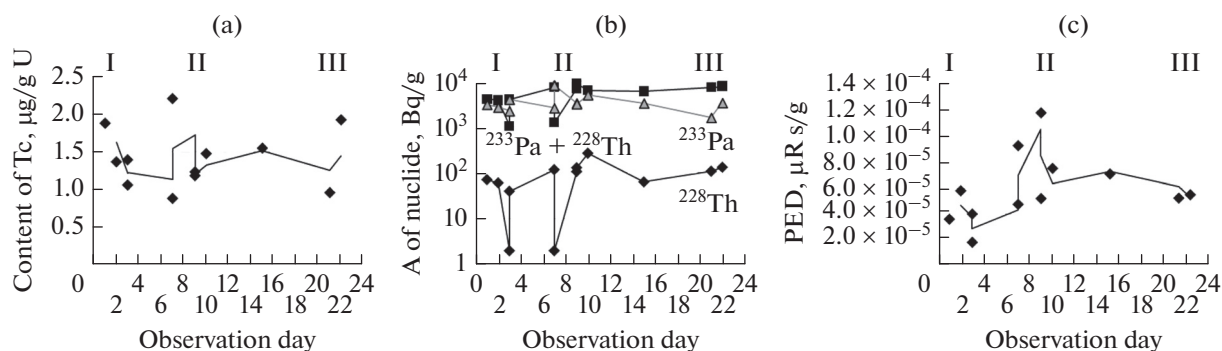


Fig. 2. Specific content of Tc (A), specific activity of ^{228}Th , ^{233}Pa , $^{234}\text{Th} + ^{234\text{m}}\text{Pa}$ (B), and specific PED (C) in the combined uranium reextract of the first cycle.

noted, where these moments first showed a sharp decline in activity, after which a rise was observed (Fig. 2c).

It has been found that the delivery of radionuclides to the combined reextract in region I is associated with the formation of a so-called pack of plutonium in the plutonium reextractor device, which occurs during the short stop and restart the extractor. As can be seen from Fig. 3, on the second or third day of the observation period, which switch off the extractors, was carried out (termination of the feeding of aqueous and organic phases), while in the device of reducing the reextraction, an increase in temperature was observed, which indicates the development of a fairly intense oxidation process, which leads to the rapid destruction

of hydrazine; consumption of U(IV); and, as a consequence, to the reoxidation of plutonium and technetium to be extracted state.

Figure 4 shows data on the change in the plutonium content in the aqueous phase of several steps of the device of reducing reextraction of plutonium. Resuming the work of this extractor entailed the movement of extractable Pu(IV), along with the organic phase from the feeding step n in the direction of the last stages of the device-reextractor of plutonium, so there was a sharp decrease in the weight concentration of plutonium in the aqueous phases of the extraction steps to the step $n + 3$, inclusive. Then, since the feed of the reducing agent has been renewed, the plutonium front in the organic phase came into contact with the hydrazine-containing front in the aqueous phase of stages $n + 3$ and $n + 4$, which has led to an increase in the plutonium content in aqueous phase of stages $n + 1$, $n + 2$, and $n + 3$; then, the plutonium front started to gradually shift toward the first stages of the extractor ($n - 5$) and, as a result, the machine operation was normalized. During this period, Tc was distributed on the device in the same way, the extraction behavior of which in the machine for reducing reextraction is similar to plutonium.

This development of the reducing process has led to an increase in feeding plutonium to the washing zone of the device for the reductive reextraction of plutonium and, then, to reextract the first extraction cycle of elements such as Tc and a number of other nuclides, e.g., Ru, exposed during the normal operation of the extractor to the effect of reducing washing solutions that contribute to the removal of these elements from uranium extract. In addition, the intake of uranium fission products and radioactive decay products of INF components could be due to fluctuations in the hydrodynamic regime of the extractor during shutdown and restart.

In the 7th–10th days of the observation period in the head extractor of the first cycle accumulation of fine sediments is observed, which contributed to the intensive formation of interfacial structures, which

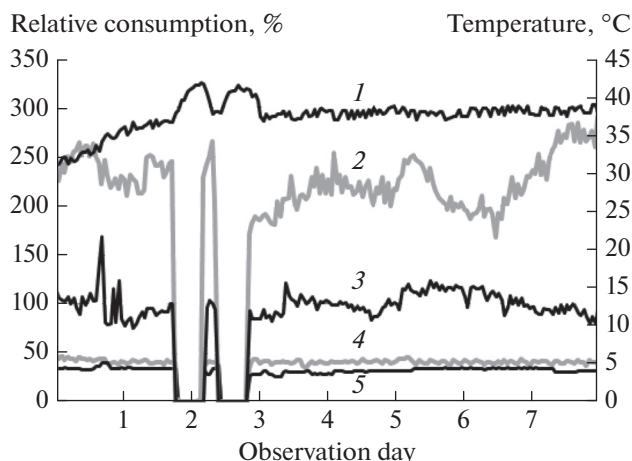


Fig. 3. Operating parameters of the head extractor device and device of reducing reextraction of plutonium. 1. average temperature in the device of reducing reextraction of Pu; 2. consumption of the organic phase to the head extractor; 3. consumption of the initial solution of INF to the head extractor; 4. consumption of organic phase to the device of reducing reextraction of Pu; 5. consumption of the washing solution to the device of reducing reextraction of Pu. Consumption is shown in percents relative to the average consumption of the aqueous phase during normal operation of the extractor.

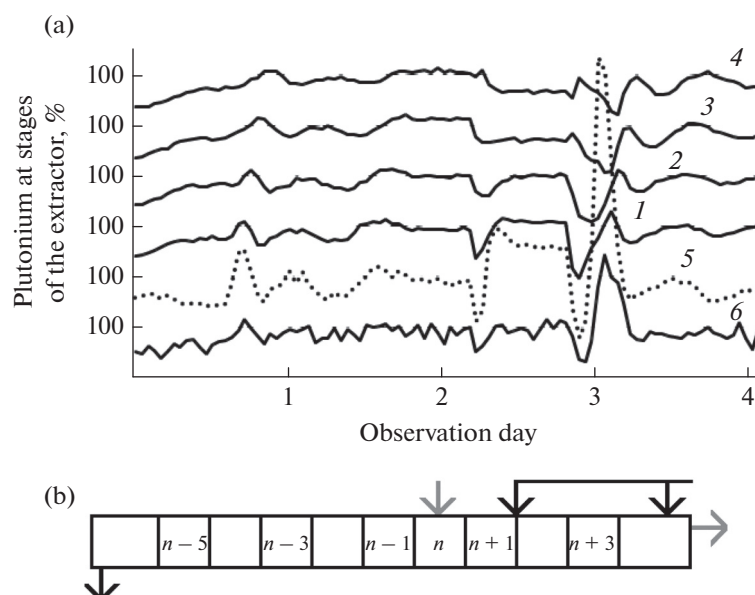


Fig. 4. Distribution of plutonium at (a) stages of the device for reducing reextraction and (b) simplified diagram of this unit. Plutonium content in 1. power stages n (feeding step to the extractor of organic phase with extracted components from the head extractor); 2. stage $n - 1$; 3. stage $n - 3$; 4. stage $n - 5$; 5. stage $n + 1$; 6. stage $n + 3$. Pu content at each stage is given in percents of the average content of Pu at this stage for the entire observation period.

concentrates FPs on them and the products of radioactive decay, as well as the transfer of these nuclides with interphase formations on the extraction cascade. At this time, there was a second maximum of the specific activity values of nuclides in the joint reextract of uranium of the first cycle (Fig. 2, region II).

Upon the resumption of the operation, specific activity of the combined uranium reextract of the first cycle of remained elevated until the end of the observation period due to the presence of these suspensions and interfacial structures. Then, upon the replacement of essential components from the extractors (Fig. 2, region III), growth in the content of technetium, europium, thorium, and protactinium in the joint uranium reextract was noted.

It has also been found in this work that, during the observation period, there were also a number of slips of undesirable radionuclides into reextract of the second extraction cycle. Data on the change in the specific activity, the content of radionuclides, and PED for this product are shown in Fig. 5.

It can be shown that, on the 4th–6th days of the observation period, for reextract of the second cycle, there was a smooth increase in the specific activity of basic β , γ -emitting nuclides (^{106}Ru , ^{137}Cs , ^{125}Sb), as well as Tc. Significant growth in the specific PED of uranium and slip of Tc to reextract of the second cycle was recorded on the 8th–9th days (Fig. 5, region IV), which is associated with the considerable intake of these components by the uranium reextract of the first cycle and second cycle as a result of the above-

described process with the accumulation of suspensions on the first cycle.

The peak values of PED were achieved on the 13th day (V), while the appearance of previously undefined ^{60}Co , ^{154}Eu in the uranium reextract of the second cycle was also noted. Increased values of PED should be noted that were achieved in field VI, which was mainly caused by ^{106}Ru , ^{137}Cs , and ^{125}Sb ; an increase in the content of Tc in the reextract was not fixed in the specified time. On day 19–20, i.e., period VII, an increase in the specific activity of all determined nuclides was noted, as well as increase in the content of Tc.

It was found that the PED peak in region V was directly related to switching off the second cycle produced on days 12–13 of the observation period (Fig. 6), wherein it can be seen that, while switching off, the flow rate of aqueous phase, directed to the second cycle, at some time was higher than the flow rate of the organic phase to the first stage of the considered extractor. This led to the exit of the uranium front to the side of first extractor stages and reducing the weight concentration of uranium in organic phase of the power stage n (Fig. 7). Decreasing the saturation of organic phase with uranium led to the extraction of gamma-active nuclides and entering them to the uranium reextract of the second cycle. Violations (fluctuations) in the hydrodynamic regime of the extractor at the restart could also contribute to the slip of undesired nuclides to reextract.

Switching off the feeding of the aqueous phase and decreasing the saturation of extractant on the 16th day

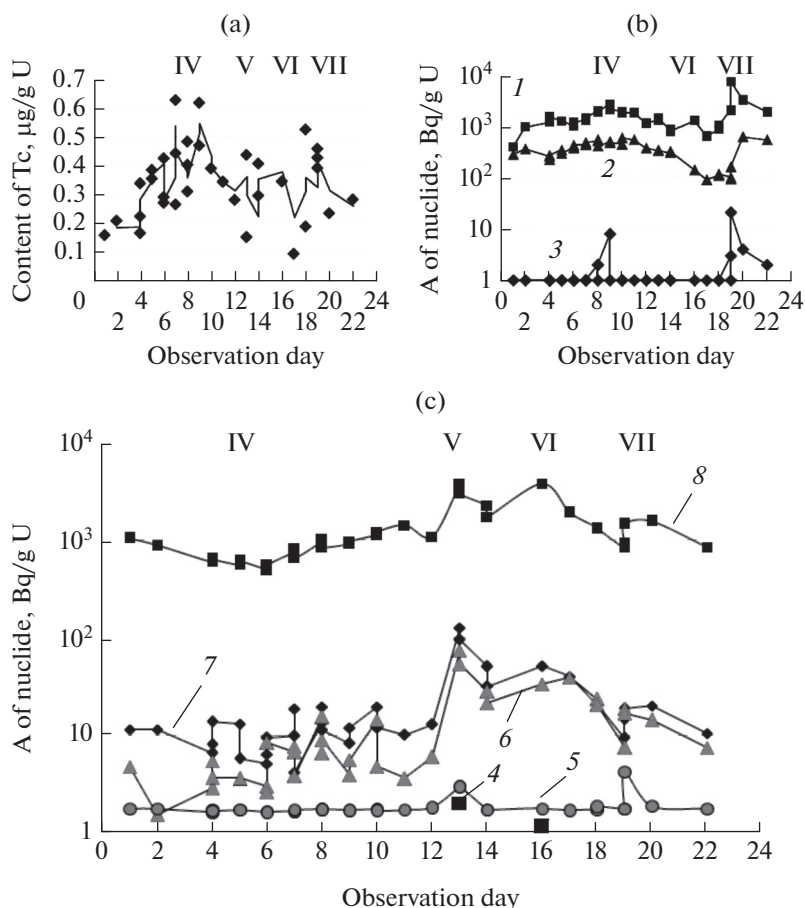


Fig. 5. Specific content of Tc (A) and specific activity of the number of nuclides of uranium reextract of second extraction cycle (B, C). 1. $2^{34}\text{Th} + {}^{234\text{m}}\text{Pa}$; 2. 2^{33}Pa ; 3. 2^{28}Th ; 4. 6^0Co ; 5. 1^{54}Eu ; 6. 1^{37}Cs ; 7. 1^{25}Sb ; 8. 1^{06}Ru .

(Figs. 6, 7) once again led to an increase in the specific activity (PED) of reextract; the main nuclide that contributes to the activity was ^{106}Ru (period VI).

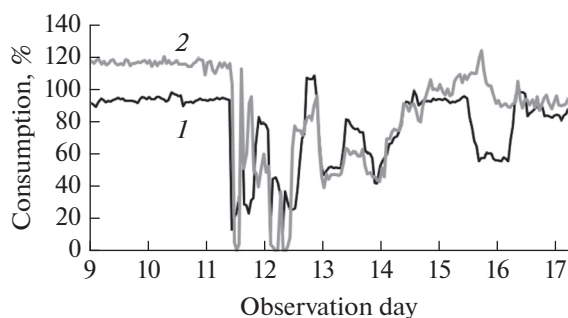


Fig. 6. Operation of the device to extract uranium in the second extraction cycle: 1. consumption of the starting uranium solution; 2. consumption of the organic phase. Consumption is given in percents relative to the average flow rate of the aqueous phase during normal operation of the extractor.

On days 19–20 (period VII), the peak increase in the specific activity of all considered nuclides occurred that was apparently due to the recommissioning of the first extraction cycle (Fig. 3) and continued (due to the above-described accumulation of the interfacial formations and contamination of the organic phase) increase in the specific activity of unwanted nuclides in the reextracts obtained in the first cycle.

Figure 8 shows the values of $K_{\text{pur}}\text{U}$ on the most important components on the second extraction cycle for the entire observation period. The data show that $K_{\text{pur}}\text{U}$ from most nuclides-pollutants have increased dramatically on days 8–9 (period IV), when the yield of these nuclides from the first extraction cycle was maximum. Thus, $K_{\text{pur}}\text{U}$ from ^{137}Cs at a given moment was ~ 4500 at normal K_{pur} from this element on the order of 100–500, $K_{\text{pur}}\text{U}$ from radio ruthenium is increased to 123 on average at $K_{\text{pur}} = 45$, $K_{\text{pur}}\text{U}$ from PED was 118 on average $K_{\text{pur}}\text{U}$ from PED = 23. These facts indicate the significant margin of opportunities

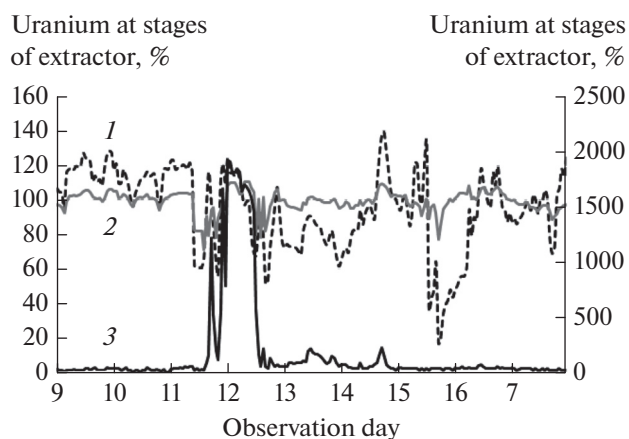


Fig. 7. Distribution of uranium at stages of the device for extracting uranium during the second extraction cycle. Uranium content in: 1. aqueous phase of the feeding stage (left axis); 2. organic phase of the feeding stage n (left axis); 3. aqueous phase of stage $n - 2$ (right axis). For each stage, uranium content is given in percents of the average value for this stage for the entire observation period.

for the extraction purification from these elements in the second extraction cycle.

The sharp decrease in K_{pur} of uranium from ^{228}Th that is observed at the time when packs or other deviations from the normal course of the technological process arise in the first extraction cycle in periods I and II is caused not by the insufficient purification of uranium from thorium in the second cycle, but the small entrance of thorium to reextract of the first cycle at the indicated moments (Fig. 2). It is important to point out that, during period IV, a significant increase in $K_{\text{pur}}\text{U}$ from thorium was observed, which also indicates the reserve of purification from thorium in the second extraction cycle.

It should be noted separately that $K_{\text{pur}}\text{U}$ from Tc in the second extraction cycle has been very low. Furthermore, at the moment of deviations in the first extraction cycle, at an elevated admission of Tc to reextract of the first cycle, $K_{\text{pur}}\text{U}$ from Tc dropped to 2–3 in period IV vs. 5–10 in the operation at normal mode. These facts indicate that the margin of the possibilities for the removing Tc at the elevated (peak)

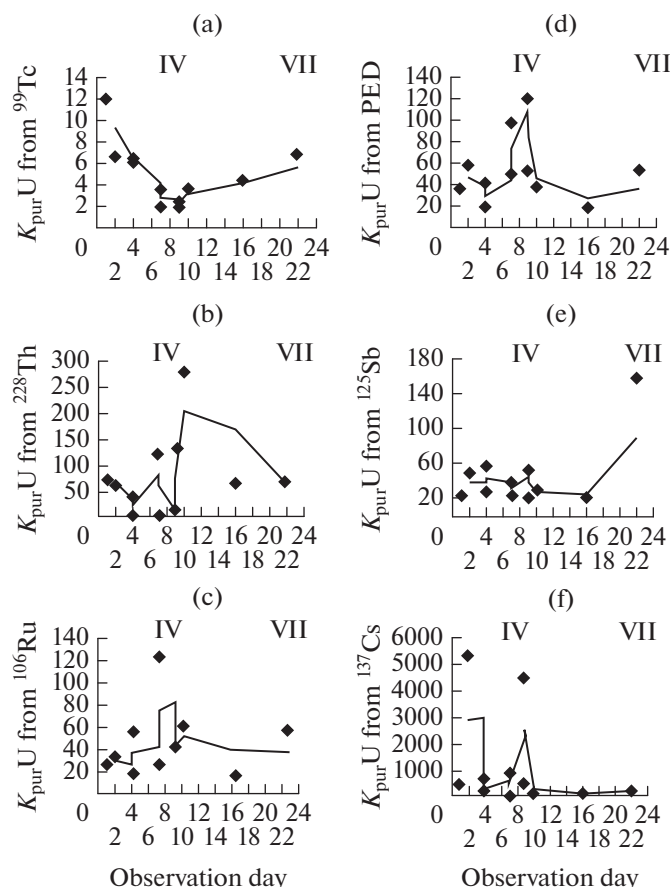


Fig. 8. Coefficients of the purification of uranium from (A) ^{99}Tc , (B) ^{228}Th , (C) ^{106}Ru , (D) PED, (D) ^{125}Sb , and (E) ^{137}Cs observed during operation during the second extraction cycle.

admission of this element to the second cycle may not be sufficient.

CONCLUSIONS

It has been shown that the short-term stop of the extraction process, the fluctuation of the hydrodynamic regime of extractors, the formation of packs of components in the first extraction cycle, and the drop in the saturation of the organic phase with uranium in the second extraction cycle, as well as the accumulation of sediments and interfacial formations in the first cycle, lead to the admission of nuclides to uranium reextracts of the first and second cycles. It has been found that, at the elevated admission of most of the considered nuclides to the extraction $K_{\text{pur}}\text{U}$ increases, i.e., there is a margin of possibilities for purification.

However, it has been revealed that $K_{\text{pur}}\text{U}$ from Tc in the second cycle is not high, i.e., it is about 5, and the margin of the possibility of purifying uranium from Tc in this cycle is not sufficient that increasing the admission of Tc may result in the increased exit of this element to the uranium product.

The maximum purification of uranium in two extraction cycles was achieved from cesium (1.5×10^{10} for $K_{\text{pur}}\text{U}$), 1.1×10^{10} for $K_{\text{pur}}\text{U}$ from europium, 1.5×10^5 for $K_{\text{pur}}\text{U}$ from ruthenium, and 2.0×10^5 for $K_{\text{pur}}\text{U}$ from antimony). The specific activity of thorium-228 was reduced by 4.0×10^4 times, the specific content of Tc was decreased by 2000 times, the total specific activity of the main nuclide pollutants (according to the analysis of the PED of the solutions) was decreased by 10^7 times. The smallest values were obtained for $K_{\text{pur}}\text{U}$ from protactinium-233, by only about 120, which is caused by both the complex chemical behavior of this element and its rapid accumulation from parent nuclides. In general, the observed $K_{\text{pur}}\text{U}$, which enables one to obtain the conditioned melt of uranyl nitrate hexahydrate on all of these nuclides.

The improvement of purification indicators for the extraction system may be achieved by developing processing methods, which allow one to increase the discharge of Tc into high-level raffinate of the first cycle

and, accordingly, reduces the intake of this element during the process of reducing the reextraction of Pu and, subsequently, via the extraction scheme. Furthermore, measures that minimize undesirable oxidation processes at the step of plutonium reextraction can be considered that increase the stability of its flow, as well as measures that lead to an increase in the purification of uranium from Tc, Ru, and Th during the second extraction cycle.

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