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BASIC APPROACHES, EXPERIENCE AND PROBLEMS RELATED TO REPROCESSING OF LIQUID RADIOACTIVE WASTE OF COMPLEX CHEMICAL COMPOSITION ACCUMULATED IN STORAGE TANKS

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The paper overviews a concept developed to manage high-level heterogeneous waste accumulated at radiochemical plant due to the defense program implementation. The paper presents flow chart describing relevant processing method, the main stages of its development and the expected results of its implementation.

Keywords: *high-level waste, storage tanks, neutralization, filtration, dissolution, precipitation, sorption, extraction, vitrification, cementation.*

Introduction

Large amounts of heterogeneous high-level waste generated by defense facilities designed for radiochemical separation of weapons grade plutonium have been accumulated at PA Mayak site. To date, after being stored for long time and after certain measures implemented to ensure safe operation of the storage tanks, this waste is composed of weakly alkaline and alkaline sediments, suspensions and clarified solutions. Basic sediment components are iron, aluminum, nickel, chromium hydroxides, iron and nickel sulfides, nickel-cesium and cesium-titanium ferrocyanides. High-salt solutions form the liquid phase with such key chemical components as hydroxide, aluminate and sodium nitrate. Radionuclide composition i. e. fission product composition differs for the clarified phase and for the sediment. Radioactivity of the liquid phase is mainly due to ^{137}Cs. As for the sediment, it also contains large amount of ^{90}Sr (in equilibrium with ^{90}Y), as well as fissile components — uranium and plutonium.

Storage facility designed for this type of waste features 14 concrete storage tanks lined with stainless steel and a capacity of 1,170 m^3 each. These storage tanks have been actively used for heterogeneous high-level waste (HLW) emplacement in 1968—1986. Figure 1 presents layout of the storage facility.

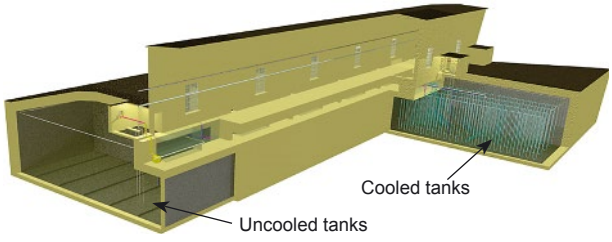


Figure 1. Storage facility for accumulated HLW

The tanks were filled up under the following mode: "acceptance — settling-decantation". As a result, most part of fission products and actinides was contained in the sediments, and intermediate-level clarified waste was discharged into Lake Karachay [1].

In 1986, its storage capacity was exhausted and it was closed in 1986. Table 1 overviews some characteristics of HLW accumulated in the storage tanks [2, 3].

Table 1. Characteristics of HLW held in the storage tanks

Storage-tank	HLW amount, m^3	Sediment layer thickness, m	Activity, kCi (at the time of emplacement)
1	1,057	4.7	1,128
2	1,065	3.2	1,105
3	1,000	0.6	4,638
4	1,041	1.40	4,974
5	886	1.1	4,885
6	899	0.9	4,988
7	967	0.9	4,816
8	980	1.0	4,998
9	1,134	2.9	6,937
10	1,150	3.1	6,636
11	1,137	3.1	7,044
12	1,123	3.3	6,988
13	1,055	4.4	7,071
14	1,070	4.4	8,129
Total	14,564	–	74,327

By late 1960's, when construction of the first storage facility section was completed, the challenges associated with radioactive waste management have been already studied relatively well. However, most of the storage tanks were not fitted with a cooling system, thus, maintaining safe temperature mode was rather complicated task to implement during their operation. By the time the tanks had reached their full capacity, the thickness of the dense sediment layer amounted to some 5 m. Thus, with large amounts of radionuclides accumulated and hindered transfer of heat generated by their decay this resulted in uncontrolled heating of the sludge. Temperature at the bottom of uncooled tanks of the first section exceeded 100 °C requiring urgent measures to be implemented. Thus, recurrent treatment of tank sediments with a concentrated solution of sodium hydroxide was launched in 1987 — as the result, significant part of the solid phase (aluminum hydroxide and part of ferrocyanides) was dissolved. Eventually, by 1994 temperature was reduced and stabilized at a safe level in most of the tanks [4].

At the same time, long-term operation of the storage facility has caused aging, corrosion and degradation of its structural elements, thus, increasing the risk of potential accidents primary associated with liquid radioactive waste (LRW) leaks due to the loss of the tanks sealing capacity. Moreover, liquid HLW long-term storage method contradicts the present-day LRW management concept suggesting that the waste should be immobilized into a stable matrix form (glass and/or cement). For this reason, further storage of high-level sludge can not be considered as an acceptable solution in terms of the long-term radiation safety. The tanks should be emptied and dismantled in the near future.

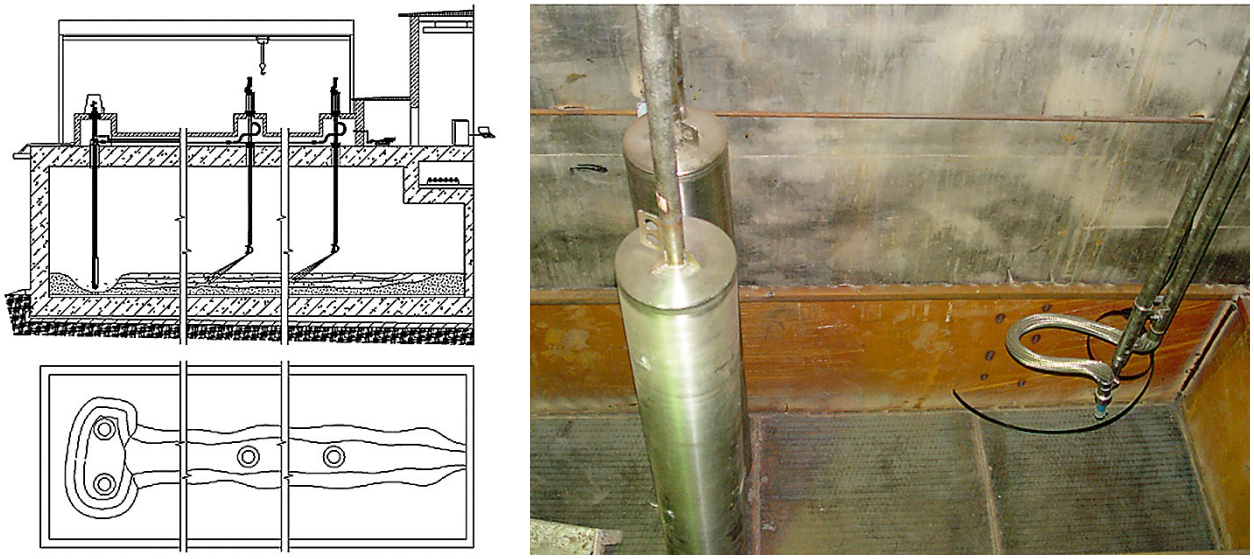
Absence of available systems enabling the sediment washing and retrieval is considered as an obstacle impeding this process. Moreover, sampling of the sediments has shown that some of the tanks contain in their bottom part some dense layer of low-mobile viscous hard-to-retrieve sediment. This necessitates the development and application of purpose-designed washing and sludge retrieval tools. Such tools can be installed only upon violating tanks integrity — by means of slab panels perforation. Given high radiation exposure conditions and absence of reliable data on the state of structure materials (concrete), this task is viewed as a quite complex one from engineering and technical perspective.

Following sludge and waste retrieval it should be immobilized providing safety of its further storage and disposal. Immediately prior the immobilization stage, the waste should be treated to minimize the volume of the solid product.

Emptying the storage tanks

An R&D program titled Development of a Processing Technology for the Accumulated Liquid Radioactive Waste and Relevant Equipment at FSUE PA Mayak [2, 3] was developed under the Federal Target Program Nuclear and Radiation Safety in 2008—2015. The purpose of the program was to empty PA Mayak's storage tanks and to treat the accumulated HLW generated in the past as the result of the defense program implementation converting it into stable matrix form.

The program was sponsored by the State Corporation Rosatom with FSUE PA Mayak acting as its contactor. A number of Russian industrial, scientific and engineering organizations also took part in this project, namely, MCC, JSC VNIINM, FSUE NPO RI, MosNPO "Radon", JSC "SverdNIIHimmash" and other.



a *b*

Figure 2. Layout of a storage tank (a) and (b) sludge retrieval facility

As a result of the program implementation, designs of test sludge retrieval installation were developed. The equipment was mounted and debugged with the engagement of FSUE MCC experts, a design-development organization having many years of experience in operating sludge retrieval equipment.

Following storage tank slab perforation, a purpose designed unit will be fitted inside the tanks containing the equipment. Figure 2a shows the way how the sludge-retrieval equipment should be installed inside storage tanks, whereas, Figure 2b shows part of the basic equipment fitted inside a radiochemical canyon.

The left side of the figure depicts the washing tool the key structure elements of which are as follows: a nozzle with a rotation lead moving both in horizontal and vertical planes equipped with a control system (fitted outside the canyon) providing manual and automatic modes of operation. Pulsating mixing device and pulsating pump designed to maintain the suspended state of the medium and to pump the suspension into another container or to supply the solution to the washing device are located to the left, closer to the wall. Purpose designed video surveillance system will be used to monitor the conditions inside the tank during waste retrieval and the configuration of the bottom sediments. Clarified HLW phase from the same tanks or salt-free condensate were purposed as a washing fluid. In 2010-2011, the abovementioned equipment was successfully tested at purpose designed model systems by FSUE PA Mayak experts.

A number of important disadvantages being specific for this method exist, namely:

- Large size of submersible equipment (pulsating pump and washing unit) not allowing its installation using already available access holes (penetrations) being relatively small;
- Integrity of storage tank will be impaired, thus, reducing its load bearing capacity;
- Increased load on the slab structure due a large number of additional equipment and biological protection (tens of tons);
- Current state of the building structures should be evaluated and the stability of the tanks under simultaneous impact of the factors indicated, as well as the implementation of correcting measures (strengthening of the structure, load distribution, etc.) should be demonstrated being considered as a rather technically challenging and costly task to accomplish.

Already existing access holes (penetrations), for example, those for monitoring measuring equipment were proposed for use by PA Mayak experts as an alternative option enabling the retrieval of clarified phase and mobile part of sludge-type high-level waste [5]. After the equipment is removed, a pipe is installed to the required depth in the hole and driven by an external stimulator (for example, inducing vacuum in the pipe), the liquid high-level waste is pumped out of the tank. Newly installed pipelines extending beyond the existing storage tanks' biological protection circuit are shielded locally by easily mounted biological protection to prevent excess personnel exposure (Figure 3).

Thanks to small diameter of the standpipe and newly installed pipeline system additional biological shielding is rather minute in its volume

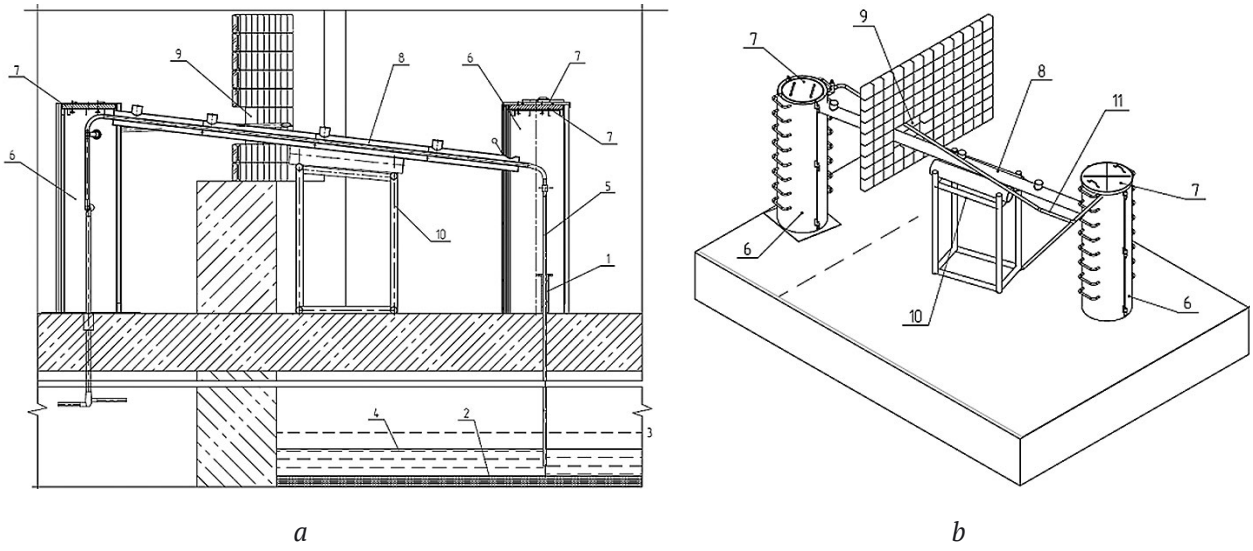


Figure 3. General layout of storage tank emptying system (a) and biological shielding for the LRW discharging pipeline segment located outside the tank (b))

1 — access hole for thermometer, 2 — layer of dense sediment at the bottom, 3 — tank decantation level, 4 — mobile LRW portion, 5 — pipeline outside the existing biological shielding system, 6 — cylindrical detachable protective casing, 7 — casing cover, 8 — protective cylindrical tray, 9 — opening in the wall, 10 — supporting structure, 11 — water supplying line for pipeline flushing

(45—55 mm thick lead layer) and its mass, thus, tank can be emptied at a minimal cost without special evaluation of building structures state and necessary structure reinforcement. Accumulation of retrieved liquid HLW in interim tank with their further discharge into reservoir-storage on the surface of the sediment layer allows its main part to be washed out by repeated circulation of the solution. The layer of the retrieved sediment accumulated in the bottom part of the interim tank is routinely handed over for further HLW processing stages. Use of liquid HLW as a medium for sludge washing enables to minimize the volume of secondary liquid waste generated during storage tank emptying operations. For additional dissolution of most dense sediment layers, low-salinity condensates can be used at the final processing stage.

To date, the developed emptying system enabled to retrieve over 110 m^3 of liquid HLW settled below the standard decantation level.

Low-mobile sediments that were not retrieved by this method will be removed by a submersible washing and sludge retrieval equipment or (if this equipment cannot be installed on the slab) specific decisions will be made to immobilize the sediment directly within the tank.

Pre-solidification HLW treatment method

Initially, when the accumulated HLW processing technology was being developed, it was proposed to solidify the retrieved waste directly (together with the liquid phase and sediments) converting it into borosilicate glass by applying two phase vitrification process. This process was supposed to be implemented using a calciner or a rotary film evaporator for LRW drying/concentrating at the first stage with the second stage involving a cold crucible type melter. However, during further development of the technology and the equipment, the processing concept has undergone some significant changes. Research carried out in 2008—2011 under the abovementioned program has shown that direct solidification of accumulated HLW into borosilicate glass could result in a huge amount of vitrified waste — over 2,000 tones per 1,000 m^3 of HLW due to the restrictions imposed on the contents of some critically important components inside the glass (for example, sulfates, chromium) and other factors. Thus, from 1987 to 2010, some 6,200 tons of glass were generated by the vitrification complex available at PA Mayak. Obviously, this approach cannot be seen as an acceptable one to address the challenge of accumulated HLW processing [2, 3].

HLW processing concept developed by the authors (based on research implemented by the Radium Institute and PA Mayak) suggests the use of waste fractioning method, i. e. segregation of initial HLW into a number of waste streams (HLW and ILW) with their further processing at vitrification units equipped with direct electric heating melter, cementation complex, as well as a vitrification unit with a cold crucible type melter. The latter one applied to treat most complex sediments (Figure 4). Technical implementation of the concept will enable to reduce the amount of vitrified waste by several times, and, what is more important, to empty the tanks within the foreseeable future [6].

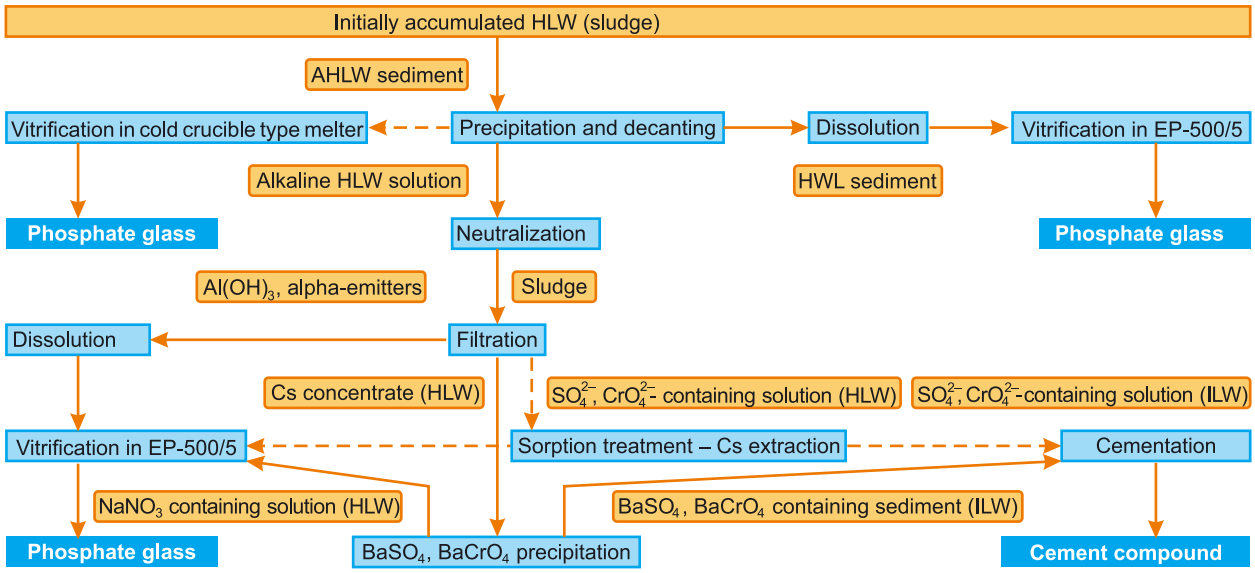


Figure 4. Process flow chart for accumulated HLW pre-solidification treatment with waste stream segregation

The following methods can be used to decrease the amount of vitrified HLW:

- use of aluminum phosphate glass for HLW solidification, for which, as compared with borosilicate glass, the restrictions on maximum allowable content of sulphates are substantially less strict (potentially up to 3% by weight instead of 1% by weight);
- substantial part of waste categorized as ILW with further solidification by the cementation method using high-capacity complex currently being developed at the enterprise;
- segregation of sulfate ions and chromium from HLW into a separate waste stream (streams).

Accumulated HLW retrieved from storage tanks (solution and sediments) are segregated into solid and liquid phase by settling and decanting of the clarified solution. Bottom sediments can be either sent for solidification in a cold crucible type melter or for dissolution in nitric acid (mixture of nitric and orthophosphoric acids) followed vitrification using an electric direct heating furnace. Alkaline clarified solution is transferred for further processing.

Neutralization was proposed as the first stage of the clarified solution processing suggesting the use of concentrated nitric acid [7, 8]. Resulting aluminum hydroxide sediment layer (with alpha-emitting nuclides fixed on it) is washed using membrane filters enabling to remove sulfates and chromates from it. The sediment gets denser [9] and after dissolution is handed over to EP-500/5 furnace as a fluxing (for aluminum) additive. This enables to substitute (save) up to 700 tons of reagent aluminum nitrate. Washing solutions are discharged to the clarified solution.

Further on, in keeping with the first processing option, if high contents of sulphates and chromates in the clarified solution is detected the solution with a high content of sodium nitrate is treated (precipitation of relevant low soluble barium compounds). Then it is handed over to the fluxing unit (by sodium) enabling the vitrification of both newly generated and accumulated HLW in electric direct heating furnace. Barium sulfate and chromate precipitates are handed over together with ILW to the cementation unit (the flow chart of the complex being currently developed does not allow for separate solidification of solutions and sludge).

In keeping with the second option, the clarified solution after most part of cesium being already extracted by sorption method (using ferrocyanide sorbents, for example at FS-10 [10]) is handed over to the evaporation unit. Cesium desorbate is sent to the vitrification unit — electric direct heating furnace. The resulting sodium containing bottom residues is handed over to the cementation unit, since, following two relevant treatment stages this solution can be solidified by applying the method indicated given the specific activity of alpha and beta-emitting nuclides. Sulphate and chromate ions present in the solution produce no significant impact on the cementation process and the quality of the resulting compound. This option can be implemented if vitrification process applied to treat the main HLW steam generated from current SNF reprocessing is temporary suspended, as well as in parallel with the first option. The possibility of redistributing high-salt sodium-containing decantate stream between the two solidification units will also enable prompt correction of HLW composition sent to the vitrification unit based on the process specifications allowing to maintain the ratios of the main glass-forming components — sodium and aluminum.

The flow chart presented below describing the management of retrieved high-level suspended mater is viewed as a basic one which has been reflected in relevant provisions of design documentation developed for a pilot-industrial unit under a state contract implemented by PA Mayak. Under this project, some calculations to evaluate the amount of solidified RW resulting from HLW processing and retrieved from one storage tank (with an averaged composition) have been carried out. Data on the amount of borosilicate and aluminophosphate glass resulting from direct HLW solidification is presented in Table 2.

Table 2. Glass mass resulting from direct vitrification of an entire tank

Matrix type	Glass from decantate, t	Glass from sediment, t	Total glass mass, t
Borosilicate glass	920	1,060	1,980
Phosphate glass	572	184	756

Table 3 presents data on the amount of vitrified and cemented waste resulted from HLW solidification suggesting waste segregation into streams based on one of flow chart options described above — handling over the high-salt sodium-containing stream to the cementation unit.

Table 3. Glass and cement mass resulting from stream segregation

Matrix type	Glass from decantate, t	Glass from sediment, t	Total glass mass, t
Borosilicate glass	117	1,060	1,177
Phosphate glass	64	354	418
Cement	1,960	–	–

It should be noted that in this case the aluminum extracted from clarified HLW phase acts as a fluxing additive introduced during aluminophosphate glass melting. Meanwhile, the activity of radionuclides from this stream present in the glass is small enough compared with the opportunities for their inclusion into glass matrix (10—15%). Thus, the resulting glass can be successfully used to vitrify other waste groups both currently generated and the accumulated ones. Aluminium extracted from the alkaline decantate in this case should be considered as a substitute for reagent aluminum.

Detailed examination of this issue may result in a statement suggesting that cement compound is the only product of accumulated HLW processing (cleared phase).

The above data indicates that the implementation of the suggested flow chart with stream segregation results in almost fivefold gain in glass mass compared to direct vitrification with borosilicate glass, and almost twofold as compared to direct vitrification with phosphate glass.

At the same time given the variety of accumulated HLW contents, as well as difficulties associated with obtaining reliable data on their characteristics, PA Mayak experts working jointly with a number of scientific and research organizations (IFKhE RAN, DVO Chemical Institute of RAN, Radium Institute and other) study additional engineering solutions associated with pre-solidification treatment of retrieved waste (both sediments and cleared phase).

As for high-level waste management technology, an option is considered suggesting solidification of its insoluble part (as well as sediments the contents of critical components in which allows their processing using joule heating melter unit) in cold crucible type melter producing aluminophosphate glass. Another option considered suggests that radionuclides are extracted from dissolved sediments by applying sorption or extraction methods with further vitrification of the concentrate and cementation of treated (to ILW level) solutions containing the main portion of stable elements considered as "hard to be vitrified" (Fe, Cr, Ni, S and etc.) [11].

The following flow chart options are being developed to enable pre-solidification treatment of the clarified HLW phase:

1) removal of alpha-emitting nuclides by ultrafiltration method with further extraction of cesium directly from an alkaline solution using, for example, recoverable resorcinol-formaldehyde resins [7, 12] or nonrecoverable inorganic ion exchangers [13];
2) cesium and alpha emitters extraction from an alkaline solution using calixarenes mixed with extraction solvents of other classes [14, 15].

The above approaches, if implemented successfully, may help to change over the category of the LRW into ILW, thus enabling its immobilization using the currently developed high-capacity cementation complex.

Upgrading of particular processing stages is also considered as an option, for example, instead of nitric acid for clarified phase neutralization carbon dioxide bubbling (carbonization) process may be applied [7, 16]. This approach was originally proposed by experts from the Radium Institute, worked out by PA Mayak experts. It was noted that this option has a number of advantages over the acidic one depending on the overall setup of clarified phase pre-treatment process.

Sorption may be also applied at the stage of sulphate and chromium ion removal from the clarified phase by using barium calcium mixed silicate-based

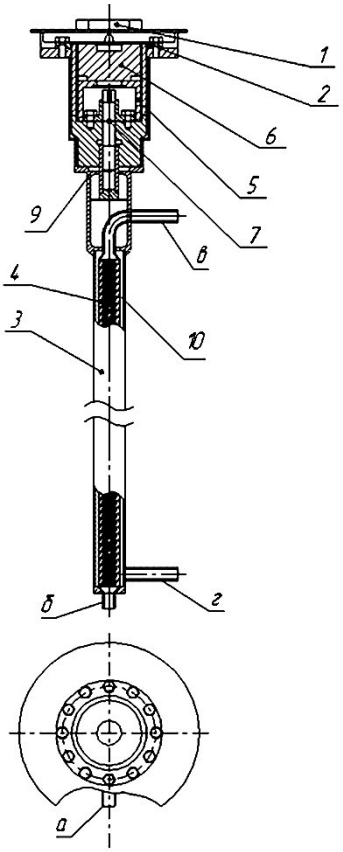
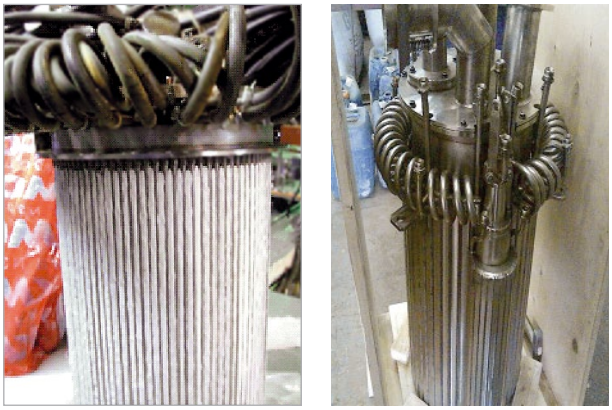


Figure 5. Layout of the filtration module

a — air inlet, b — initial suspension inlet, c — concentrated suspension outlet, g — filtrate output unit, 1 — lid, 2&5 — inserts, 3 — case, 4 — casing with filtering element, 6 — schield, 7 — screw, 9&10 — cavities

treating agents. The use of sorption columns is considered more feasible as compared to the sedimentation process implying further segregation of solid and liquid phases.

Remotely controlled device for liquid radioactive waste filtering applied at membrane separation stages was developed and patented (figure 5) [17]. Its design enables to implement the entire working cycle in a fully remote mode, including installation and disassembly stages. It's operated based on flow-through (tangential) filtration involving no accumulation of contaminating components (solid phase, colloids) on the filter surface, as they are washed away by original solution flow moving parallelly to the membrane surface. As the flow moves along the filter, the initial suspension stream is concentrated. Multichannel (19 channels) ceramic γ-Al_2O_3 modules with TiO_2 (or ZrO_2)-based filtering layer applied on the inner surface of the channels are used as filtering elements. Filtering layer pore size accounts from over 10 nm (for ultrafiltration elements) to up to 300 nm (for microfiltration). Both filters produced abroad (MEMBRAFLOW control systems GmbH) and in Russia (NPO Keramikfilter) can be used.



a *b*

Figure 6. Cold crucible type melters designed by FSUE MosNPO Radon (a) and JSC VNIINM (b) respectively

Processing of retrieved HLW after its prior treatment will be performed jointly together with other waste streams from radiochemical production units. For this purpose, EP-type electric furnace and cementation complex facilities are going to be used. The only exception can be a small amount of so-called complex sediments the vitrification of which requires the use of an induction melter (IPCP) only producing aluminophosphate glass. Separate chambers have been considered in the designs of the newly developed vitrification complex allowing to emplace such melters [18]. To date, no operating IPCP type facilities with extensive operational experience in treating actual HLW at radiochemical production units (allowing entirely remote operation supported by relevant peripheral devices complex) exist. Crucibles developed by Moscow Radon and VNIINM OJSC and considered as prototypes of such a melter were tested using a purpose designed stand at PA Mayak (Figure 6).

The tested "cold crucibles" are cylindrical structures composed of water-cooled elements with insulating inserts. HLW is continuously supplied to the unit, melt discharge is carried out either continuously or periodically. The VNIINM's inductor operating frequency accounted for (0.90 ± 0.01) MHz, the one developed by FSUE MosNPO "Radon" — (1.76 ± 0.44) MHz. Relatively low performance of the crucibles in terms of glass production (15—25 kg/h) will enable to vitrify some part of the overall flow of accumulated HLW with chemical and phase composition being considered as most hard-to-treat.

It should be noted that both tested structures have a common structural disadvantage —absence of bottom melt drain potentially causing accumulation of some fractions having low solubility in the melt and increased density (for example, noble metals, spinels). These can get accumulated inside

the void volume of the units impeding glass melting process.

Currently it's believed rather difficult to estimate the performance of the proposed AHLW processing complex as, firstly, no experience on washing dense bottom sediments in HLW storage facilities exits to date. Secondly, this process should involve installations the main task of which will be solidification of LRW currently generated by radiochemical productions. Based on assessment calculations performed to date it was assumed that average rate of storage draining and AHLW processing will account for 1 storage unit (over 1,000 m^3) per 3 years. The indicated time-frame can be decreased if relevant know-how and experience is gained in storage unit draining and bottom sediment removal, as well as pre-solidification treatment of retrieved waste with continuous optimization of each flow chart stage and commissioning of additional units.

It can be stated unequivocally that the proposed concept suggesting segregation (fractioning) of accumulated waste with solidification of the final streams into aluminophosphate glass and cement compound will reduce the amount of vitrified HLW by 3—5 times compared to the originally proposed direct vitrification option with borosilicate glass generated.

After the government contract completion in 2011, development and improvement of particular AHLW processing stages and of the process flow chart on the whole was continued by PA Mayak. Table 4 present current state of development as it comes to particular stages of AHLW pre-solidification treatment process.

Table 4. Current development status for individual AHLW pre-solidification treatment stages

Stage	Laboratory testing		Experimental testing	
	Models	Actual product	Models	Actual product
Sediment washing by sludge lifting equipment	-	-	Successfully	-
Sampling LRW at the bottom level using method [5]	-	-	-	Successfully
Bottom sediment dissolution	Successfully	Successfully	-	-
Decantate neutralization by HNO_3	Successfully [8]	Successfully	Successfully	Successfully
Decantate neutralization by CO_2	Successfully [16]	-	-	-
Thickening of hydroxide sludge by filtration	Successfully [9]	Controversial results	Successfully	Successfully
Removal of sulphates and chromates	Successfully [19]	Controversial results	-	Controversial results
Cs sorption by FS-10	Successfully [10]	Successfully [10]	-	Successful practice using similar products at PA Mayak
Cs sorption by resorcinol formaldehyde resins	Successfully [12]	Successfully	-	Successful practice using similar products at Savannah River site
Cs sorption by inorganic ionite manufactured abroad	Successfully	Successfully [13]	-	Successfully
Extraction with calixarenes	Successfully [14]	Successfully [15]	-	-

It should be particularly noted that almost all flow chart options involve similar elements linked in different sequence. From this perspective, development of a pilot-industrial unit based on the most extensive flow chart with further testing and upgrading with actual solutions being applied at all RW management stages is considered as the most feasible option. The abovementioned approach will enable joint implementation of two processes – to start processing of actual waste and to gain lacking information needed for the development of an industrial facility. It should be also noted that given the lack of reliable data on the properties of accumulated HLW (chemical and radiochemical composition, physical and chemical specific properties of the sediment) availability of a flexible multi-functional flow chart enabling waste pre-solidification treatment can be viewed as an urgent need.

To test and commission the new pilot-industrial technology under FTP NRS, it was originally expected to develop pilot-industrial facilities to empty the storage tanks and implement pre-solidification treatment of the accumulated HLW. In keeping with initial operational schedule [2], these facilities were to be commissioned in late 2015. However, due to federal budget cutting they were not constructed. Upgrading and retrofitting of individual devices and installations for pilot-industrial operations with liquid HLW (retrieval from the depth, neutralization, filtration) was carried out at the site.

However, as particular aspects of certain operations have not been accounted for, the listed equipment can not function in an optimal mode. Current operation is believed to be too time consuming and in a number of cases does not provide the best performance indicators. Thus, development of a purpose designed pilot-industrial facility enabling HLW pre-solidification treatment is still an urgent task to be addressed.

Conclusion

In 2018, a contract on processing the first batch of accumulated HLW (over 60 m^3) was signed between PA Mayak and the State Corporation Rostom suggesting the implementation of already developed process flow chart to demonstrate the technical capabilities of the enterprise in this field. Full-scale testing of the technology can be carried out following ILW cementation complex commissioning, i. e. not earlier than 2019.

The technology developed will enable to empty all 14 storage tanks with accumulated HLW generating some 5,000—6,000 tons of glass within an acceptable timeframe. At the same time, efficient segregation of RW by solidification methods (cementation and vitrification) is ensured by the proposed technology, thus, minimizing the amount of vitrified HLW and decreasing, in the long-run, further costs associated with vitrified waste storage and transfer to FSUE NO RAO for disposal.

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