METHODS FOR MEASURING THE RADIATION CHARACTERISTICS OF WASTE

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For radioactive waste generated at nuclear facilities, identification of RW radiation characteristics during its conditioning in accordance with waste acceptance criteria is considered as a relevant task to accomplish. JSC VNIINM has developed a Unified Guidance for Measuring the Radiation Characteristics of Waste. The Guidance specifies the pre-measurement and measurement procedures, including those applied to the waste generated during decommissioning, and standard procedures for measuring the specific activity of radionuclides in packages and in samples considering sampling flowchart for solid RW in the form of construction waste, plastic compound, fragments of equipment, PPE, soils. Algorithms allowing to check the measurement accuracy using certified items were developed.

Keywords: *measuring procedure, radiation characteristics, radioactive waste, measurement accuracy control.*

Enterprises applying radioactive substances and nuclear materials during their operation generate solid and liquid radioactive waste of various morphologies: soil, equipment, metal, construction waste, plastic compound, personal protective equipment, decontamination solutions, etc. By December 31, 2018 the inventory of RW accumulated on the territory of Russia amounted to $5.65 \cdot 10^8$ m^3, of which $5.53 \cdot 10^8$ m^3 are categorized as nuclear legacy waste [1].

In keeping with OSPORB-99/2010 provisions [2], waste is divided into the following categories according to the specific activity of radionuclides:

- very low-, low-, intermediate- and high-level solid radioactive waste (hereinafter SRW) and low-, intermediate- and high-level liquid radioactive waste (hereinafter LRW);
- restricted use materials contaminated with technogenic radionuclides the management of which requires appropriate radiation control;
- industrial waste not requiring any radiation control.

Nuclear decommissioning generates large amounts of waste with various morphology and different radionuclide ratios [3].

Organization operating the site where the waste was generated is responsible for the management and pre-disposal treatment of waste with a radionuclide content being higher than the established standard values. The only exception is the waste containing nuclear materials that is allowed to be exclusively under federal ownership [4].

RW disposal costs covered by the organization depend on RW class specified based on the specific activity of the radionuclides contained in the waste.

Radiation characteristics of RW shall be identified at all stages of waste management at nuclear facilities (NF), namely, radionuclide composition and specific activity of radionuclides. IAEA recommendations [5] provide for the following procedure that can be applied to identify the radiation characteristics of facilities: direct in-situ measurements by non-destructive testing methods, laboratory measurements of representative samples, radionuclide vector method (ratios of radionuclide activities) [6]. There is a wide variety of destructive and non-destructive testing methods, as well as sampling and sample preparation procedures [7, 8]: these are selected based on the morphology, radionuclide composition and the expected specific activity of the waste material.

In keeping with Federal Law No. 102 On the Uniformity of Measurements [9], measurements performed as part of activities implemented in the field of atomic energy use shall be based on certified methods.

Methods applied at enterprises to measure the specific activity of radionuclides by alpha, beta, gamma spectrometry and radiometry often do not comply with the regulatory requirements stated in SC Rosatom's Order of October 31, 2013. No. 1/10-NPA [10]. Sections on the quality control of measurements are missing there similar to error characteristics depending on the ranges of measured values. It also provides no information on measurement procedures specifying the flowchart to be followed during the preparation of counting samples with different morphologies and the conditions suitable for the measurement of their radiation characteristics [6].

According to GOST R 8.932-2017 [11], if measurement operations and relevant rules for their implementation are similar for different items subject to such measurements and/or different but similar measurement conditions and for several enterprises in the industry, then it should be considered feasible to develop standard measurement methods in the form of an industrial guidance regulating general rules for all similar facilities and/or conditions. Metrological characteristics for such typical methods shall be specified in the widest possible range of measurement conditions. Accuracy indices of operating procedures are identified based on experiments or calculations taking into account relevant area of application.

Sampling and sample preparation procedures, as well as measurement tools and methods shall be unified and formally approved at the industry-wide level as regulations and a system of standard measurement procedures allowing to optimize RW management procedure on and from its generation, to reduce its volume and to exclude the possibility of managing materials assigned to RW category or restricted use materials as industrial waste. Reliability in the measurements of radiation waste characteristics can be attained through the development of standard procedures for accuracy control taking into account relevant specific features of the measured items.

JSC VNIINM was assigned with the following tasks to regulate the measurement of waste radiation characteristics:

- to develop requirements to measurement conditions;
- to develop recommendations on measurement method application;
- to establish requirements for measurement methods and tools enabling their unification;
- to establish requirements for measurement processing and presentation;
- to develop measurement accuracy control algorithms.

The work was focused on the development and approval of [6]:

- Unified industry-wide guidelines on the Measurement of Radiation Waste Characteristics;
- standard techniques allowing to measure the specific activity of radionuclides in samples and waste containers by means of gamma spectrometry and liquid scintillation spectrometry.

Unified Industry-Wide Methodological Measurement Guidelines (UIMMG) for Radiation Waste Characteristics

The Guidelines for Radiation Waste Characteristic Measurements at the enterprises of the State Atomic Energy Corporation Rosatom are seeking to establish a general procedure for measuring the radiation characteristics of waste, including the waste from nuclear decommissioning.

UIMMG present the following instructions and recommendations on the measurement of radiation waste characteristics:

- selection of methods and equipment, processing and presentation of measurement results;
- selection and application of waste sampling procedures (sampling location, quantity, volume and size of material element) for construction waste, plastic compound, elements of equipment, PPE, soil;
- instrumental control of waste characteristics using α- and β-radiometry with portable and stationary radiometers, liquid scintillation and gamma spectrometry.

UIMMG systematizes data on the main sources of errors in the measurements of radiation waste characteristics, presents their quantitative assessment and their minimization methods.

Compliance with UIMMG requirements during the measurement of radiation waste characteristics will help to minimize the amount of RW and materials contaminated with radioactive substances, excluding RW ingress into the category of industrial waste or restricted use materials.

The approaches, procedures and algorithms described in the UIMMG should be used in the development of nuclear decommissioning programs allowing to optimize relevant plans regarding the measurement of radiation waste characteristics.

UIMMG will be enforced by an order approved at the industry level with their application extended to the operations performed at the enterprises of the State Atomic Energy Corporation Rosatom.

Typical measurement methods

The essence of typical measurement procedures must comply with the requirements set in GOST R 8.932-2017 [11] and the order of the State Atomic Energy Corporation Rosatom of October 31, 2013. No. 1/10-NPA [10].

Section on the accuracy control is mandatorily included into the section discussing measurement procedures.

Measurement accuracy control should verify whether metrological characteristics of the measurement method can be deemed constant over time.

Types of accuracy control can be summarized as follows [12]:

- use of reference samples or certified items;
- additive method;
- dilution method;
- combination of dilution and additive methods.

The latter three methods are applicable only under laboratory research and cannot be implemented if the waste is accommodated in packages and during direct in-situ measurements. In the latter cases, method of reference samples or certified items is applied for measurement accuracy control purposes. In keeping with relevant provisions of Federal Law No. 102 On the Uniformity of Measurements [9], a standard sample is a sample of a substance (material) characterized by values identified during relevant tests representing the composition or properties of this substance. A standard sample is identical to the controlled one possessing all of its physical and chemical characteristics. Certified items are items providing the function of reference samples, but not being them [10]. Certified item allows simulating individual characteristics of controlled item.

Due to wide variety of materials, packaging geometry and physical and chemical properties of waste, fabrication of standard samples considering all possible practical options seems to be infeasible. Development of several certified items allowing to confirm and control the correctness of applied methods was proposed to monitor the accuracy of measured specific radionuclide activities.

Gamma spectrometry is considered as the main in-situ method used to measure the specific activity of waste radionuclides in packages or studied facilities. Available purpose-developed software for calculating the registration efficiency of gamma quanta assumes the use of various measurement geometry templates: cylinder (canister), parallelepiped, cone, surface, etc. Geometry templates used in waste characterization involve cylindrical vessels (cans, drums) and rectangular items (boxes, containers) made of various materials.

JSC VNIINM has developed algorithms to monitor the accuracy associated with the measurement of radionuclide specific activities for waste accommodated in various packages using mathematical modeling and reference radionuclide sources such as OSGI (reference spectrometric gamma source) and ORR (reference radionuclide solution).

Measurement accuracy control algorithms are designed for two cases:

- measurement of small items (up to 100 l);
- measurement of large volume packages (drums, containers).

The essence of accuracy control in case of small items can be summarized as follows: certified items are manufactured as small packages (cylindrical reservoir, box, canister) filled with material the density of which should be similar to the one of the studied item. The material inside the reservoir is impregnated with ORR-based solution. To control the accuracy in a wide range of energies, the following ORR set is recommended: ^{241}Am (59 keV), ^{137}Cs (662 keV), ^{60}Co (1173 and 1332 keV).

Accuracy control is considered to be completed successfully if for each radionuclide in the certified item the modulus of the difference between the measured and passport activity value does not exceed the square root from the sum of the squares of the statistical measurement uncertainty and the statistical activity error.

When it comes to large volume measurements, computational methods are used to calibrate the spectrometer according to the detection efficiency of gamma quanta characterized with various energies. The methods used in the software are based on the integration of response functions (specific activity) over the volume of the investigated item from a “point” source (part of the investigated item). In this case, detection efficiency calculation algorithm takes into account the absorption of gamma quanta by the source material and the material of the package wall, as well as the distance between the “source” and the “detector” and the position of the detector relative to the source. Figure 1 shows an algorithm for a cylinder allowing to integrate the response functions over its volume, where Q is the point of the package (canister) surface on the “source - detector” line connecting the detector point (point D) and the “point” source inside the container (point K).

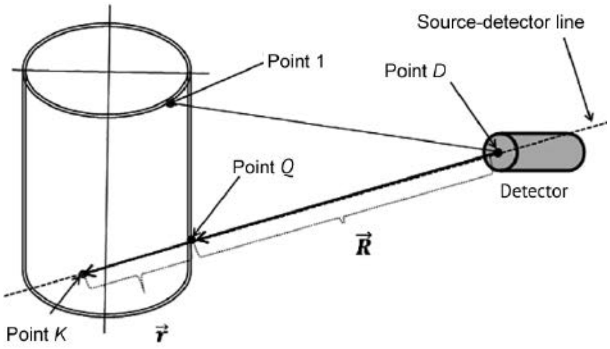

Figure 1. Integration by packaging volume

The following equation can be basically used to calculate the specific activity using integration over the package volume:

$$A_m = \frac{1}{m} \iiint_V \tilde{A}_V \left(\vec{r}, \mu, \varepsilon\right) dV,$$

were m stands for the weight of the object (waste) in a package, kg;
$\tilde{A}_V$ is the volumetric activity of the radionuclide, Bq/cm identified by the software of the gamma spectrometer according to the measurements in k "point" areas;
$\vec{r}$ is the radius vector of a “point” source (point K) inside the container relative to point Q (Figure 1);
$\mu(\vec{r})$ stands for mass gamma-quanta attenuation coefficient taking into account the source material, external environment and packaging walls;
$\varepsilon(\vec{R}, \vec{r}, \mu)$ is the dependence of registration efficiency for gamma quanta of a certain energy level calculated for “point” areas, $\vec{R}$ is the radius vector defining the coordinates of Q point relative to the detector (point D).

For container measurements, point sources such as OSGI were proposed for accuracy control purposes.

A “point-source-behind-two-layers” model is built using purpose-developed software (ISOCS, EffMaker, etc.): its first layer simulates the container wall, and the second - the radiation source material (waste material). Point sources such as OSGI are located behind two layers (Figure 2).

Several positions "detector — distance to layer" are selected based on the assumed overall dimensions of the package (the figure shows two positions). It is

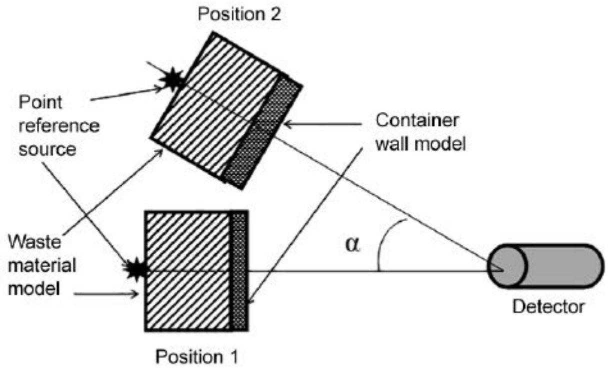

Figure 2. Model of a “point-source- behind-two-layers”

recommended to choose an axial orientation of the detector (position 1) and 2—3 positions at a certain angle α according to the package dimensions.

To measure the reference source activity, the following model parameters are introduced into registration efficiency calculation conditions given the software applied:

- "point source" measurement geometry; reference source material is indicated in accordance with the passport characteristics;
- distance "detector — object", "atmospheric air" is indicated as the measurement medium; if collimator is applied, the distance should ensure the full incidence of gamma quanta from the object into the solid angle of the collimator;
- thickness d_{st} and characteristics (type, density, etc.) of the packaging wall material based on the technical characteristics of the packaging (drums, containers);
- thickness D_{mat} and characteristics (type, density, etc.) of the material simulating the measured waste;
- environmental parameters (temperature, humidity, pressure) to account for the influence of gamma quanta absorption by the atmospheric air.

Reference point source activity is measured using specialized software. ^{152}Eu is recommended as a reference point source with its gamma lines falling into the range from 122 to 1408 keV, or several other sources with gamma quantum energies corresponding to the energies of radionuclides present in waste materials. The recommended activity of the source accounts for 10^5—10^7 Bq.

Accuracy control is considered to be successfully completed if the modulus showing the difference between measured and passport reference source activities does not exceed the square root of the sum of squares presenting the statistical measurement uncertainty and the statistical error of activity for all positions of the point source. Compliance with this condition confirms the reliability of the calculation model used.

Non-destructive gamma spectrometry method is used for prompt detection of radiation waste characteristics. Its application is somewhat limited as it's unable to measure hard-to-detect radionuclides present in the waste (for example, ^{3}H, ^{14}C, ^{36}Cl, ^{63}Ni, ^{90}Sr, $^{234, 238}$U, $^{239, 241}$Pu). Gamma radiation emitted during the decay of hard-to-detect radionuclides is either absent or its intensity or energy is insufficient for such registration by gamma-spectrometry methods [6].

Liquid scintillation spectrometry method was proposed for prompt detection of hard-to-detect radionuclide content in the waste (for example, isotopes of uranium and plutonium): it allows an express analysis of hard-to-detect radionuclides by the "screening" method without complete radiochemical separation of radionuclides during the preparation of the counting sample [13, 14].

The standard methods developed to measure the specific activity of radionuclides in samples and containers with waste by the methods of gamma and liquid scintillation spectrometry comply with GOST R 8.932-2017 [11] and the Order of the State Corporation Rosatom of October 31, 2013. No. 1/10-NPA [10]. Typical methods are formalized as industrial guides and are specified in the industry register of the State Corporation Rosatom. These methods were subject to metrological review based on experimental measurements involving the assessment of factors influencing the degree of measurement errors and the ratio between their random and systematic components.

Table 1 provides the list of developed methods formalized as industrial guides.

In 2020, VNIINM will complete the development of a Standard Procedure for Applying the Radionuclide Vector Method in the Identification of Hard-to-Detect Radionuclides (^{90}Sr, $^{234, 238}$U, $^{239, 241}$Pu) in SRW Based on Reference Radionuclide (^{60}Co, $^{134, 137}$Cs, ^{237}Np, ^{235}U, ^{241}Am) Specific Activity Measurements.

The essence of the "radionuclide vector" method consists in establishing correlations and calculating the scaling factors between the content of hard-to-detect and easily-detectable radionuclides. Specific activity of hard-to-detect radionuclides is measured by non-destructive methods (gamma spectrometry) based on the specific activity measurement performed for easily detectable radionuclides using the obtained scaling factors [15].

The "radionuclide vector" method was tested by VNIINM experts at FSUE RADON facilities when radiation characteristics of typical RW streams containing hard-to-detect radionuclides were identified [16].

This method has proved its feasibility in case of large waste streams with a constant radionuclide ratio.

Table 1. Standard methods used to measure RW specific activity

Mothod ID #	Name
OI 001.871-2019	Specific activity measurements for gamma-emitting radionuclides in the waste in the form of construction waste, plastic compound, equipment elements, PPE, soils in packages and standard containers with a volume of up to 3.1 m^3
OI 001.872-2019	Preparation of counting samples and specific activity measurements for gamma-emitting radionuclides by gamma-spectrometry method for waste samples in the form of construction waste, plastic compound, equipment elements, PPE, soil
OI 001.873-2019	Preparation of counting samples and specific activity measurements for plutonium isotopes by liquid scintillation-spectrometry method for waste samples in the form of construction waste, plastic, equipment elements, PPE, soils
OI 001.874-2019	Preparation of counting samples and specific activity measurements for uranium isotopes by liquid scintillation-spectrometry method for waste samples in the form of construction waste, plastic compound, equipment fragments, PPE, soil
OI 001.875-2019	Preparation of counting samples and specific activity measurements for Sr-90 by liquid scintillation-spectrometry method for waste samples in the form of construction waste, plastic compound, equipment fragments, PPE, soil

Standard methods are seen as the basis for the development of working methods accounting for the requirements of organizations: applied measuring equipment, morphology and radionuclide composition of waste, specific features of performed operations. These methods can be either developed by the organization itself and by contracted outside experts.

Certification of measurement techniques (methods) falling under state regulation of measurement uniformity is carried out by legal entities and individual entrepreneurs having appropriate accreditation in the field of measurement uniformity assurance.

Conclusions

Based on evaluated scientific and technical information, requirements of Russian regulatory legal acts in the field of atomic energy use, international and Russian radioactive waste management practice, JSC VNIINM has developed a UIMMG Measurement of Radiation Waste Characteristics. The UIMMG provides recommendations on the selection of sampling procedures accounting for waste of various morphologies and measuring equipment, preparation of counting samples, measurements and processing of measurement results, as well as operational safety requirements.

Standard methods have been developed to measure the specific activity of radionuclides in packages and in samples taking into account the sampling procedures for SRW present in the form of construction waste, plastic compounds, equipment elements, PPE, soils:

- for gamma-emitting radionuclides in SRW samples and SRW held in packages and standard containers with a volume of up to 3.1 m^3;
- isotopes of uranium, plutonium, strontium by the method of LS-spectrometry in SRW samples.

Algorithms have been developed to control the specific activity measurement accuracy for radionuclides contained in the waste using certified items.

To monitor the content of hard-to-detect radionuclides in radioactive waste, JSC VNIINM is developing a standard methodology suggesting the application of the “radionuclide vector” method.

Sampling guidelines for various spatially inhomogeneous objects and standard methods allowing direct in-situ measurements of radionuclide specific activities or measurements during waste segregation are planned to be developed by JSC VNIINM to further improve the metrological support for the measurement of radiation waste characteristics.

The developed set of documents has laid a basis for future plans drawn regarding relevant activities associated with the measurement of radiation waste characteristics and is aimed at unifying RW management approaches, inter alia, covering the waste generated from nuclear decommissioning.

According to NP-093-14 [17], to demonstrate waste compliance with waste acceptance criteria for disposal, physical and chemical properties of the waste, as well as the content of nuclear hazardous fissile radionuclides shall be monitored in addition to its radiation characteristics. These parameters can be identified based on data concerning RW processing flowcharts. However, it’s considered impossible to trace the origin for some part of waste generated from nuclear decommissioning. Only analytical methods can help to identify physical and chemical characteristics of such waste and the content of fissile nuclear hazardous radionuclides in such wastes. JSC VNIINM plans to develop standard methods allowing to monitor the parameters of waste held in large containers and to assess the compliance of conditioned RW packages with acceptance criteria for disposal.

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