

RADIONUCLIDE COMPOSITION OF SOLID RADIOACTIVE WASTE FROM CONVERSION PRODUCTION

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The paper overviews the study on the radionuclide composition of solid radioactive waste (RW) held in a near-surface storage facility at a conversion production site. The total α - and β -activity of the waste ranges from 1.2 to 1,250 kBq/kg and from 1.1 to 560 kBq/kg respectively. Three radioactive waste categories have been identified: those with the predominance of ^{226}Ra in combination with short-lived daughter decay products (specific activity of up to 100 kBq/kg); waste with a predominant content of ^{238}U in equilibrium with ^{234}Th , ^{234}Pa (specific activity of up to 30 kBq/kg); waste with a predominance of ^{210}Pb in equilibrium with ^{210}Bi and ^{210}Po (specific activity of 70 kBq/kg). The increased total alpha activity level is explained by ^{230}Th found in the waste. At low ^{226}Ra activities, one can focus on the total alpha activity levels to identify areas with possible high ^{210}Po and ^{230}Th activities. Most of the waste inventory contains uranium with its isotopic composition being similar to the natural one.

Keywords: radioactive waste, radionuclide analysis, thorium-230, radium-226, uranium.

Introduction

The Chepetsky Mechanical Plant (Glazov, Udmurt Republic) is currently seen as a largest world and the only Russian producer of zirconium and zirconium-based alloy products, as well as of hafnium, calcium and low-temperature superconducting materials. CMP also holds a leading position in niobium, titanium and titanium-based alloy production. Nevertheless, current environmental contamination of its site is associated with uranium production that was launched there back in 1946 under the USSR Atomic Project and discontinued in 2016 by a formal decision on deploying the entire sublimate process cycle at the MCC site (Seversk, Tomsk region).

Even though uranium production was moved from the CMP site, its nuclear legacy involving a

triad considered classic for nuclear industry enterprises established back in 1940–1950s still remains a problem to be addressed [2]:

1) buildings, sites and processing facilities used in the past to produce industrial reactor fuel and other materials for defense purposes being no longer in use but haven't been subjected to proper decommissioning;

2) radioactively contaminated areas that are still present at the industrial sites;

3) large RW inventories (both in terms of their volume and specific activities) accumulated in near-surface storage facilities.

In the early days of CMP operation, many environmental problems were casted aside with a

priority placed on the main goal — Russian atomic bomb and its development [2]. Therefore, some large-scale efforts are now required to reduce the negative environmental impacts from the enterprise. A most important task in this respect is to condition the RW held in near-surface storage facilities of the enterprise [1], [2].

Tailing storage facility No. 1 was commissioned at the CMP site in 1951 to store solid radioactive waste with the second one commissioned in 1965 (tailings storage facility No. 2). The latter is still in operation and is used to store waste from other production facilities of the enterprise. Therefore, current efforts are mainly focused on TSF No. 1 decommissioned in 1980. For thirty years, it was accepting waste from three production cycles: solutions and sludge from uranium production, hydrate and dry waste from calcium and zirconium productions. This was done, in particular, to prevent dust formation in case of uranium-containing waste and to reduce its specific activity. Such long-term operation has resulted in an inventory of about 2 million tons of RW accommodated in the facility. Regional environmental experts and scientists dealing with nuclear legacy issues note that the challenges associated with their conditioning should be addressed [1], urgently [2]. Such haste and high relevance of this issue are explained by CMP's site location on the left bank of the Cheptsya River in close proximity to the river bed, which remains an important waterway for a large number of settlements, including villages and hamlets. Therefore, TSF poses high risks to the river ecosystem, as well as households and the health of the residents. Multiple sections of engineering structures have been erected to prevent the releases of harmful substances into the environment; the storage facility is surrounded by a system of reinforcements, drainages and observation wells. However, global experience shows that oftentimes such measures turn out to be insufficient. At such sites, uranium releases beyond the storage facilities' boundaries and those of its decay products are often caused by their infiltration into groundwater, dust transfer, as well as various emergency situations [4]–[15]. It's hard to rectify their consequences since such radioactive cleanup of soils and water bodies involves high financial and labor costs. Obviously, the damage inflicted to the health and well-being of the population cannot be rectified at all. Therefore, RW accommodated in near-surface storage facilities at uranium production sites should be conditioned, which is currently seen as an important environmental task. Moreover, the opportunities offered by uranium extraction and its further use in the nuclear fuel cycle make this task even more relevant for the nuclear energy.

CMP's TSF No. 1 involves three zones separated by dams and protected by an external embankment dam. Its total area, including the embankment dams, accounts for 50 hectares. At the final stage of its service life, the facility was mothballed: the stored waste was dehydrated and the facility itself backfilled with two layers of soil having a total thickness of 0.65 m [1]. In the early 2000s, to provide better RW isolation from the environment, the layer thickness was increased via some additional backfilling with soil extracted by a dredger in the water area of the Cheptsya River.

According to previous estimates, the protective screening soil layer at the TSF No. 1 had a thickness of 3–4 m with quartz accounting for over 60% of it by mass. Its phase composition also involves alunogen, anorthite, albite, gismondine, muscovite, calcite and gypsum. It is underlaid by solid radioactive waste of quite highly heterogeneous composition both in terms of their location and the storage depth.

Section 1 accommodates some uranium production waste with the predominance of gypsum (64–87% wt.) and calcite (about 8% wt.) and only some 0.01–0.05% wt. accounting for uranium inventory.

Section 2 is divided into two zones. The first one involves a layer of zirconium production waste stored at a depth of 4–5 m and mostly accounting for calcite (about 60% wt.) and fluorite (over 31% wt.) and only some 0.5–0.65% wt. of uranium. This layer is underlaid by uranium production waste: mostly gypsum (over 80%), calcite (5–9% wt.) and quartz (6–8% wt.). Whereas, the uranium inventory in them accounts for only 0.1% wt. The second zone in this section involves a layer of calcium production waste from CMP operations disposed at a depth of 3–4 m with over 90% wt. of its inventory represented by calcite and only some 0.51% wt. by uranium. This layer is underlaid by waste from uranium production predominantly consisting of gypsum (up to 84% wt.); containing some calcite (5–11% wt.) and quartz (more than 5% wt.). This layer accounts for only some 0.02–0.04% wt. of uranium.

In the past, Section 3 was accepting not only the sludge cake from uranium ore leaching but also some calcium production waste seeking to prevent dust formation and reduce the background radiation. The waste in this section accounts for 63% wt. of calcite with the uranium content in it amounting to 0.3–0.5% wt. [16].

Thus, uranium is present throughout the entire SRW inventory accommodated in the TSF No. 1. Moreover, it has been identified in a few layers of samples collected from the protective soil layer [16]. Therefore, a relevant task is seen in specifying the radiation characteristics of waste from TSF No. 1 to provide further selection of appropriate SRW conditioning methods. Present study focuses on this task specifically.

Experimental part

Soil samples were collected from the protective shielding layer and SRW from three TSF No. 1 sections. Since the SRW storage depth amounted to 7 m, an excavator was used to remove a soil-vegetation layer with the soil samples themselves collected by the excavator's bucket from the newly exposed surface areas at 1-meter increments with a separate soil dump being established. This dump was further leveled into a layer being no less than 40 cm thick. A shovel was used to take the samples of an approximately same volume: these were collected from 10 sampling points, i. e., from equidistant holes located at different depths in a checkerboard pattern. All samples collected from a single dump were poured into a container. The total sample mass eventually amounted to about 30 kg. The procedure was repeatedly followed for each subsequent layer up to a depth of 7 m. At the next stage, each combined sample corresponding to a certain SRW layer was averaged and reduced by the ring and cone method to a mass of 1.5–2.0 kg. For research purposes, the reference samples were dried and dispersed in a ceramic mortar.

Atomtech MKS AT-1315 gamma-beta spectrometer (Atomtech, Belarus) with a scintillation NaI (Tl) detector of 63 × 63 mm was used to measure the specific activity of the solid samples: the samples were emplaced into a standard Marinelli vessel with a capacity of 1 dm³, the mass was recorded (in most cases the mass was ranging from 850 to 1,200 g) and measured for 300–2,000 sec depending on the sample activity.

Alpha-beta activity of the samples was measured using a low-background alpha-beta radiometer UMF-2000; alpha activity was measured by a Multi-rad-AS alpha spectrometer (NPP Doza, Russia) with a silicon surface-barrier semiconductor detector.

Discussion

Gamma spectrometry showed that according to their radionuclide composition the soil samples collected from the protective shielding layer and SRW involved some decay products of the ²³⁸U series. Thus, three categories of samples could be distinguished. The first one featured the samples with a predominant content of ²²⁶Ra in combination with its short-lived daughter decay products, i. e., most of the soil samples collected from the SF protective shielding layer and SRW. The specific activity of ²²⁶Ra and its daughter products in them was found to be amounting to 70 kBq/kg. The second category included samples with a predominant content of ²³⁸U being in equilibrium with its daughter

products, namely, ²³⁴Th, ²³⁴Pa, i. e., samples of zirconium production waste stored at a depth of 4–5 m in the first zone of Section 2, as well as protective soil layer sample collected from a sampling point in Section 1. Their specific activity amounted to up to 30 kBq/kg. The third category involved samples with a predominant content of ²¹⁰Pb, which most probably appeared to be in equilibrium with ²¹⁰Bi and ²¹⁰Po, namely, calcium production waste stored at a depth of 3–4 m in the second zone of Section 2. Their specific activity accounted for 70 kBq/kg. Figure 1 shows the gamma spectra characteristic of the discussed soil and SRW sample categories.

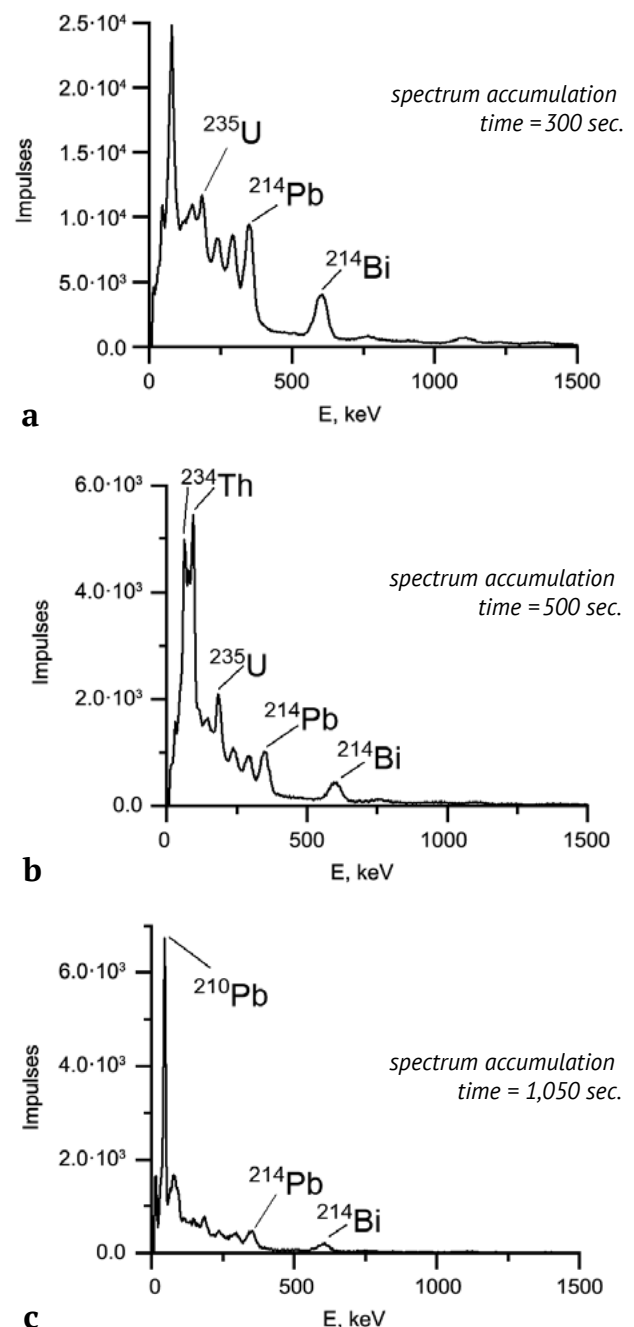


Figure 1. Gamma spectra of SRW samples with a predominant content of ²²⁶Ra (a), ²³⁸U (b), ²¹⁰Pb (c)

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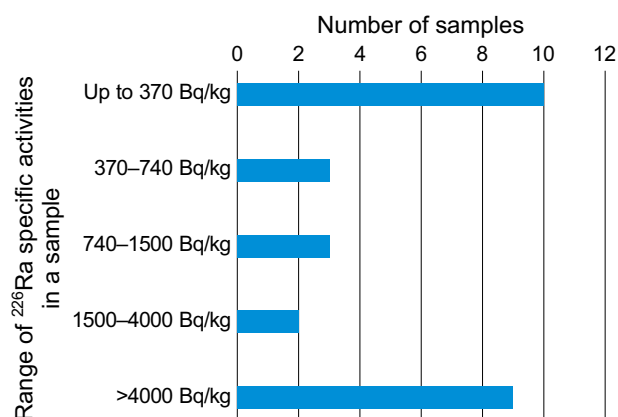


Figure 2. Distribution of samples according to their number and ²²⁶Ra specific activity levels

Figure 2 shows the distribution of a few samples studied according to ²²⁶Ra content; the ranges of specific activities were selected in accordance with NRB-99/2009 (clause 5.3.4).

Tables 1–3 summarize relevant findings on the specific alpha-beta activity of soil samples collected from the protective shielding layer and SRW samples from the storage facility.

Tables 1–3 show that the specific alpha activities of the soil samples from the remediation layer and SRW were ranging from 1.2 to 1,200 kBq/kg, whereas the specific beta activities – from 1.1 to 560 kBq/kg. Moreover, most of the studied samples turned out to have unexpectedly higher alpha activity levels than those of beta activity and in a quite considerable way (up to 3.5 times). Thus, one may conclude that the samples contained some alpha-emitting radionuclides that could not be measured by gamma spectrometry.

Additional alpha spectrometric measurements have been implemented to get a more precise picture of the waste in terms of its radionuclide composition: a weighed 1.5 g sample collected from uranium production waste with a maximum ²²⁶Ra

Table 1. Measured alpha-beta activities of soil samples from the SF's remediation layer

Sample	Specific activity of Radium-226, Bq/kg	Specific α -activity, Bq/kg	Specific β -activity, Bq/kg	Ratio between specific α -and Radium-226 activities	Ratio between specific β -and Radium-226 activities	Section number (zone)
Z-1	794	2.87E+04	1.85E+04	36.1	23.3	1
Z-2	12,900	2.97E+05	8.55E+04	23.0	6.6	
Z-3	714	1.59E+04	5.69E+03	22.3	8.0	2 (1)
Z-4	342	2.13E+04	8.28E+03	62.4	24.2	
Z-5	1420	4.40E+04	4.68E+04	31.0	33.0	
Z-6	95.7	3.49E+03	1.30E+03	36.5	13.6	
Z-7	53.8	1.95E+03	1.20E+03	36.3	22.2	
Z-8	74.4	1.64E+03	1.37E+03	22.1	18.4	
Z-9	64	1.96E+03	1.69E+03	30.7	26.4	2(2)
Z-10	442	6.19E+03	3.43E+03	14.0	7.8	
Z-11	1,360	9.58E+03	3.87E+03	7.0	2.8	3
Z-12	45.1	1.09E+03	1.02E+03	24.2	22.6	
Z-13	56.1	1.87E+04	6.17E+03	332.6	109.9	
Z-14	76.4	1.91E+03	1.28E+03	25.1	16.7	

Table 2. Measured alpha-beta activities of samples from uranium production waste held in the storage facility

Sample	Specific activity of Radium-226, Bq/kg	Specific α -activity, Bq/kg	Specific β -activity, Bq/kg	Ratio between specific α -and Radium-226 activities	Ratio between specific β -and Radium-226 activities	Section number (zone)
U-1	69,500	1.23E+06	5.55E+05	17.7	8.0	1
U-2	22,300	2.60E+05	1.54E+05	11.6	6.9	
U-3	41,000	7.40E+05	3.03E+05	18.0	7.4	2 (1)
U-4	57,100	5.59E+05	2.29E+05	9.8	4.0	
U-5	26,300	1.00E+06	4.80E+05	38.1	18.2	
U-6	55,800	1.22E+06	5.60E+05	21.8	10.0	2(2)
U-7	45,200	1.07E+06	5.00E+05	23.7	11.1	
U-8	37,100	1.14E+06	3.26E+05	30.7	8.8	

Table 3. Measured alpha-beta activity of samples from zirconium and calcium production waste held in the storage facility

Sample	Specific activity of Radium-226, Bq/kg	Specific α -activity, Bq/kg	Specific β -activity, Bq/kg	Ratio between specific α - and Radium-226 activities	Ratio between specific β - and Radium-226 activities	Section number (zone)
Zr-1	3,320	1.02E+05	1.27E+05	30.9	38.2	2(1)
Zr-2	2,310	9.25E+04	1.14E+05	40.0	49.5	
Ca-1	470	4.24E+04	4.18E+04	90.1	89.0	2 (2)
Ca-2	103	1.24E+03	1.09E+03	12.0	10.6	3
Ca-3	53.6	1.27E+03	1.15E+03	23.7	21.4	

activity (Table 2, U-1) was leached using 20 cm³ of 3 M nitric acid, thereby, providing maximum radionuclide transfer into a dissolved state. At the next stage, the sample was filtered and the solution was neutralized with ammonia to a pH level of 6–7. Then the solution was filtered again and the filtrate was poured into a 100 cm³ bottle. A thin-layer MnO₂ sorbent – PE (manganese dioxide deposited on polyethylene) disk was installed under its lid. Although it was primarily designed to support analytical studies of radium isotopes in aqueous samples, due to the inherent properties of manganese dioxide being a non-selective sorption-active material it is also capable of extracting other ions from solutions, especially heavy metal ions. The lid with the sorbent sample was screwed so that the edges of the disk would directly cover the bottle neck and provide tightness of the system. Then the bottle with the solution and sorbent was turned upside down, secured with magnets on a shaker with an adjustable rotation speed. The time of the sorption process onset was recorded and the stirring mode was turned on. When the specified phase contact time elapsed, the shaker was tuned off, the bottle with the solution and sorbent was turned upside down, the solution was drained, sorbent sample was removed, washed under a rush of distilled water, dried in air for several minutes and measured by an alpha spectrometer Multirad-AS. Figure 3 shows the alpha spectra of the sorbent resulted from the exposure of a uranium production waste sample to a leached solution for 28 hours and 11 days.

At the post-leaching phase, the filtered waste sample was re-filled with 20 cm³ of 3 M nitric acid. Since all of the carbonates got dissolved under the first leaching stage, the acidity of the solutions at this stage turned out to be much higher. Upon 24 hour-long treatment, a sample was prepared in the same way as described above with the sorption process implemented using the MnO₂ – PE sorbent. Figure 4 presents the measured alpha spectrum.

Gamma spectrometry showed that the RW sample contained only ²²⁶Ra with its short-lived daughter radionuclides. However, high-resolution alpha

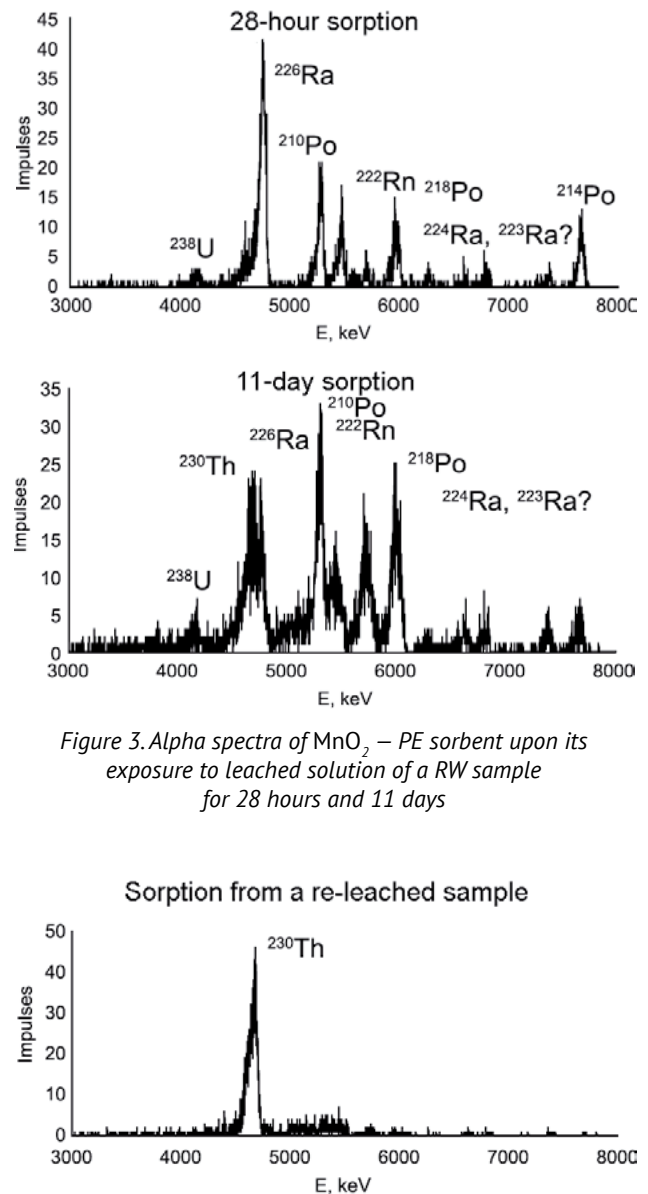


Figure 3. Alpha spectra of MnO₂ – PE sorbent upon its exposure to leached solution of a RW sample for 28 hours and 11 days

Figure 4. Alpha spectra of MnO₂ – PE sorbent upon its exposure to a re-leached solution of a RW sample

spectrometry evidenced that the waste contained not only ²²⁶Ra, but also ²¹⁰Po, ²³⁸U and ²³⁰Th. At the same time, sorption from a re-leached sample showed that it was the ²³⁰Th that have most likely contributed to the high alpha activity of the RW

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samples. The presence of ^{210}Po can be explained by substantial age of the waste since their long storage time has resulted in a partial accumulation of a relatively long-lived ^{210}Pb radionuclide and its daughters ^{210}Po and ^{210}Bi . The presence of ^{238}U and ^{230}Th logically follows from the fact that all these radionuclides belong to the same natural ^{238}U decay series, but their behavior seems in no way related to the one of ^{226}Ra . At the same time, gamma spectrometry cannot be used to measure the ^{210}Po and ^{230}Th activities.

Isotopic composition of uranium contained in SRW was measured due to its potential commercial value. It was done based on a technique developed earlier to measure the isotopic uranium composition in pregnant borehole leach solutions [17]. This method is considered effective in case of low uranium concentrations in a solution ($0.01\text{--}0.1\text{ g/dm}^3$) against the background of high iron ($0.5\text{--}1.5\text{ g/dm}^3$) and aluminum ($1.3\text{--}3.2\text{ g/dm}^3$) ones. The method provided for a few stages starting from the selective uranium separation from the sample by sorption on an anion exchanger. This stage was followed by nitrate-sulfate desorption of uranium from the saturated ion exchanger phase. Then, a load was produced by electrolytic deposition of uranium onto a corrosion-resistant steel substrate from a 0.6 M ammonium sulfate solution with some addition of the resulting desorbate. At the final stage, upon completing the calibration of the alpha spectrometer based on the energy of the detected alpha particles, the alpha radiation spectrum of the load was measured. The data obtained were used to calculate the peak areas according to ^{234}U , ^{235}U and ^{238}U alpha radiation in certain pre-specified energy intervals and the mass fraction of ^{234}U .

6 samples were studied under the first test. Three of them mostly contained uranium rather than other natural radionuclides, namely, two samples from zirconium production waste (Table 3) and one sample collected from the protective shielding soil layer (Table 1, sample Z-1). Two other samples referred to the category of uranium production waste and were characterized by the highest specific activity of ^{226}Ra (Table 2, U-1 and U-5). A sample of calcium production waste with a predominant ^{210}Pb content and probably an equilibrium content of ^{210}Bi and ^{210}Po was studied as well (Table 3, Ca-1). The following flowchart was implemented to measure the isotopic uranium composition of the samples:

1. A load of 2 g was leached with 10 cm^3 of 3 M sulfuric acid solution for one week. Abundant gas evolution was observed in all samples along this process, which was mainly explained by calcite being present in the waste composition.

2. The pulp was diluted by 50% and filtered through a paper filter.

3. The filtrate was adjusted with ammonia to pH $1.5\text{--}2$.

4. 50 mg of AMP anion exchanger was added to the neutralized solution ($20\text{--}25\text{ cm}^3$) seeking to provide uranium sorption and kept up like this for a week.

5. A week later the solution was drained. The resin was washed with distilled water, and then 0.5 cm^3 of a solution containing 80 g/dm^3 of ammonium nitrate and 15 g/dm^3 of sulfuric acid was poured in and held like this for at least a day seeking to provide uranium desorption.

6. At the post-desorption stage, the solution was added to 25 cm^3 of 0.6 M ammonium sulfate solution. Uranium was electrolytically deposited from the resulting composition onto a corrosion-resistant steel substrate for 45 minutes at a current of 1.5 A .

7. The ultimate load was dried in air and measured on a Multirad-AS alpha spectrometer (NTC Amplitude, Moscow, Russia).

Figure 5 shows the measured alpha spectra of the samples. The analyzed uranium isotopic composition has revealed two problems. Firstly, in addition to uranium, almost all samples (still not clear in case of Zr-1 and Zr-2 samples) were quite high in ^{230}Th , a long-lived alpha-emitting isotope from the ^{238}U decay series. Alpha radiation energy from ^{230}Th (4.69 MeV) turned out to be nearly equal to the one of ^{234}U (4.77 MeV): for this reason, these peaks were getting merged and it was difficult to calculate the $^{238}\text{U}\text{--}^{234}\text{U}$ activity ratio accurately. Secondly, zirconium waste samples contained far too much uranium, iron and aluminum making the energy resolution of the resulting alpha spectra not high enough to come up with any definitive conclusions.

Table 4 summarizes the software processing results for the alpha spectra of samples with adequate energy resolution. It was not possible to process the waste samples from zirconium production. The measured ^{235}U content turned out to be characterized with very large error due to the low content of this radionuclide, whereas the one of ^{234}U may have been overestimated due to the interfering influence produced by ^{230}Th . Nevertheless, as a first approximation, the following conclusions could be drawn:

1. The studied samples of waste from uranium and calcium production, as well as the Zr-1 sample contained uranium with its isotopic composition being close to the natural one.

2. Most probably the surface soil layer sample contained some depleted uranium: the ^{235}U count rate turned out to be less than 2% of the ^{238}U count rate (in natural uranium — approximately 7%), which corresponded to a ^{235}U content of 0.2% wt.

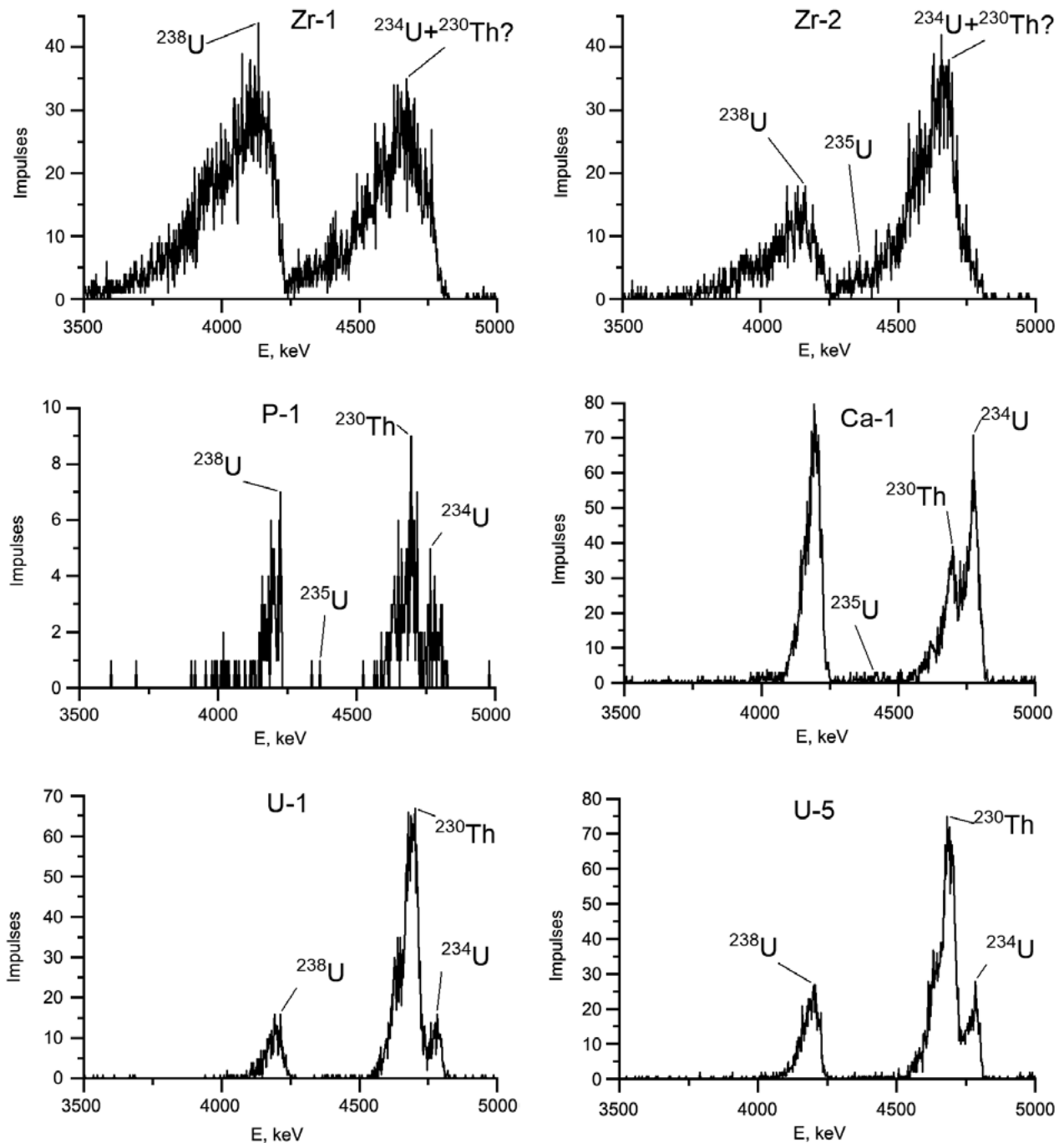


Figure 5. Alpha spectra of uranium in waste samples given the measurement time ranging from 2 to 67.5 hours

Table 4. Evaluated isotopic composition of uranium in a few samples

Sample	$A(^{238}\text{U})$, Bq	$A(^{234}\text{U})$, Bq	$A(^{235}\text{U})$, Bq	$A(^{235}\text{U})/A(^{238}\text{U})$	$A(^{234}\text{U})/A(^{238}\text{U})$
Z-1	0.052 ± 0.014	0.0012 ± 0.002	0.047 ± 0.014	0.0231	0.898
U-1	0.054 ± 0.009	0.0033 ± 0.0024	0.091 ± 0.017	0.060	1.680
U-5	0.064 ± 0.009	0.0014 ± 0.0011	0.079 ± 0.013	0.0217	1.231
Ca-1	0.045 ± 0.006	0.00087 ± 0.0004	0.043 ± 0.006	0.0195	0.959

^{234}U content in this sample fell below the standard as well. Uranium amounts found in the protective shielding soil layer indicated its active transport during SRW storage. The problem of depleted

uranium requires some additional research to identify the areas with waste contaminated with it.

3. The zirconium production waste sample Zr-2 could contain some enriched uranium. However, it

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was not possible to determine whether the sample contained ^{230}Th due to the low quality of the obtained alpha spectrum. Therefore, this result could not be considered reliable.

4. ^{230}Th was found in the composition of all samples, which is a pure alpha emitter and could not be detected by gamma spectrometry. This aspect should be taken into account in case of future waste processing, since this radionuclide would therefore require appropriate control measurements.

Further experiments are expected to improve the methods used to measure uranium and ^{230}Th isotopes in SRW samples and provide a more detailed idea of the near-surface repository layout.

Conclusions

This paper explores the radionuclide composition of the protective shielding soil layer and SRW accommodated in the tailing SF No. 1 at the CMP site. Alpha activity of the samples was found to be ranging from 1.2 to 1,250 kBq/kg, the beta activity — from 1.1 to 560 kBq/kg. Gamma spectrometry measurements showed that according to their content, the studied samples were mostly predominated by ^{226}Ra along with its short-lived daughter products (with specific activity levels of up to 100 kBq/kg). The content of zirconium production waste, as well as the one of a shielding soil layer sample collected from a sampling point within section 1, was found to be predominated by ^{238}U being in equilibrium with its daughters ^{234}Th , ^{234}Pa (specific activity of up to 30 kBq/kg). Calcium production waste mostly containing ^{210}Pb and being most probably in equilibrium with ^{210}Po and ^{210}Bi (specific activity of 70 kBq/kg) was found to be stored at a depth of 3–4 m within zone 2, section 2.

Alpha spectrometric studies showed that the high alpha activity of waste could be most likely contributed by ^{230}Th , whose presence was explained by the considerable storage time of the waste, which resulted in partial accumulation of relatively long-lived ^{210}Pb and its daughters ^{210}Po and ^{210}Bi . At the same time, gamma spectrometry is unable to detect ^{210}Po and ^{230}Th .

Thus, gamma spectrometric measurements alone are far not enough to enable waste categorization. The measured alpha activity is expected to provide fairly reliable indication in case of relatively low ^{226}Ra content, since this method is thought to support the identification of areas with potentially high ^{210}Po and ^{230}Th activities.

SRS inventory stored in TSF No.1 at the CMP site was found to contain natural uranium presenting some commercial interest. Depleted uranium was found in one of the soil samples collected from

the shielding soil layer, and, therefore, requires some additional research to identify the areas with such waste. Moreover, further SRW processing and, in particular, uranium extraction, would require TSF mapping according to its isotopic composition and specific activity levels of the waste stored in it.

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