COMPREHENSIVE FLOWCHART PROPOSED FOR PROCESSING SOLUTION AND SEDIMENT PARTS OF ACCUMULATED HLW OF COMPLEX CHEMICAL COMPOSITION FROM THE MAYAK PA RADIOCHEMICAL PLANT

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The paper presents a comprehensive multi-option flowchart proposed to process the accumulated HLW inventories of complex chemical composition describing the core processes, the characteristics of products formed at each processing stage and the relationship between the individual elements of the proposed flowchart. It specifies the process equipment (or its prototypes) to be introduced under the proposed flowchart. It presents the thresholds for the preliminary feasibility assessment of the processing options proposed for the solution and the sedimentary parts of the accumulated HLW inventories. The study demonstrates the feasibility of further efforts seeking to select optimal design options under the proposed flowchart.

Keywords: *storage tanks, high-level waste, sediment, microfiltration, neutralization, sorption, extraction, cementation, vitrification, electric furnace, cold crucible induction heater, radioactive waste.*

Mayak PA storage tanks hold approximately 14,000 m^3 of alkaline high-level pulps that resulted from the precipitation treatment applied in the reprocessing of irradiated fuel from nuclear reactors. To date, the challenge associated with the processing and conditioning of accumulated high-level waste (AHLW) still remains unresolved.

The buildup of this waste group and the evolution of its physicochemical state was described in detail in [1], [2]. At present, this waste mostly refers to alkaline systems of sediments and clarified solutions with a predominating solution part. The sediments consist mostly of aluminum, iron, nickel, chromium, manganese and silicon compounds. The liquid phase comprises highly mineralized solutions, with sodium nitrate, hydroxide, aluminate and nitrite prevailing in their chemical composition. Composition and activity of the AHLW solution part depend on the height of its location above the sediment. In lower layers of the clarified phase, the total beta activity is higher, $^{90}Sr + ^{90}Y$ contribution to this activity is greater and concentration of major macro components of liquid radioactive waste (LRW) is higher as well.

In terms of radionuclide composition, AHLW involves fission products and actinides, the concentration of which in the sediment is much (tens to hundreds of times) higher than in the clarified phase. The sediment activity is attributed to cesium, strontium, plutonium and americium radionuclides. As regards the solution part, 99,9 % of its activity is due to ^{137}Cs.

There are two factors due to which the processing and conditioning of this waste group is considered challenging: there is no regular system for washing out the dense sediment layer and the chemical composition of this waste is way much complex and may differ considerably depending on a particular

tank. Data on the sediments and their composition are extremely fragmentary and heterogeneous. On the other hand, analytical evaluation of waste composition is also challenged by the complexity of the representative sample collection process and by high activity of the materials, which results in large errors. Early in the selection of appropriate methods, a calculation method was applied to evaluate the chemical composition of the stored slurries, which was based on the available records on the chemical composition and volumes of waste kept in the tanks. Fig. 1 summaries the calculated data on the elemental composition of the slurries that had been discharged into the storage tanks [1].

In 2021—2022, Mayak PA developed a comprehensive multi-option flowchart providing the extraction and processing of the solution and sedimentary waste parts [2] (Fig. 2).

This flowchart (both its individual elements and their couplings) was developed under the state contract No. H.4d.241.20.22.1057 signed on April 4, 2022 between the Rosatom State Corporation and the Mayak PA (Development and Feasibility Study of Processing Options for High-Level Waste of Complex Chemical Composition, Including Experimental Design Development and Pilot Testing of Equipment) that was implemented in 2022—2024. This research is expected to support the selection of most promising options for further implementation.

At the initial stage, four options were proposed to process the solution part of the waste (Fig. 3—6). Preliminary removal of alpha-emitting radionuclides associated with suspensions and colloids from the solution using tangential modules with ceramic filters was a stage common for all of the proposed options. In particular, this operation contributed to lower alpha activity of the solution so that the final compound could be referred to

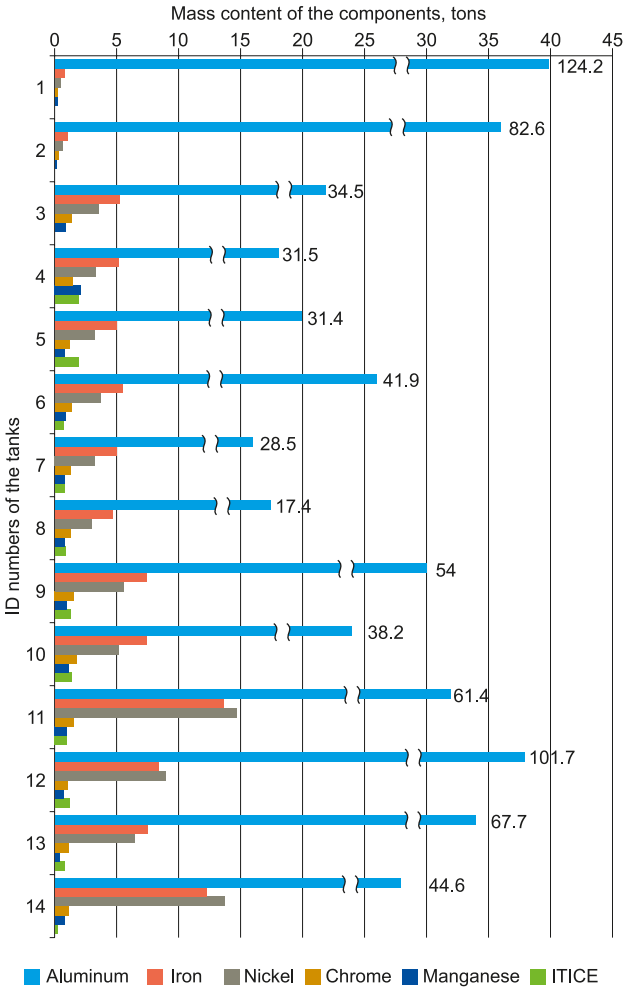

Fig. 1. Mass content of the main elements and components (except sodium) in the storage tanks (ITICE — inorganic titanium-iron cation exchanger)

Class 3 radioactive waste (RW) according to [3], [4]. Moreover, this is true not only when it comes to the processing of well-settled upper LRW layers with a minimum content of suspensions, but also when handling a moving layer of easily disturbed

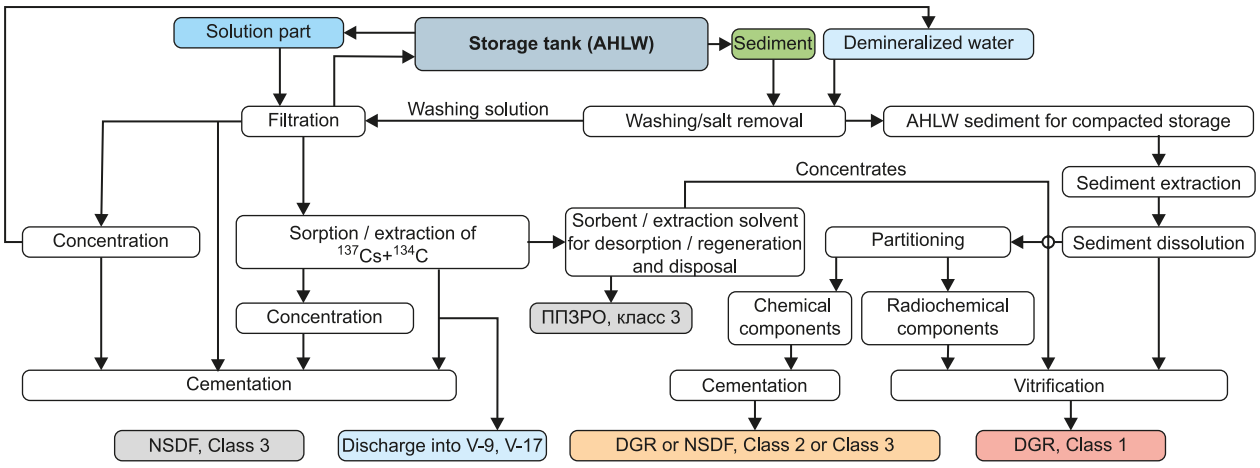

Fig. 2. Comprehensive AHLW processing flowchart providing for waste stream separation

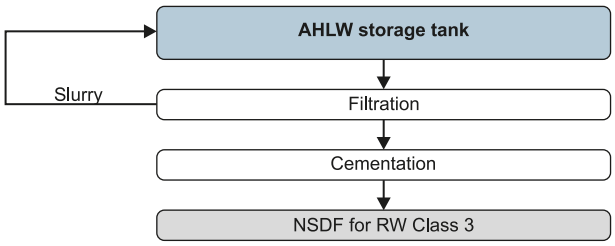

Fig. 3. Processing flowchart for the solution part of the waste according to option No. 1

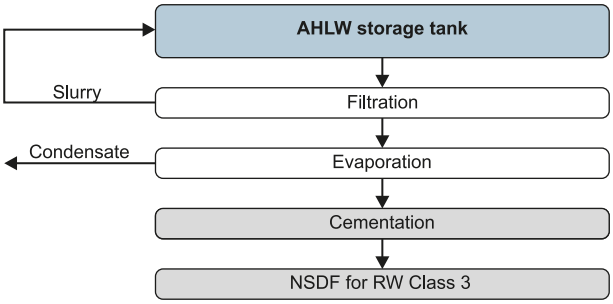

Fig. 4. Processing flowchart for the solution part of the waste according to option No. 2

suspension separating the clarified solution and the dense sediment. The suspensions removed from the solution during filtration in the form of concentrated slurry are sent for processing together with the dense sediment. Solutions can be filtered in a unit similar to the one developed by research and production company (RPC) OOO Sosny designed to separate alpha-emitting nuclides at the Cementation Complex for Liquid Intermediate-Level Waste (ILW) resulting from the operation of Mayak's radiochemical plant.

Option No. 1 provides for direct solidification of the filtered AHLW solution portion by the cementation method without any additional pre-treatment (Fig. 3). Slag-alkali cements are used for cementation purposes, which ensures the required quality of the compounds resulting from the immobilization of salt solutions high in free alkali [5]—[7]. The cementation process can be implemented both in a unit developed by RPC Sosny (the hardening mixture is produced in an insert constituting to NZK-150-1.5P or KZH-2ks-01 container designs with a mixing device operating based on "adrift mixer" principle (basic option)) and in mixers of the ILW cementation complex operated at the radiochemical plant providing for compound emplacement into compartments belonging to a plastic cellular multi-tiered storage system.

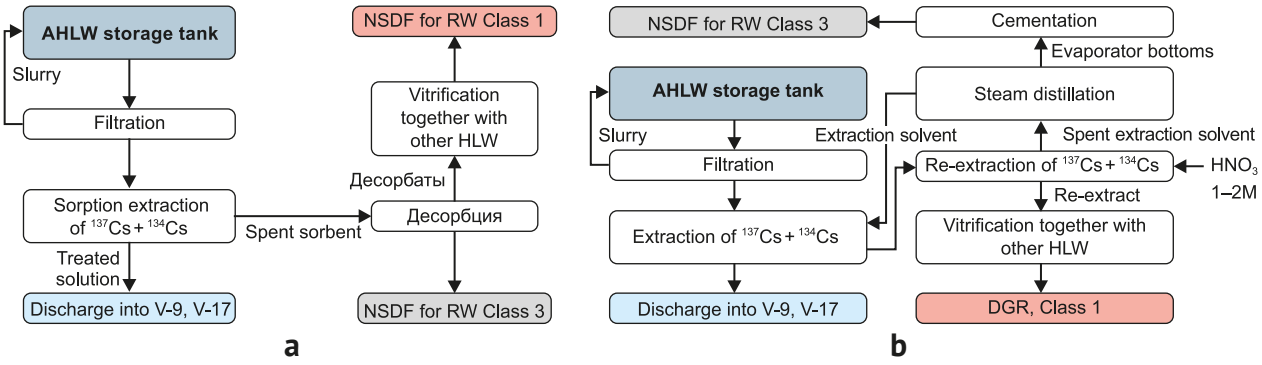

Fig. 5. Processing flowchart for the solution part of the waste according to option No. 3: a) sorption, b) extraction

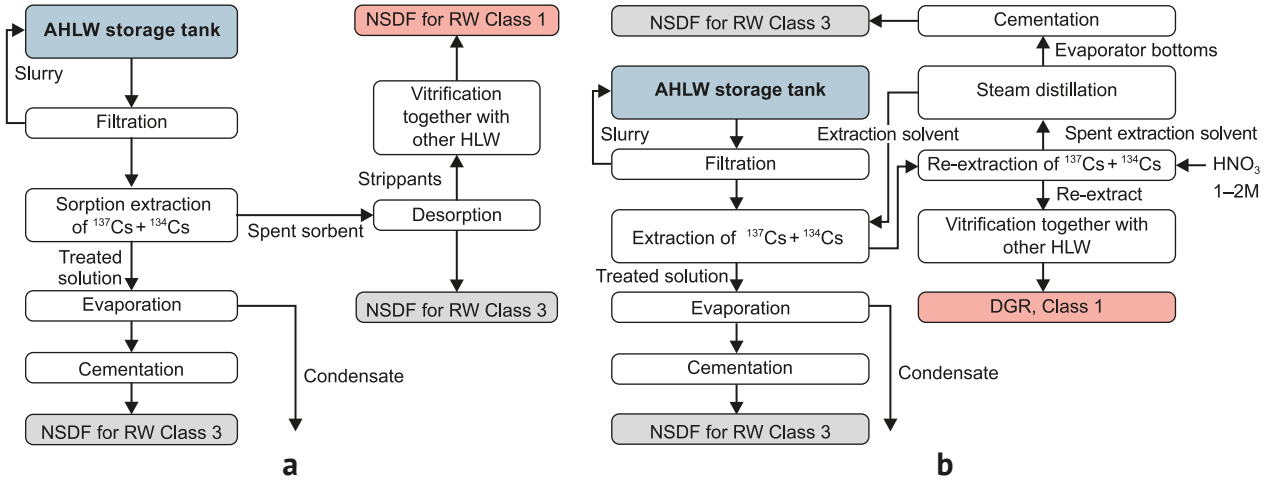

Fig. 6. Processing flowchart for the solution part of the waste according to option No. 4: a) sorption, b) extraction

Option No. 2 provides for an evaporation stage preceding cementation to reduce the waste volume (Fig. 4). Evaporation resulting in a decrease of the waste volume by about a factor of two is suggested due to virtually highest possible concentration of dissolved compounds of 650—700 g/dm^3 reached in the treated part corresponding to the degree of salt component inclusion into cement compound of about 23 % by weight [7]. This process can be implemented in a direct-flow evaporator.

In accordance with the evaporation efficiency, LRW concentration via option No. 2 may provide approximately two-fold reduction in the volume of the resulting conditioned RW Class 3. The condensate formed during evaporation can be used to deslurry and wash the sedimentary AHLW part.

Option No. 3 accounts for a cost-effective processing scenario. This flowchart provides for sorption or extraction separation of cesium radionuclides from the pre-filtered solution part and the subsequent emplacement of treated liquids in one of the industrial reservoirs operated at the Mayak PA site (V-9 or V-17) (Fig. 5). Under this option, preliminary filtration is thought not only to remove most of alpha-emitting radionuclides associated with suspensions and colloids, but rather to provide stable hydrodynamic sorption or extraction treatment modes.

As it comes to potential disadvantages associated with the third option, one may note the uncertainty in the behavior of the chemical waste components (for example, pore clogging or desorbing effect produced on the radionuclides that have been already settled down in the rocks) when discharged into a specially designed industrial reservoir (SDIR).

To evaluate the prospects for the safe implementation of this option, in 2023—2024, Federal State Budgetary Institution Gidrospetsgeologiya and Mayak PA launched a joint project providing for the experimental simulation of the LRW solution part and its discharges into the specified reservoirs. These efforts include:

1. Modelling a source of groundwater contamination and establishing a threshold discharge volume based on calculation and experimental studies of reservoirs V-17 and V-9 and their water-chemical state.

2. Experimental study of the geological massif in potential groundwater discharge areas and their water-conducting properties.

3. Updating the model of reservoir V-17 and the regional model of reservoir V-9, predictive calculations of the contaminant spread with groundwater from each reservoir.

To implement sorption treatment, resorcinol-formaldehyde resins selective for cesium in highly alkaline media or the ferrocyanide sorbent Fersal can be used [8], [9]. In the former case, the process may be carried out in columns using a dynamic sorption mode, in the latter one — the use of either dynamic or static sorption is possible. As it comes to cesium, the solution treatment factor observed in the studies ranged from two to four orders of magnitude.

The volume of the spent sorbent accounted for approximately 1 % of the treated LRW volume. After its single or multiple use, cesium is ultimately desorbed both to reduce the RW category to Class 3 and to decrease the radiation and thermal load on the sorbent material. For ease of handling, the sorption unit may be designed as a module emplaced into an NZK type container with the internal voids filled with bentonite clay, which at the post-desorption stage should be emptied by vacuuming, disconnected from the connection lines, sealed and handed over for storage/disposal. In this case, some increase in the volume of the packed sorbent can be certainly observed due to packaging. Commonly used sorption columns can be applied as well provided that the sorbent is unloaded from them at the end of their service life and then immobilized into cement compound together with the treated solution. Strippants, regenerates and wash solutions, which in terms of the corresponding macro-components mainly contain only the nitric acid and practically only cesium, if talking of the radionuclide inventory, are added to other HLW streams subject to vitrification with almost no impact on the volume of the final product.

Cesium separation by extraction, which is viewed as an alternative to sorption, is carried out using a mixture based on para-isononylcalix[6]arene (IN6) [10]. The degree of cesium removal from the solution observed in the studies accounted for about two orders of magnitude. Centrifugal extractors were proposed for consideration as possible equipment under this process. To test the design elements and process modes, in 2022—2023, RPC Sosny developed the designs and manufactured a two-stage prototype unit, which was expected to complete its testing by Mayak employees at the end of 2024.

Handling of re-extracts appears to be similar to the previously described approach applied in case of strippants and regenerates. To recycle the spent organic matter, steam distillation and subsequent solidification of the still residue by cementation or vitrification methods were proposed for consideration. This approach generally corresponds to the one contemplated by the enterprise for other spent extractant streams.

Option No. 4 is viewed as a combination of options No. 2 and No. 3 providing for sorption or extraction

separation of cesium radionuclides from the solution part with subsequent evaporation and cementation of the treated solutions (Fig. 6). This scenario, compared to the others, provides no technical and economic advantages, but is viewed as the most environmentally friendly one that fully complies with the regulatory requirements for the safe RW management.

Decision on a specific AHLW processing method can be made based on a feasibility study of proposed options taking into account relevant sociopolitical factors.

The below approaches are being considered as processing options for the sedimentary part.

At present, the designs of a new vitrification complex at Mayak PA provide for a method enabling sedimentary part extraction via slurry lifting devices developed by RPC Sosny (in 2022, a prototype was manufactured and successfully tested on simulation systems). The suspension is collected through existing small-diameter service openings arranged at different points of the tank slab. Owning to this approach, the mobile part of the sediment can be smoothly extracted from the tanks. To remove its compacted part, washing is required: the washing liquid is supplied and, due to chemical and hydrodynamic impact, it peptizes the clay-like sedimentary material to enable its transportation through pipelines. At present time, water (demineralized condensate) is considered as a most appropriate washing liquid option, which, as experiments on real sediment have shown, may effectively remove soluble components from it. The sediment can be largely washed from both soluble chemical macro-components, such as sodium salts, and a number of radionuclides (cesium, etc.).

After such treatment, the sediment loses a significant proportion of heat-generating radionuclides, thereby, enabling its collection from several containers and temporary storage in so-called compacted mode until it is handed over for processing and vitrification.

Thus, when handling dense sediments, the washing stage may solve three problems:

- ensure their mobility, and therefore their extraction from the storage tank;
- reduce the amount of soluble macro-components in them, which may potentially decrease the vitrified HLW volume;
- reduce the activity and heat release of the sediment enabling its temporary storage in a more compact volume in a compact manner, until necessary vitrification capacities are commissioned (construction of high-performance furnaces).

Due to this approach, even before the furnaces with the required capacity are commissioned, several storage tanks can be freed up and used as backup ones in case if the filled ones lose their integrity.

To address the case when one fails to perform complete removal of all sediment from most inaccessible areas of the tanks (assuming reasonable operating and time costs), a joint project was launched under an agreement signed with the State Corporation Rosatom: in 2024—2025, experts from Mayak PA and the Nuclear Safety Institute will focus on a design-basis justification of the in-situ disposal options proposed for the sedimentary HLW part of complex chemical composition (i. e. in-situ disposal in storage tanks), which is supposed to change the formal status of the facilities into facilities holding non-retrievable (special) RW.

The industrial waters resulted from washing are filtered to prevent the entrainment of sediment with long-lived alpha-emitting nuclides. After that, they are processed according to one of the four handling options proposed for the solution HLW part. The need to consider the option with washing is corroborated by the fact that a similar approach was applied for disposal of the sediment part of waste at the Hanford WTP plant.

As regards further handling of the sediment, which contains, in particular, long-lived radionuclides, three possible processing options are being considered (Fig. 7):

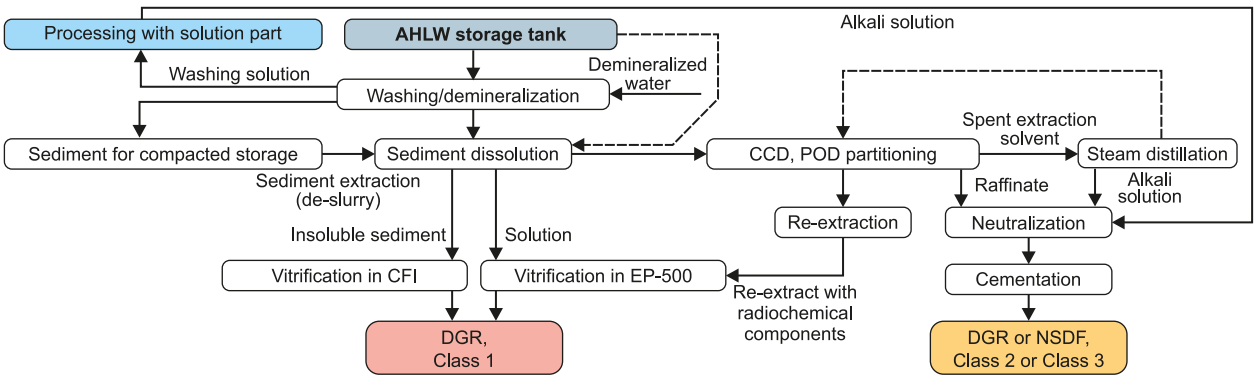

Fig. 7. Processing flow chart for the sedimentary waste part

1) the basic option involves their dissolution (with nitric acid or acid mixtures) and vitrification in direct electric heating furnaces (in EP-250/6 that is currently under construction, or in furnaces EP/7 and EP/8 of the New Vitrification Complex that are at the design development stage),

2) vitrification of the washed sediment suspension in a cold crucible induction furnace (CFI). Undissolved residues from the dissolution stage can be also potentially subject to vitrification in this way. Thanks to CFI designs, the highly concentrated suspensions can be fed into it, unlike EP furnaces, but the CFI, in turn, has a much lower capacity, thus, it's impossible to use it as the main unit for direct AHLW sediment vitrification,

3) an alternative option involves acidic dissolution of the sediment with subsequent partitioning of radionuclides by extraction methods using, for example, heteroradical phosphine oxide (POD) and chlorinated cobalt dicarbollide (CCD) as extractants. The radionuclides separated in this way (primarily transuranic elements, cesium and strontium) are vitrified to produce RW Class 1, and the treated nitric acid solution accounting for the biggest inventory of chemical components is subject to cementation. Since only a neutral or alkaline solution can be cemented, it must be treated with a reagent, such as, treated alkaline decantates and liquid resulted from AHLW sediment washing. Depending on the treatment efficiency achieved in these operations, the solidified solution can be categorized either as RW Class 2 or 3.

The first and second options result in generation of large amounts of AHLW Class 1 if the glass-like compound complies with the existing standards set forth under the process regulations for vitrification that impose restrictions on the contents of waste components (Table 1). The specified restrictions are supposed to provide compliance of the generated AHLW with the requirements for the glass-like compound set forth in the NP-019-2015 [11], and also to ensure acceptable performance of the vitrification unit in terms of maintaining the necessary thermoviscosimetric properties of the melt to enable its controlled discharge into cans and acceptable corrosion activity with respect to refractory and electrode materials. A few experiments exploring the prospects for increasing the specified concentration limits as regards waste component inclusion into glass have yielded some encouraging results [12]—[14]. However, yet some systematic research is required to address this problem.

The third option is supposed to reduce the volume of vitrified HLW (Class 1 RW) in a quite considerable way, thereby, providing considerable reduction in the required capacity of electric furnaces designed to process the HLW. A preliminary feasibility study of this option launched by Mayak PA on model systems shows that transuranic elements can be removed from the dissolved sediment with a treatment factor of over two orders of magnitude.

Table 1. Regulatory restrictions imposed on the content of AHLW components, wt. %

Component	Content
Al_2O_3	14÷18
Al_2O_3+ Me_2O_x	19÷23
Na_2O+ Me_2O	22÷26
P_2O_5	49÷55
B_2O_3	6.0
Fe_2O_3	1.6
Cr_2O_3	0.4
NiO	0.3
CaO	1.0
MgO	2.5
MnO_2	0.09
SO_3	0.42
U_3O_8	5.0
PuO_2	0.034
MoO_3	1.8

Conclusion

The decision in favor of a particular management option for the solution and sedimentary HLW parts will largely depend on the findings of the feasibility study. At the initial stage, the preliminary feasibility study was focused on the most elaborated options proposed to implement the flowchart. At the same time, considered of paramount importance in these assessments were the fees associated with the disposal of conditioned RW of the corresponding classes, as well as the cost of consumables (sorbents, extractants, dry mixture, containers, etc.)

Depending on the design option, the estimated cost required to process the entire volume of the solution AHLW part was found to be ranging from 0.6 to 12.7 billion rubles. Such wide cost variability indicates the advisability of choosing the optimal option.

Approximately 50.6 billion rubles will be required to process the entire volume of the sedimentary AHLW part (according to the basic option, in accordance with the existing standards regulating the content of components in the HLW established in the process regulations for the vitrification electric furnaces). However, it is still difficult to evaluate

other options today, which is due to insufficient experimental data both as regards the initial characteristics of the sedimentary part and the resulting secondary LRW streams. However, such high cost of the basic option also indicates that the optimal solution should be selected and in particular with due account of the implementation costs.

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