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ANALYSIS OF RADIONUCLIDES INVENTORY CONTAINED IN LIQUID EFFLUENTS RESULTED FROM DECOMMISSIONING OF VVR-S NUCLEAR RESEARCH REACTOR

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Abstract. Activity concentration of the radionuclides presented in liquid effluents has been evaluated in IFIN-HH laboratories. Gamma-ray spectrometry analyses have been performed for liquid and residue samples, and gross beta activity has been evaluated only for residue samples. ^{60}Co, ^{137}Cs, ^{152}Eu, ^{154}Eu and ^{108m}Ag radionuclides have been found with a higher concentration in residue samples. Taking into account previous assessments, the radionuclide inventory has increased qualitatively due to the presence of ^{54}Mn and ^{241}Am radionuclides, and quantitatively due to the progress of the decommissioning project. For the first time, ^{90}Sr activity has been evaluated through gross beta measurements and it has been found that is comparable with ^{137}Cs activity. The results obtained is this study would be used for a proper radiological characterization of liquid effluents in order to transfer them in a controlled manner to the treatment plant which is in care of Radioactive Waste Management Department, IFIN-HH. The radiological risk assessment for the workers needs to be done. The comparison between the gross beta and gamma-ray spectrometry analysis can be a proper way to improve the method used in decommissioning process for liquid effluents monitoring.

Key words: nuclear reactor decommissioning, liquid effluents, activity concentration, gamma-ray spectrometry, gross beta activity measurement.

1. INTRODUCTION

The paper presents studies regarding the evaluation of the radionuclides inventory contained in the liquid effluents which results from decommissioning of the VVR-S nuclear research reactor owned by Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH) Bucharest-Magurele, Romania. The effluents derive from two sources. The first source is represented by the decommissioning of the systems, structures, equipment's and components

(SSEC) of the nuclear research reactor (*e.g.* the primary and secondary circuit, the active zone, the demolition of the reactor block, the hot cells, the degasser, the underground structures (pond and pipes), etc.). The second source is represented by the pools from the Nuclear Spent Fuel Storage (DCNU) that contains radioactive water. The operation history and decommissioning process of this reactor are presented in details by Tuca and coauthors [1]. The reactor is in its last decommissioning phase which consists of: (i) dismantling and demolition of internal parts of reactor block and of cooling pond for nuclear spent fuel; (ii) demolition and dismantling of biological protection of de-aerator; (iii) decommissioning of hot cells; (iv) removing of the radioactive drains far to the buffer tank; (v) the final radiological characterization of the site.

The radiological characterization was performed for the radioactive liquid effluents arising from reactor decommissioning operations (*e.g.* (i)÷(iv) activities) – source 1 – and from the decommissioning of Nuclear Spent Fuel Storage (DCNU) – source 2. The characterization involves the identification and determination of individual concentrations of radionuclide constituents of radioactive effluents by gamma-ray spectrometry analysis and gross beta activity measurements. The effluents arising from decommissioning activities are released through a special collection system of the nuclear reactor (RN) in a buffer tank of 30 m^3 volume, where they are temporarily stored and then transferred into a buffer tank of 300 m^3 from the treatment plant for radioactive waste from IFIN-HH. After treatment, the water is unconditionally released into the Ciorogarla River respecting the derived emission limits approved by the National Commission for Nuclear Activities Control (CNCAN).

The short-term (one year) storage of the nuclear spent fuel assemblies consist of a cooling pond with 8 m^3 storage capacity containing deionized water, located in the reactor building. For the extension of storage capacity and long term storage of nuclear spent fuel in deionized water, other four pools were built, located into the Nuclear Spent Fuel Storage building (DCNU) [2], away from reactor building. Each pool consists of a concrete tank with 1.5 m thickness (biological protection) lined with $AlMg_3$ (approx. 964 kg) and covered with three cast iron lids. Outside, the pools are protected against corrosion with two felt moistened in bitumen. Inside, the pools contain an Al rack (60 kg) forming a network of 60 cells for nuclear spent fuel assemblies storage. There are also other materials used for pools construction such as: a vertical Al plate (29 kg) for the separation of the handling area; two Al pipes with 30 mm and 50 mm diameter for emptying and/or filling to maintain the water at a proper level; channels for input/evacuation of air. Each pool contains a water column with a minimum of 3.75 m thickness (~7.21 m^3). Pool no.1 was designed for emergency storage of nuclear spent fuel and the other three pools were designed for normal operating situations. Currently, the pools store have just contaminated water because the nuclear fuel was repatriated into the Russia Federation, first in 2009 (S-36) and then in 2012 (EK-10) [2]. After

emptying and decontaminating the pools, these can be used for interim storage purposes of aluminum and graphite wastes resulted from VVR-S nuclear research reactor decommissioning [3].

2. MATERIALS AND EQUIPMENT

The radiological characterization of liquid effluents from the two sources was made in order to transfer them to the treatment plant and to decommission these objectives. Thus, water samples were taken from the two sources in order to determine the activity concentration. The measurements were carried out in parallel in several laboratories from IFIN-HH notified by CNCAN, in compliance with EN/ISO IEC 17025:2005, according to the specific work procedures [4, 5, 6, 7]. Laboratories from IFIN-HH that were involved in this study and the equipment used for measurements are presented in Table 1.

Table 1

Laboratories and equipment used

Laboratory	Equipment
Radiologic Characterization Laboratory (LCR) from Reactor Decommissioning Department (DDR)	Gamma-ray spectrometry system consists of a GEM60P4-95 high-purity germanium coaxial detector (HPGe), a DSPEC jr.2.0 digital signal processing and a low-background shield. The main performance specifications of the detector warranted by the producer are: relative efficiency 60%, resolution (FWHM) 1.95 keV, peak-to-Compton ratio 70:1, peak shape (FWFM/FWHM) 3.0, all evaluated at 1.33 MeV peak of ^{60}Co. The support stand and the shield jacket are made from low carbon steel and a graded lined of copper and tin layers is provided for the suppression of lead X-rays. Maestro-32 and GammaVizion-32 software are used for spectra acquisition and analysis.
Spectrometric Analysis Laboratory (LAS) from Radioactive Waste Management Department (DMDR)	Gamma-ray spectrometer from CANBERRA with a REGe semiconductor detector with relative efficiency 40% and a multichannel analyzer model DSA 1000. Gamma-ray spectrometer from CANBERRA contain a HPGe with relative efficiency 30%; a multichannel analyzer model DSA 1000 with a specially designed shield consisting in 10 cm lead, 1 mm cadmium and 2 mm copper; and the spectral analysis system, operation and analysis software.
Spectrometric Analysis Laboratory Gamma Spec (LGS) from Nuclear Physics Department (DFN)	Gamma-ray spectrometer, in low background, contains an HPGe, AMETEK ORTEC (USA) with measurement geometry Marinelli type. The main performance specifications of the detector warranted by the producer are: relative efficiency 30%, resolution 2.1 keV to 1332 keV energy of ^{60}Co, placed in a Pb shield (10 cm thickness). Measurements can be performed by gamma spectrometry for: radionuclide in environmental samples; artificial radionuclide (*e.g.* ^{137}Cs, ^{60}Co, ^{241}Am, etc.); natural radionuclide (*e.g.* ^{238}U radioactive descendant's series, ^{226}Ra and ^{232}Th, ^{235}U, ^{40}K, ^{7}Be); solid sampled

Laboratory	Equipment
	with mass of ~30–200 g; liquid samples with mass of 100 g – 1 kg.
Personal Dosimetry and Environment Laboratory (LDPM) from Life and Environmental Physics Department (DFVM)	9300-PG, GFR, PROTEAN EQUIPMENT for the gross alpha and beta activity measurements with flow gas, window less proportional counter. Installation for alpha-beta-gamma activity gross measurement, with automatic exchange of samples, Canberra USA, S5XLB-G type, with measuring range of 0.01...10.000 Bq for alpha channel, 0.06...10.000 Bq for beta channel and up to 10.000 Bq for gamma channel. Both equipments were calibrated with a ^{90}(Sr+Y) standard source. The uncertainties were less than 10%. Equipment for primary processing of samples: evaporation, drying and calcinations. Analytical balance Model WAX 220, PARTNER, 10^{-4} measurement accuracy.

3. METHODS

Gamma-ray spectrometry method and gross alpha-beta activity measurement method were used in parallel for activity measurement.

3.1. GAMMA-RAY SPECTROMETRY

3.1.1. Sampling

Water samples were taken several times from two locations: (i) the buffer tank which collect the radioactive water resulted from the reactor systems decommissioning (*e.g*. primary and secondary circuit, biological protection, wet cutting of reactor components, tools, equipment's and workers decontamination, and cooling pond for nuclear spent fuel) and (ii) DCNU pools. First, in 2013 and 2014, samples of 0.5 l of liquid effluent were taken from the buffer tank in order to characterize them for the transfer of radioactive liquids to the treatment plant and from the pools of DCNU for continuous monitoring of activity content. For comparative analysis of the results, the samples were measured by LAS and LGS laboratories. Secondly, in 2015, the DCNU decommissioning activities (*e.g*. the transfer of liquid radioactive inventory to DMDR, the removal of aluminum racks and pools cleaning) have imposed the necessity of colleting water samples from each pool of DCNU, for radiological characterization purpose. Thus, two samples of water were collected, one at the pool bottom, close to the rack, and the other from 1 m below the water surface. A third sample of the mixture (water and sludge) from three different points was taken in order to assess the entire activity concentration of the pool. Due to the special physic-chemical behavior of some radionuclides, the water-soluble compounds and those filed in sludge were analyzed by LCR, using gamma-ray spectrometry method [8]. Thus, the sediment

and water were analyzed separately for each pool; on this purpose, the mixture was decanted and evaporated to dryness.

3.1.2. Sample preparation

The water samples measured in 0.5 l cylindrical plastic containers were set directly upon the end cap of the detector in the lead castle. In order to obtain the solid residue, the water samples were collected in 5 l plastic containers and then allowed to settle for several days. After settling, the water was pumped out and the sludge was allowed to evaporate to dryness. The residue was collected in Sarpagan type containers. The samples were placed in the lead castle directly upon the end cap of the detector and measured by gamma-ray spectrometry.

3.1.3. Samples analysis

To analyze samples by LCR, LAS or LGS laboratories from IFIN-HH, the energy calibration of the spectrometric systems was performed with standard sources, products by Radionuclide Metrology Laboratory (RML) of IFIN-HH. The sources were located on the symmetry axis of detector and they covered the energy domain of interest (50÷2000 keV) [9, 10]. The spectrometers calibration was done by the same laboratory, RML [11].

3.2. GROSS ALPHA-BETA ACTIVITY

The natural and artificial radioactive contaminants like ^{60}Co, ^{134}Cs, ^{137}Cs, ^{154}Eu, ^{235}U, ^{238}U and ^{241}Am, resulted from decommissioning activities and identified through gamma-ray spectrometry method are highly-energetic alpha and beta emitters. The problems arise due to the fact that the ^{89}Sr and ^{90}Sr radionuclides are pure beta emitters and they cannot be measured by gamma-ray spectrometry; alpha emitters require a double assessment due to gamma energy and emission intensity. Thus, the activity concentration of alpha and beta emitters identified in liquid effluents is determined by direct measurement of samples, using gross alpha-beta activity measuring method. Gross beta activity measurements of solid samples obtained after physical processing having a relatively low surface density (mg/cm^2) and expression of the results in equivalent activity of the reference radionuclide (^{90}Sr, for which the calibration of the measuring system was done) were performed. The measurements were done in order to determine ^{90}Sr content in the mixture (sludge) [12]. ^{90}Sr is a pure beta decaying radionuclide; a secular equilibrium is established with its daughter ^{90}Y, a pure beta emitter too. ^{89}Sr was neglected in previous assessment due to the short half live (50.57 d) and complete decay after the nuclear spent fuel removal.

The standard method consists of chemical separation from the sample and the measurement of the pure ^{90}(Sr+Y) sample in equilibrium conditions. Because

LDPM does not perform chemical separation, an indirect method was used as alternative. Gross alpha measurements were not performed because all alpha emitters were measured by gamma-ray spectrometry.

3.2.1. Sampling

In order to measure the gross beta activity of samples from the DCNU pools and buffer tank, there were used samples with known mass, conditioned for gamma-ray spectrometry measurements.

3.2.2. Sample preparation

The samples were gravimetrically prepared and dried according to the proper procedure [7]. Then, they were transferred and uniformly distributed over the surface of a stainless steel plate with 50 ± 0.2 mm diameter. To avoid the spread of the solid residue in the proportional counter volume, the samples were fitted with alcoholized water and then measured into P10 gas atmosphere.

3.2.3. Working procedure

In order to estimate the ^{90}Sr content of liquid effluents only gross beta determinations were performed as follows. The counting rates, N_β, were recorded and corrected for the background contribution. The equivalent beta ^{90}Sr activity was calculated applying eq. 1:

$$N_\beta = \varepsilon \times s \times f \times A_{echiv} \tag{1}$$

with

$$N_\beta = \varepsilon_{etalon} \times s \times f \times A_{^{90}Sr},$$

where: N_β is the counting rate for sample and standard source corrected for the background contribution; A_{echiv} is the sample activity; ε is the detection efficiency (the same for standard source and sample); s is the beta radiation emission intensity (the same for standard source and sample); f is the self-absorption factor of radiations in thick samples. There was considered that the total beta activity was expressed in equivalent activity of ^{90}Sr, consisting of known beta-gamma emitters activity and ^{90}Sr activity which will be determined and ε = 0.425 s^{-1}/Bq. In this case, eq. 1 allows calculation of the equivalent activity, starting from N_β determined according to eq. 2:

$$N_\beta = 0.425 \times \sum_i^n f_i A_i + 0.425 \times 2 \times f \times A_{^{90}Sr}, \tag{2}$$

where: f_i is the individual self-absorption factor of radionuclides from beta-gamma emitters mixture and f is the mean self-absorption factor corresponding to the mixture to the equilibrium ^{90}(Sr + Y). For thin samples $f_i = f = 1$ and for thick samples, the self-absorption factor f is calculated using eq. 3:

$$I = I_0 e^{-\frac{\rho_x}{R_{\max}}}, \tag{3}$$

where: ρ_x is sample thickness [mg/cm^2], obtained according to eq. 4 and $R_{\max}$ is the maximum range of beta radiation [13].

$$\rho_x = \frac{m_{sample}}{S_{plate}}, \tag{4}$$

where: m_{sample} is the mass sample [mg]; S_{plate} is the plate surface [cm^2].

4. RESULTS AND DISCUSSIONS

4.1. GAMMA-RAY SPECTROMETRY MEASUREMENTS

Table 2 presents the total activity evaluated for each pool, which was obtained by summing of all constituent radionuclides activity, corresponding to the volume of liquid contained into the pool (34.8 m^3 for source 1 (buffer tank of reactor) and 7.21 m^3 for each of the DCNU pools respectively). They were calculated as a product between concentration of activity (Bq/l) and effluent volume of each pool. The measurement uncertainties are between 3.0 ÷ 16.5%.

The radioactive liquid effluents arising from decommissioning reactor contain fission products, activation products and actinides which are gamma emitters. The fission products can contaminate the water in case of a nuclear fuel assembly defect. The experimental data showed that the radioactive effluents arising from source 1 contain ^{54}Mn, ^{60}Co, ^{108m}Ag, ^{134}Cs, ^{137}Cs, ^{235}U, ^{238}U radionuclides. ^{108m}Ag, ^{235}U and ^{238}U come from primary and secondary circuit of the reactor. ^{54}Mn radionuclide occurs in nuclear reactor by ^{54}Fe(n,p)^{54}Mn reaction [14]. ^{60}Co is an activation product and occurs through activation of cobalt in the metallic structure of nuclear reactor. ^{137}Cs is a fission product which may occur as a result of fuel leakages. Both of them have a higher activity concentration compared to others radionuclides, due to the provenience. ^{108m}Ag and ^{110m}Ag radionuclides are activation products of ^{107}Ag and ^{109}Ag [15]. They are produced in nuclear reactor for normal and abnormal operation conditions and are mainly in pipes alloy membranes (Ag-In-Cd) and the reactor primary coolant circuit. This is the main reason that reactors are equipped with two or three cooling loops

connected by a heat exchanger. Although ^{110m}Ag is the major constituent, the high half-life of ^{108m}Ag radionuclide determines to analyze it carefully in order to store the radioactive waste from decommissioning.

Table 2

The conservative values of the activity for liquid effluents

Radio-nuclide	Activity (Bq)				
	Source 1	Source 2 (DCNU)			
	Buffer tank	1st pool	2nd pool	3rd pool	4th pool
^{60}Co	2.76E+07	4.59E+04	4.61E+04	2.80E+06	8.45E+05
^{134}Cs	8.71E+05	5.05E+03	7.21E+03	2.67E+04	8.65E+03
^{137}Cs	4.26E+07	1.67E+05	6.09E+05	3.16E+08	4.70E+06
^{108m}Ag	1.11E+05	2.43E+04	8.57E+03	1.57E+06	4.74E+04
^{152}Eu	-	-	6.26E+03	2.75E+05	3.99E+04
^{154}Eu	-	-	1.47E+04	1.36E+05	2.05E+04
^{54}Mn	8.07E+04	-	-	-	-
^{235}U	2.39E+05	-	-	-	-
^{238}U	2.94E+06	-	-	-	-
^{241}Am	-	-	-	-	2.81E+05
Total	7.29E+07	2.42E+05	6.92E+05	3.21E+08	5.94E+06

The activity inventory of source 1 (Table 2) is comparatively analyzed with the similar one presented by Tuca and coauthors in their paper [1] that shows that ^{54}Mn occurs as a constituent of liquid effluents and there is a significant increase in activity concentration of 3.52 times for ^{60}Co, 1.19 times for ^{134}Cs and 3.38 time for ^{137}Cs, due to the reactor decommissioning progress and introduction of new quantities if liquid waste in this source. For the other radionuclides, the concentration is the same. These effluents do not meet the criteria for unconditional release into the environment because they have the activity concentrations higher than derived emission limits (DEL). The results regarding concentration of activity for liquid effluents provided from both sources was presented in NSR-01 [16]. Note that these values are lower than those mentioned in Table 2, because the author didn't take into account the progress of decommissioning activities.

The existence of ^{60}Co, ^{134}Cs, ^{137}Cs, ^{108m}Ag, ^{152}Eu, ^{154}Eu and ^{241}Am radionuclides that arise from sources 2 (DCNU) – planned to be used as a repository for aluminum and graphite radioactive waste – allow the assessment of the activity inventory of liquid effluents contained in the pools. There was observed that ^{152}Eu and ^{154}Eu radionuclides occur in the 2nd, 3rd and 4th pools and, in addition, ^{241}Am in the 4th one. The explanation for the occurrence of ^{152}Eu and ^{154}Eu is that water samples were collected from the top of the pool and sludge samples were collected from the bottom of pools. Europium was accumulated in the sludge due to the high adhesion of its salt at this component. The significantly higher concentration of activity in the 3rd pool is because during the operation of DCNU many leakages from the fuel assemblies were detected and confirmed, since 1994, by the presence

of ^{137}Cs radionuclide. In Fig. 1 there can be observed that in February 2007 a maximum total activity of 4.9E + 07 Bq was registered. For the same radionuclide, in 2015, the activity of the sludge sampled from the rack determined by gamma-ray spectrometric measurements was 3.16E + 08 Bq; this value is comparable with that of water samples. ^{241}Am radionuclide was expected to be detected in this pool because is a prevalent isotope in the nuclear waste. Its occurrence in the 4th pool is explained by the possibility of a fuel failure unidentified during the operation period.

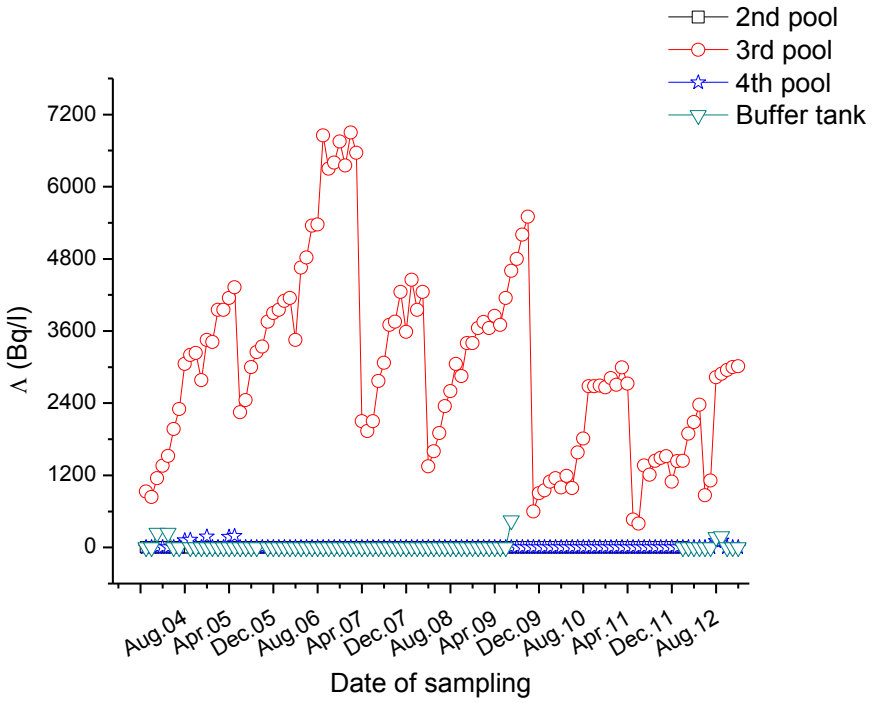

Fig. 1 – ^{137}Cs activity concentration in DCNU during 2004–2012.

4.2. GROSS BETA MEASUREMENTS

In order to estimate the ^{90}Sr content of liquid effluents, there were performed gross beta activity determinations, according to the proper working procedure. The ^{90}Sr activity uncertainty can be evaluated in the range of 30÷60% due to the counting statistics, gamma emitters' inventory uncertainties and approximations for relatively thick samples measurement. Eq. 2 was used for ^{90}Sr activity calculation with f and f_i values evaluated taking into account the maximum ranges of beta

radiation in the measured samples matrix. The obtained values are presented in Table 3.

Table 3

Gross beta activity values measured and calculated as ^{90}Sr equivalent and comparison with ^{137}Cs

Location of measurement	ρ_x (mg/cm^2)	N_β (s^{-1}/l)	$A_{^{90}Sr}$ (Bq/l)	$\frac{A_{^{90}Sr}}{A_{^{137}Cs}}$
Pool 1	17.707	1.66E+01	4.35E+00	0.18
Pool 2	3.412	2.52E+02	1.73E+02	2.04
Pool 3	2.141	3.71E+04	2.18E+04	0.49
Pool 4	18.751	3.24E+02	8.20E+01	0.12
Buffer tank	1.783	1.10E+04	1.18E+04	2.04

There was observed that the lowest values of $\frac{A_{^{90}Sr}}{A_{^{137}Cs}}$ ratio are for the thick samples. It can be considered that the self-absorption effect was underestimated, so the method can be further improved. However, the ^{90}Sr activity was found comparable with ^{137}Cs activity for sludge samples. The evaluation of ^{90}Sr activity becomes important as it is known that ^{90}Sr occurs with ^{137}Cs in the burned fuel and has a high radiotoxicity for internal exposure, at least two times higher than that of ^{137}Cs [17]. The comparison between the gross beta and gamma-ray spectrometry analysis is a proper way to improve the method used in decommissioning process for the liquid effluents monitoring.

4.3. THE FUTURE USE OF RESULTS

The values presented in Table 2 and Table 3 are used for comparative analysis of existing activity values with derived emission limits in force and for radiological characterization and risk occurrence assessment during the liquid waste transfer operation. The comparative analysis of activity concentrations of each radionuclide with the derived emission limits in force, calculated for nuclear reactor decommissioning by Tuca and coauthors [1] indicates that the radioactive liquid effluents arising from the two sources do not meet the criteria for unconditioned release into the environment (Ciorogarla River). Therefore, it is necessary to perform the radiological characterization of liquid effluents and to transfer them to DMDR for treatment.

The effluents are mechanically handled by workers, using stainless steel containers having a 1.2 m^3 capacity. Each container must be accompanied by an analysis report. In these circumstances, it is necessary to assess the radiological risk of workers for two different situations. The 1st situation is for normal working conditions, when the workers are exposed to the external irradiation during the

container transfer. The risk should be assessed based on dose rate of the container walls at different distances from container and time exposure. The dose rate can be calculated using the activity of the liquid effluents and compared with direct measurements made with calibrated dose rate-meters. The 2nd situation is for abnormal working conditions – during the transfer – when the container can fall from the handling machine and an uncontrolled discharge a 1 m^3 of radioactive effluents occurs. In this case, the workers can be exposed both externally and internally. It requires an internal contamination control of workers, according to domestic and international legal provisions. There also exists the risk of working, and adjacent, surfaces contamination which constitutes an additional risk for persons that are not involved in operations. Furthermore the surface decontamination operation, involves an additional exposure of workers.

5. CONCLUSIONS

There was developed a working methodology for radioactive inventory identification of two sources of liquid effluents implied in the decommissioning process. The activities of all radionuclide components of radioactive liquid effluents arising from buffer tank and DCNU pools were measured by gamma-ray spectrometry and beta global methods. Comparing with previous evaluations, the gamma radionuclides inventory was qualitatively increased by ^{54}Mn and ^{241}Am radionuclides detection and quantitatively due to the progress of decommissioning. There has been found that ^{108m}Ag, ^{152}Eu and ^{154}Eu radionuclides are concentrated in the sludge, imposing the recalculation of DEL for liquid effluents. For the first time, ^{90}Sr activity was evaluated through gross beta measurements and was found that is comparable with the ^{137}Cs activity for sludge samples. The comparison between the gross beta and gamma-ray spectrometry analysis is a proper way to improve the method used in decommissioning process for the liquid effluents monitoring. The necessary data both for a controlled transfer of the liquid effluents to the treatment plant and for the assessment of the radiological risk to workers during operation were obtained.

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