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PURPOSES OF BITUMINOUS COMPOUNDS AND REQUIREMENTS FOR THEIR DISPOSAL

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The article summarizes the study of bituminous radioactive waste properties, including the results of long-term tests performed under conditions similar to those at a near-surface disposal facility. It investigates the compliance of their characteristics with the requirements of federal rules and regulations in the field of atomic energy use specified for disposal purposes.

Key words: *nuclear power plants, liquid radioactive waste, bituminization, bituminous compound, bituminous compound disposal.*

At present time, rather intensively discussed are the opportunities and the requirements on the safe disposal of bituminous compounds as secondary products of liquid radioactive waste (LRW) processing via bituminization. The main issue discussed is the potential fire hazard and, under certain conditions, potential explosion hazard of this product.

LRW bituminization method was developed in 1960's and has been applied in many countries: France, Belgium, Germany, USSR, the United Kingdom, Japan and other. It involves LRW evaporation followed by mixing of the formed salts with molten bitumen. The product generated (bituminous compound) being in a flowable state is discharged into a forming package or via a heated bitumen line transferred to special storage facilities.

In 1964, first batch-operated industrial unit of this type was developed in Mol (Belgium).

The reasons for quite wide application of this technology are believed to be as follows:

- simple instrumentation and process implementation;
- high versatility, the possibility of processing heterogeneous LRW (with high content of suspensions, ion exchange resins and filter perlite powder) of various chemical composition and mineralization level;
- more then 2 time decease in the amount of reprocessing products as compared to earlier developed cementation method;
- high water resistance capacity of the bituminous compound.

In the USSR, studies of bituminization method application for LRW reprocessing purposes were initiated in 1970's. Scientific support during the implementation of this project was provided by All-Union Scientific Research Institute of Inorganic Materials named after the Member of the Academy of Sciences A.A. Bochvar (VNIINM named after A.A. Bochvar). For the first time, pilot-industrial operations on bituminization were implemented in the USSR at the Central Radiation Safety Station in Moscow (TsSRB, later on MosNPO "Radon"). At this site were developed and operated some three types of bituminization installations: BO-75 (boiler type), 1970; UBD-200 (two-stage bituminization with an extruder mixer), 1974; URB-8 (installation with a rotary film evaporator), 1976. Processed were the bottom residues generated following LRW evaporation at the Moscow treatment station. The first industrial bituminization facility was commissioned at Leningrad NPP in 1984, then this technology was implemented at Kalinin NPP.

Main results of bituminous compound properties study

Design development and introduction of bituminization process to LRW processing flow chart at NPPs was preceded by quite lengthy scientific research that involved the selection of bitumen considered as most suitable for this process, identifying the technological parameters of the process, study of bitumen interaction with LRW components, study of bitumen compound properties, including those associated with their long-term storage under conditions similar to the disposal ones. Research on fire- and explosion safety of the compounds was performed as well.

Bitumen is a mix of various hydrocarbons produced by oxidation of residual refinery product – tar with atmospheric air. Three main fractions are typically found in its composition: oils, resins and asphaltenes. Bitumen properties mainly depend on the ratio of these fractions [2].

Based on the research, "road" bitumen of type BND 60/90 and BND 40/60 was chosen as a matrix material for LRW bituminization at industrial facilities [3].

Main properties of bituminous compound important in terms of the long-term disposal safety were studied:

- water resistance (leaching rate of LRW components);
- fire and explosion safety;
- radiation resistance;
- microbiological resistance.

Water resistance of bituminous compound

Leaching rate is considered as the key indicator specifying the ability of a matrix material to contain LRW components [4].

The following dependences were obtained for the water resistance based on the study of bituminous compound properties [3, 5].

- leaching rate of radionuclides and LRW macrocomponents present in the compound amounted to 10^{-4}–10^{-5} g/cm^2·day;
- leaching rate of a component depends on the form of the compound in LRW salts. Leaching of water-soluble compounds is higher than that of insoluble ones;
- leaching rate depends on composition of bitumen used for LRW processing purposes. Bitumen-based compounds with a high content of asphaltenes ("hard" bitumen) are characterized by a higher leaching rate;
- Leaching rate depends on the ratio of salts and bitumen in the bituminous compound (salt content or salt filling). The higher is the salt content, the higher is the leaching rate. Moreover, depending on the sort of bitumen applied, if the salt content exceeds 50—55 %, the leaching rate increases dramatically. Based on these results, for industrial bituminization installations, the degree of compound filling was specified at a level of 40 %.

Fire and explosion safety of bituminous compounds

Obviously, fire and explosion safety of bituminous compounds is the main cause of concern when considering options for bituminous compound disposal. Below are discussed the properties of bituminous compound's main components resulting from LRW bituminization in light of the associated fire hazard.

Bitumen is referred to combustible substances and is characterized by the following thermal resistance indicators [6]:

- flash point in an open crucible — 240—299 °C;
- flash point in a closed crucible — 212—270 °C;
- ignition temperature — 300—351 °C;
- self-ignition temperature — 380—397 °C.

Sodium nitrate: non-flammable oxidizing agent contributing to spontaneous combustion of combustible substances, classified as fire-hazardous material the properties of which are as follows [6]:

- melting point — 308 °C;
- decomposition temperature — 380 °C.

Decomposition of sodium nitrate results in oxygen release being capable of supporting the burning of combustible substances even under inert conditions.

Fire hazard study of bituminous compound with sodium nitrate imitating the composition of waste generated at NPPs with RBMK units has shown that their flash point and ignition temperature are slightly lower compared to pure bitumen accounting for 228—231 and 280—290 °C respectively. For compounds filled with dry salts similarly to LRW from NPPs with WWER reactor units, the ignition temperature decreases to 250 °C [7]. Table 1 summarizes the results of the study on the thermal resistance of bituminous compounds based on BNK-2 sort of bitumen [8].

Research studying the combustion propagation for compounds simulating LRW from NPPs with RBMK reactor units has shown that it occurs when the salt content exceeds 62 wt. %. Experiments

Table 1. Thermal resistance of bituminous compounds [8]

Filling	Ratio between bitumen and filling, mass.%	Temperature, °C		
		flash	ignition	Self-ignition
Sodium nitrate	40/60	279	354	506
	60/40	293	356	548
Mixture simulating NPP waste	40/60	293	346	532
	60/40	305	356	538
BNK-2	–	293	345	–

evaluating the possibility of bitumen compound burning in a nitrogen atmosphere have shown that after being inflamed with a high-temperature source under conditions with salt content exceeding 65 %, the samples burned out completely at a burning rate of 0.11 kg/s·m^2 [7].

Experiments studying the explosion capacity of bituminous compound were carried out using the samples based on BNK 45/180 and BND 60/90 sorts of bitumen and sodium nitrate salt simulating LRW from NPPs with RBMK reactor units. It was found that the lower limit of detonability accounted for 6—7 wt. % from the bitumen content in the compound. Compounds retain their detonation ability if the content of bitumen is lower than 15 wt. % [8]. At the same time, bituminous compound structure also affects its detonation ability. When characterized with a lumpy structure, it retains detonability at bitumen content up to 25—30 %. When heated to a liquid state, mixture do not detonate and retain this property even when the molten bitumen solidifies [3, 9]. Therefore, optimal conditions for initiating the detonation of weakly detonating mixtures correspond to a large mass of the mixture, solid shell and a powerful initiator [8].

Radiation resistance of bituminous compounds

Estimates shows that for typical NPP wastes, the dose of radiation before the decay of the radionuclides contained within shall not exceed 10^5 Gy and will not cause changes in bitumen compounds properties [3].

Microbiological resistance

Field studies were carried out for 1.5 years and involved bituminous compound samples based on BNK-2 and BN-5 bitumen types with 50 % content of LRW from NPPs with RBMK reactor units. It was demonstrated that the increased growth of fungi is observed in areas that are contaminated by soil and are affected by moisture ingress. Thus, it was concluded that for the material being disposed of, contacts with soil and moisture should be avoided. The research conducted has demonstrated that the bitumen compound formed by bituminization of LRW from NPPs can be considered as biostable owning to LRW high alkalinity [10].

Behavior of bituminous compounds under long-term storage

Study of bituminous compounds behavior was performed for 15—20 years both under laboratory conditions and conditions simulating actual waste disposal [1, 11]. The following issues were studied:

- leaching of radionuclides and LRW macro-components contained in the compound under long-term storage conditions;
- changes in bitumen fraction composition;
- distribution of radionuclides between bitumen and LRW salts contained therein.

Leaching of radionuclides and macro components

Long-term observations over the behavior of bituminous compounds under conditions imitating actual soil trench disposal of waste were performed at MosNPO Radon site. These observations lasting for over 15 years have demonstrated water resistance of bitumen compounds and diffusion driven release of LRW components.

Changes in bitumen fraction composition

Research has shown that bitumen fraction composition is subject to some alterations during its storage. These alterations are due to the increase in asphaltene fraction. Most apparent changes were observed in the parts of the compound blocks being immediately adjacent to the soil layer. After 14 years of storage, the proportion of asphaltenes increased by some 4 %.

Radionuclide distribution

Various studies have demonstrated that the process of bituminous compound generation and storage involves interaction between bitumen components and the substances present in LRW. Most intense are the interactions occurring at bituminization stage characterized by the presence of highly concentrated solutions and elevated temperature. Under laboratory conditions, when stored underwater, activity of the bitumen compound components (bitumen and salt filler) is practically equal with the bulk activity, over 90 %, contained in the asphaltene fraction. Spectral analysis of asphaltenes revealed the presence of about 1.6 % of sodium. However, sodium was not identified in asphaltenes isolated from the initial bitumen.

Based on the research performed to date, one can state that long-term storage of bituminous compounds involves some reactions resulting in the changes of their fractional composition and redistribution of LRW components between bitumen and salt filler. Presence of sodium in the composition of asphaltenes may indicate that denitration processes may occur. These processes may change compound composition and increase the moisture content. When very thick compound layers are stored, salt sedimentation cannot be ruled out, thus, resulting in an increase in the salt filling of the compound's underlying layers. It seems necessary to perform some purpose investigations to study the compounds being stored at NPPs and the dynamics of the abovementioned processes.

State-of-art in bituminous compounds storage and disposal requirements

The amount of bituminized RW accumulated to date accounts for over 26,000 m^3. Most part of this waste is stored at Leningrad NPP (some 25,000 m^3) and Kalinin NPP (1,000 m^3). Designs of these of these NPP involves appropriate vessel-type storage facilities being arranged at the sites. These are concrete structures with compartments designed to accommodate bituminous compound discharged there via a heated compound pipeline [5]. Research has shown that to maintain the required fluidity and to eliminate delamination of the compound when pumping it over via the compound pipeline, its temperature should be no less than 100—200 °C. It's believed that this temperature ensures compound spreading by 8—9 m with delamination avoided.

Federal norms and rules in the field of atomic energy use allow bituminous compound disposal [12,13] with some requirements to be complied with:

- compounds are categorized according to RW classification system in keeping with the criteria specified for retrievable waste;
- characteristics of bituminous compounds should comply with regulatory requirements;
- specific engineering solutions should be provided for bituminous compound disposal systems.

One of the key issues refers to assigning bituminous compounds from the solidification of LRW bottom residues to a specific class of retrievable radioactive waste specified based on the presence of long-lived and short-lived radionuclides and their specific activity in the waste intended for disposal. Based on the composition of processed LRW, the main radionuclides determining its radiological hazard were identified, namely: ^{137}Cs, ^{90}Sr, ^{60}Co, ^{63}Ni, ^{54}Mn, ^{51}Cr, ^{55}Fe, ^{59}Fe. All the above-mentioned radionuclides except for ^{63}Ni refer to the category of short-lived. According to RW inventory lists, bituminous compounds generated from reprocessing of LRW bottom residues at Leningrad NPP only ^{137}Cs and ^{60}Co are present with the specific activity of these radionuclides accounting for $1.37{\cdot}10^4$ and $2.27{\cdot}10^1$ Bq/g respectively, whereas those generated at Kalinin NPP contain ^{137}Cs, ^{134}Cs, ^{60}Co, ^{54}Mn, with their specific activity accounting for $1.02{\cdot}10^4$, $2.2{\cdot}10^3$, $3.9{\cdot}10^2$ and $7.0{\cdot}10^1$ Bq/g respectively. It should be noted that most probably the presence of long-lived ^{63}Ni in bituminous compound was not identified as it is regarded as a hardly detectable one. Moreover, one can hardly expect that its specific activity in bituminized LRW exceeds $1{\cdot}10^5$ Bq/g (the limit indicating that materials containing the radionuclide can be attributed to the category of radioactive waste [14]). Thus, bituminous compounds resulting from reprocessing of LRW bottom sediments generated at NPPs can be categorized as retrievable RW Class 3 requiring disposal in near-surface disposal facilities [14].

In keeping with the provisions of fire hazard classification system, bituminous compounds are categorized as "combustible" substances [15] and can be disposed of in packages (containers) the fire resistance of which had been demonstrated in disposal facility designs [13]. Thus, disposal of bituminous compounds is possible if relevant provisions are provided for in disposal facility's designs including, in particular, some specific requirements on the fire safety of disposal packages (containers).

Moreover, federal norms specify the main quality indicators for bituminous compounds associated with their radiation indicators, water resistance, water content, thermal and radiation resistance (table 2) [12].

Table 2. Main quality indicators for bituminous compounds [12]

Quality indicator	Acceptable values
Compound's specific activity, beta-activity, alpha-activity	No more than 10^{10} Bq/kg No more than 10^6 Bq/kg
Water resistance (radionuclide leaching rate for ^{137}Cs and ^{90}Sr)	Less than $1{\cdot}10^{-4}$ g/cm^2·day
Free moisture content	Less than 3 % with ion exchange resins Less than 1 % with salt solution
Thermal resistance	T flash point of over 200 °C T ignition of over 250 °C T self- ignition of over 400 °C
Radiation resistance	Increase in the volume by less than 10 % after 10^6 Gy exposure

When the compliance of bituminous compounds stored at NPP sites with the established quality indicators is being evaluated of most concern are believed to be the indicators demonstrating the free moisture content. Moreover, given the explosion safety requirements mentioned above, compound re-melting at the conditioning stage should be provided for allowing to bring its moisture content indicators, along with other indicators, in line with the established parameters.

Bituminous compound emplacement in vessel-type storage facilities of large volume caused some difficulties in terms of their further conditioning and disposal. Engineering solutions ensuring the retrieval process of bituminous compounds from such storage facilities are now being developed and investigated. Considered are the following key proposals:

- heating and buildup of heated bitumen;
- deep cooling of the compound in the storage compartment with its subsequent mechanical destruction and removal of the resulting pieces.

In addition to this an option suggesting dissolution of bitumen compound matrix in organic solvents, followed by separate processing of organic and inorganic components is being considered. Application of this method seems to be hardly probable due to its complexity and considerable labor intensity.

The methods suggesting bitumen heating and its pumping in a liquid form are widely used in various industries with detailed designs and industrial equipment already available for these purposes. Moreover, compound pumping method was provided for LRW bituminization operations at NPPs. Thus, the know-how on such operations is already available. For common industrial purposes, steam and electric heating systems are most widely used for bitumen heating. In addition to that, systems with gas heating, infrared radiation or high-frequency heating are used. Given high-level fire safety requirements in the field of radioactive waste management, we believe that steam heating should be regarded as the most preferable option.

In general, retrieval of the bituminous compound from the storage facility and its conditioning should be performed in two stages. The first stage involves local heating of the selected area of the storage facility and pumping of the compound via heated pipeline into intermediate reservoir (bitumen melting vessel). The second stage provides for bituminous compound additional heating in the bitumen melting vessel, its mixing and conditioning to attain the required parameters in terms of moisture content with subsequent pumping into molding primary package.

In keeping with relevant provisions of regulations on the safe transportation of radioactive materials, transport package of type A should be used for bituminous compound transportation [16]. There are several design options available: one of them suggests the use of NZK-150-1.5P non-returnable shielding container. This container can be used both for transportation and for disposal purposes.

The following flow chart was proposed for bituminous compound conditioning: molten bitumen compound is poured into molding package, for example, thin-walled metal 200-l drums. Four drums filled with bituminous compound are placed into NZK-150-1.5P containers with the voids filled with clay-based buffer material. Compound pouring directly into container is not considered feasible due to its large mass and long compound cooling time. Sealing of the container is ensured by a lid placed on its top (figure 1).

Calculation of temperature distribution in DFRW compartment in case of emergency ignition of bituminous compound

To assess the safety in case of emergency ignition of bituminous compound, calculation evaluating temperature buildup in DFRW compartment was performed. The following conditions were considered in this assessment.

Characteristics of bituminous compound

The compound was produced using bitumen and sodium nitrate at a ratio of 35/65 by mass. The burning rate accounted for 0.11 kg/s·m^2 [8].

Bitumen. Key components, % (mass): carbon — up to 85, hydrogen — up to 15, sulfur — no more than 1.5, oxygen ~ 2, nitrogen — fractions of a percent.
Density: 1.02 g/cm^3.
Self-ignition temperature: 380—397 °C [3].
Specific heat capacity: 2.3·10^{-3} J/kg·deg (at T = 300 °C) [2].
Heat conductivity coefficient: 1.45 W/m·deg [2].
Calorific value: 40.95 MJ/kg [17].

Salt filler. Sodium nitrate.
Molar mass: 84.99 g/mol.
Density: 2.26 g/cm^3.
Decomposition temperature: 380 °C.
Molar heat capacity: 67 J/mol·K.

When heated, the salt decomposes in the following way:

$$2NaNO_3 \rightarrow 2NaNO_2 + O_2\uparrow.$$

Figure 1. RW disposal package with NZK-150-1.5P container

NZK-150-1.5P container main characteristics

Empty container mass (with lid), t	4.3
Container weight with waste (not more than), t	7.3
Container capacity, m^3	1.5
Wall thickness, mm	150
Container dimensions, mm	1650×1650×1375

Figure 2. RW packages installed in DFRW compartment

Conditioning and disposal flow chart

Bituminous compound is packaged into thin-walled metal 200-liter drums. Four drums filled with a bituminous compound are packaged into NZK-150-1.5P container, the voids are filled with buffer clay-based powder, the container is closed with a lid and sealed (disposal package) (figure 1).

RW packages are stacked in DF RW compartment layer by layer with the voids in-between filled with clay-based powder. The height of container layers accounts for 6 rows (figure 2).

Thermal mode calculations

The calculation was aimed at identifying temperature changes inside containers of the storage facility in case if one of the containers catches fire. It was assumed that 0.8 m^3 of compound were contained inside the container. Thus, bitumen mass in 800 liters of compound would account for $1.272 \cdot 0.35 = 445.2$ kg. Heat output resulting from this amount of bitumen being incinerated would amount to $41 \cdot 445.2 = 182 \cdot 10^8 = 1.82 \cdot 10^{10}$ [MJ]. At the incineration rate of 0.11 kg/cm·m^2 and incineration space area of 1 m^2, the time corresponding to compound burning inside the container would account to approximately 4,000 seconds. If the energy emitted is recalculated accounting for the rate emitted by m^3 inside the burning container, the following value can be obtained: $1.82 \cdot 10^{10}/(4 \cdot 10^3 \cdot 0.8)$ [J/(s·m^3)] = $= 5.7 \cdot 10^6$ [W/m^3].

To calculate heat distribution 3DMKE software FENIA [18] was applied. Meshing was performed for the disposal area involving the burning container and several others surrounding it. Figure 3 shows the vertical cross section of the mesh one-by-one representing all disposal components and the general grid.

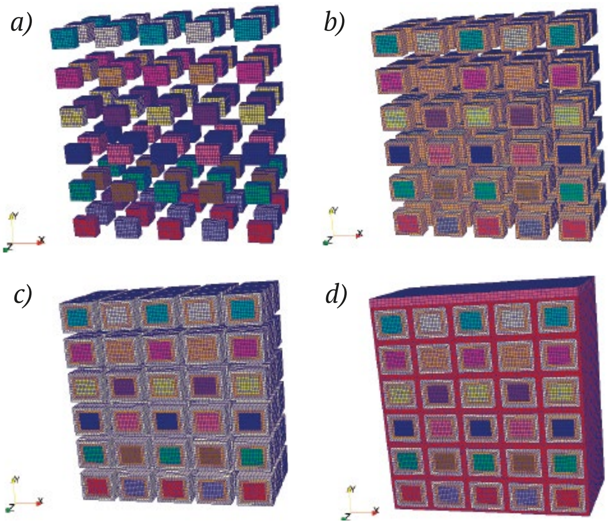

Figure 3. Vertical cross section of the mesh: a) compound; b) compound and backfilling inside the container; c) compound, backfilling inside the container and container concrete; d) compound, backfilling inside the container, container concrete and backfilling between the containers and a concrete slab

Absence of a heat flux on the external surfaces was indicated as boundary condition (conservative estimate). The software allowed to calculate the heat mode inside disposal containers in case if the middle one (third from the top) caught fire. It should be noted, that the burning rate indicated above is somewhat overestimated as the drums inside the container will impede the expansion of the burning surface. Therefore, in order to remove the indicated uncertainty, the calculations were carried out for different burning rates (the

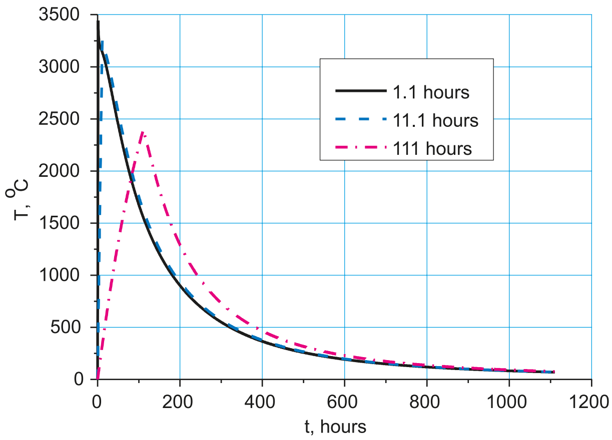

Figure 4. Temperature-time dependacy for the burning compound under different burning rate of the compound inside the container

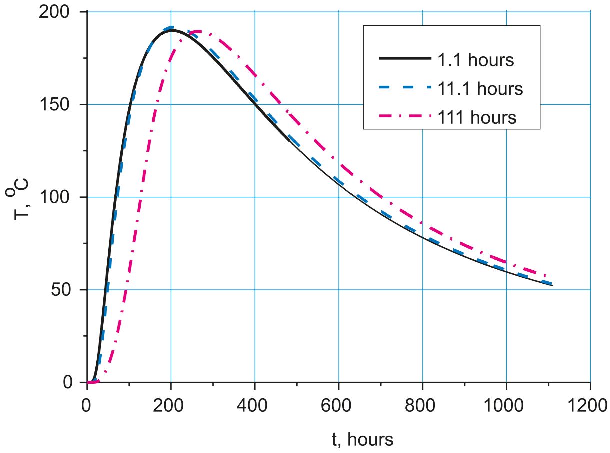

Figure 5. Temperature-time dependacy for the container located to the side from the burning one

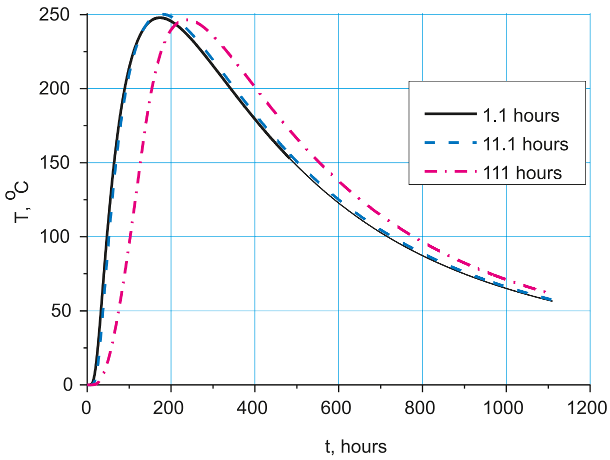

Figure 6. Temperature-time dependacy for the container located below the burning one

combustion time of the bitumen in the container was taken equal to 1.1 h = 4000 s, 11.1 h, 111 h). Figures 4—6 represent the results of calculations with relevant temperature-time dependences estimated for different burning times. At the same time, for the container located from one side of the burning one (figure 5) and the container located below the burning one (figure 6), the grid points closest to the burning container were selected (i.e. the nodes with the highest temperature in the corresponding container). It should be noted that at low burning rates (entire container material got burned out during less than 11 hours), the change in the burning rate has a small effect on the temperature inside the containers. Additional calculations were performed for another possible case suggesting ignition of the upper container. In this case the temperature in the adjacent containers will be lower than in case if the middle container catches fire. Therefore, the results of this calculation were not presented in the paper. Thus, if one container catches fire inside the DFRW, the temperature of adjacent containers will be less than 250 °C which is much lower than the self-ignition temperature typical for bituminous compounds.

Conclusions

1 Bituminous compound resulting from bituminization of LRW bottom residues at NPPs may be categorized as retrievable radioactive waste Class 3.

2 Under long-term storage, bituminous compound may be subject to some processes resulting in altered fractional composition of bitumen with an increase in the content of asphaltene fraction and sedimentation of the filler's salt particles. These processes may change its properties potentially producing a negative effect on the safety of its disposal, especially when pored into large capacity compartments.

3 Most favorable is considered the option when bituminous compound is disposed of in separate packages. One of bituminous compound conditioning options suggests that transportation packages and disposal packages are manufactured based on non-returnable shielded container NZK-150-1.5P.

4 Requirements to the systems and methods for emplacing packages with bituminous compounds inside DFRW, as well as those considering their fire safety should be discussed and demonstrated within relevant designs.

5 Development of a conditioning technology for bituminous compound should provide for its transfer into a monolith state, as well as to a form complying with main quality indicators specified in federal norms and rules in the field of atomic energy use.

6 The calculations performed have demonstrated that the use of non-returnable shielded container NZK-150-1.5P as a disposal package prevents the spread of fire caught by bituminous compound.

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