

APPROACHES TO THE CHARACTERIZATION OF GRAPHITE UGR BLOCKS BASED ON BETA-EMITTING ^{14}C AND ^{36}Cl RADIONUCLIDES. TESTING AT RBMK-1000-TYPE REACTORS

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This paper presents some methodological approaches to the characterization of graphite blocks based on difficult-to-measure ^{14}C and ^{36}Cl radionuclides during graphite stack dismantlement at uranium-graphite reactors (UGR) developed by JSC PDC UGR. It presents the testing results of the developed methods applied during RBMK-1000 graphite stack block characterization at the Leningrad NPP.

Keywords: irradiated graphite, difficult-to-measure radionuclides, radioactive waste, radiation characteristics, non-destructive method, radionuclide vector.

Introduction

Long-lived β -emitters ^{14}C ($T_{1/2} = 5,730$ years) and ^{36}Cl ($T_{1/2} = 301,000$ years) are seen as the key radionuclides mainly contributing to the potential hazard of the irradiated graphite waste generated from the dismantlement of shut-down uranium-graphite reactor (UGR) stacks, which is seen as a most important features of this type of RW distinguishing it from other RW types [1].

The ^{14}C isotope accounts for ~95 % of the total RW activity and is responsible for the class assigned to the graphite waste. Specific activity of ^{36}Cl in the irradiated graphite appears to be

lower than the one of ^{14}C (on average by 3–4 orders of magnitude) [2, 3]. In addition, for ^{36}Cl , the parameters describing the intensity of radionuclide leaching from graphite RW are by the same 3–4 orders of magnitude higher than those being considered typical for ^{14}C [2]. Thus, under the long-term predictive assessments focused on the safe management of this RW type (including its disposal in RWDF), both radionuclides are deemed to be comparably important considering their potential impact on the environment and the population [4].

Relevance and issues associated with the characterization of graphite RW by ^{14}C and ^{36}Cl

When it comes to the demonstration of graphite RW management safety, graphite RW characterization by β -emitting ^{14}C and ^{36}Cl is considered as a crucial aspect no matter which design option is applied for the decommissioning of uranium-graphite reactors. If the entombment option is considered than the amount of these isotopes accumulated in the graphite stack of the reactor appears to be essential. This parameter is seen as a crucial one when it comes to the predictive calculations of radionuclide releases from a disposal facility being under development. If the “immediate dismantlement” option is chosen, the class of graphite RW is specified based on the characterization process along with an appropriate disposal method. At the same time, data on graphite RW parameters as regards ^{14}C and ^{36}Cl and their leaching rate are essential to demonstrate the safety of the selected disposal option and to specify the acceptance criteria for a particular RWDF [5].

Complete dismantlement of a reactor unit (including the graphite stack) is seen as an essential feature of reactor decommissioning assuming the “immediate dismantlement” option. Therefore, packaging of the removed URG graphite blocks accounts for a stage of graphite RW management process.

In this case, certification of the filled packages (containers) will be seen as a required procedure. During such certification, isotopic composition, average specific and total activity of radionuclides contained in the RW packages should be specified for each RW package. In case of graphite RW, during the graphite UGR stack dismantlement such RW package certification according to ^{14}C and ^{36}Cl isotopes is considered as a serious methodological problem.

^{14}C ($E_{\beta\text{max}} = 156 \text{ keV}$, $E_{\beta\text{av}} = 49 \text{ keV}$) and ^{36}Cl ($E_{\beta\text{max}} = 710 \text{ keV}$, $E_{\beta\text{av}} = 251 \text{ keV}$) isotopes are pure β -emitters belonging to the category of difficult-to-measure radionuclides. Methods providing the direct detection of the spectra emitted by these isotopes (without sampling and sample preparation) cannot be applied to determine the activity characteristics of packages with irradiated graphite (which is typical for the vast majority of pure β -emitters mixed with other radionuclides). Therefore, assuming all currently applied methods, to specify the inventory of ^{14}C and ^{36}Cl radionuclides present in the irradiated graphite, a rather long flowchart should be implemented, including the following steps:

- sampling;
- sample manufacturing;

- preliminary treatment of samples;
- radiochemical separation of the “target radionuclide”;
- production of a load;
- β -spectrometric analysis.

These methods have proven to be extremely ineffective, both technically and economically, when applied for the certification of packages with dismantled UGR graphite blocks due to the large number of such blocks (a graphite stack of one RBMK-1000 reactor involves over 35,000 blocks).

Assessing the opportunities for applying the Radionuclide Vector method in the characterization of graphite RW by ^{14}C and ^{36}Cl

The Radionuclide Vector (or isotopic correlation method, scaling factor method) is seen as a method proposed to certify the RW packages under the above conditions [6–8]. In this case, the inventory of difficult-to-measure radionuclide is measured according to the radiation spectrum of another (easy-to-measure) radionuclide, the activity of which is associated (correlated) with the activity of the considered radionuclide.

This ratio can be of a physical origin (a decay chain). A case in point is the identification of the ^{238}U isotope content according to the gamma radiation spectrum of the $^{234\text{m}}\text{Pa}$ isotope. The second option suggests that the isotopes are not considered as links of the same decay chain, but the activity of the isotope of interest is unambiguously related to the activity of the measured isotope through the parameters of irradiation-driven accumulation. This case can be exemplified by the identification of $^{59,63}\text{Ni}$ isotope content in irradiated stainless steels based on the gamma radiation spectrum of the ^{60}Co isotope.

The option suggesting that the “radionuclide vector” is built along the ^{14}C and ^{36}Cl radioactive decay chain is deemed impossible, since these radionuclides decay along a simple chain with stable isotopes being generated (^{14}N and ^{36}Ar , respectively). After some 10 years of cooling following the final shutdown of the UGR, ^{60}Co isotope can be considered as the only reliably detectable gamma-emitting radionuclide of activation origin found in the graphite. However, in this case due to a number of reasons, the correlation between the specific activities of ^{60}Co relative to ^{14}C and ^{36}Cl appears to be unsatisfactory due to huge ranges of uncertainty in the correlation coefficients. This is mainly explained by the fact that in unirradiated graphite, cobalt impurities vary over a wide range [9]:

- for graphite GR-220 — $0.7 \cdot 10^{-7} - 3.8 \cdot 10^{-6} \text{ wt \%}$;

- for graphite GR-280 — $2.1 \cdot 10^{-7}$ — $6.2 \cdot 10^{-6}$ %wt%.
- Such variations in the cobalt content are typical both for different blocks within one stack and over the entire volume of an individual block. A similar situation is typical for the chlorine impurity (^{35}Cl) from which ^{36}Cl is formed [10]. In addition, the resulting uncertainty is somehow contributed by [11]:
- the nonlinearity of ^{60}Co and ^{36}Cl accumulation laws (in contrast to ^{14}C , the accumulation of which takes place according to a near-linear law);
 - the dependence between the ^{60}Co and ^{36}Cl accumulation curves, the thermal neutron fluxes and the irradiation temperature.

Figure 1 presents the ratio of ^{14}C and ^{60}Co specific activities in graphite blocks of RBMK-1000 reactors units, namely of unit No. 2 and No. 3 of the Leningrad NPP. To construct the diagrams, applied were the experimental data of the Kurchatov Institute [12] and JSC PDC UGR obtained in 2018 and 2020–2021. Values are given for a series involving 43 graphite blocks. The series include blocks located along the entire height of the graphite stack (including the end reflectors) from the “plateau” cells of the reactor cores in power units No. 2 and No. 3 of the Leningrad NPP.

Experimental results (Figure 1) showed that relative to the average value, the ratio of ^{14}C and ^{60}Co activities varies over a very wide range (from –99% to +700%). The range of values for the ratio of ^{14}C and ^{60}Co activities amounts to almost 3 orders of magnitude. The ranges characterizing individual RW classes according to the existing RW classification system also amount to some 3 orders of magnitude. Obviously, in case of graphite RW certification, such big uncertainty in the isotope ratio is considered unacceptable. Thus, when graphite RW packages are certified according to this ratio, not only there is a high uncertainty

associated with the measured ^{14}C activity, but also a high probability of overestimating or underestimating the RW class (considering that it is the ^{14}C content that governs RW assignment to a particular RW class). Thus, the “radionuclide vector” method cannot be applied to specify the activity characteristics of irradiated graphite packages in terms of ^{14}C and ^{36}Cl inventory.

Development of industry-specific instructions (methods) allowing graphite RW characterization by ^{14}C and ^{36}Cl

Specific features related to the accumulation and spatial distribution of ^{14}C and ^{36}Cl in graphite UGR stacks were studied by JSC PDC UGR in 2019–2020. These studies also involved certain experts from specialized organizations of the A. N. Frumkin Institute of Physical Chemistry of the Russian Academy of Sciences and the Metrological Department of JSC VNIINM named after A. A. Bochvar. Based on them, other principles and algorithms enabling the certification of packages with graphite RW during the dismantlement of graphite UGR stacks could be developed [13]. These formed a basis for the methods developed to characterize the irradiated graphite by ^{14}C and ^{36}Cl isotopes, i. e., industry-specific instructions OI 001.911-2020 and OI 001.912-2020 [14, 15].

They involve the following main parts:

- a flowchart describing the sampling process from a graphite block (Appendix A);
- a method providing the measurement of radionuclide (^{14}C and ^{36}Cl) specific activity in irradiated graphite samples;
- a flowchart providing the certification of packages with graphite blocks by isotope content (^{14}C or ^{36}Cl) during the graphite UGR stack dismantlement (Appendix B).

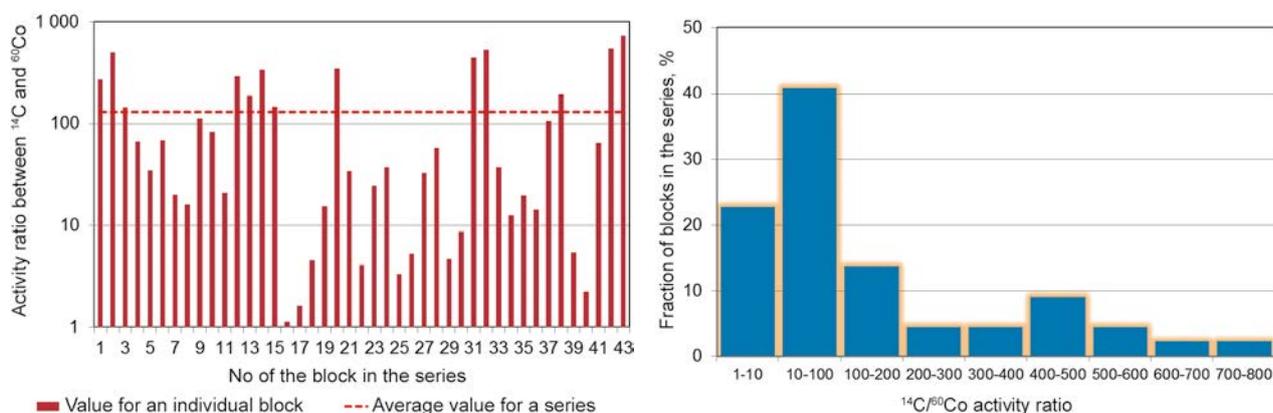


Figure 1. Diagrams representing ^{14}C and ^{60}Co specific activity ratio for graphite blocks of RBMK-1000 power units No. 2 and No. 3 of the Leningrad NPP: ^{14}C and ^{60}Co activity ratios in individual graphite blocks in the series (left); function showing the distribution of graphite blocks in the series by ^{14}C and ^{60}Co activity ratios (right)

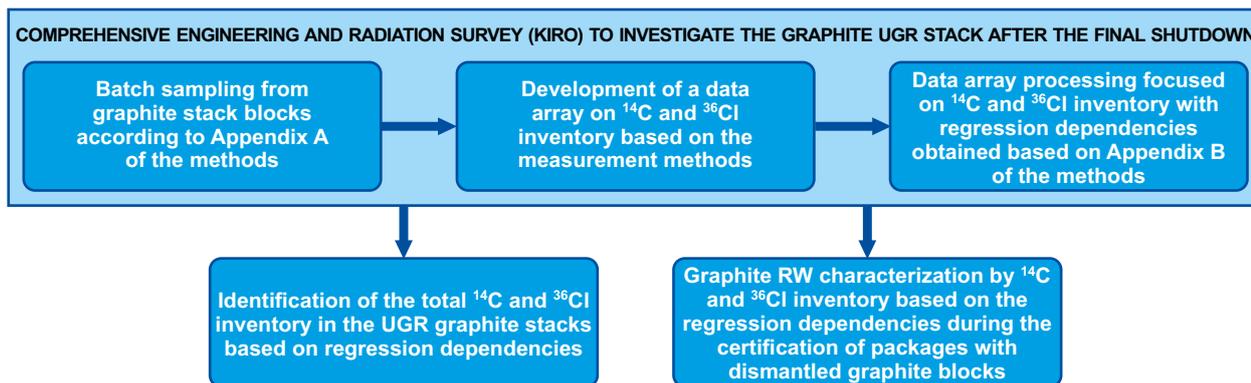


Figure 2. The flowchart explaining how the regression dependencies can be obtained and applied during the characterization of graphite RW by ^{14}C and ^{36}Cl isotopes in accordance with OI 001.911-2020 [14] and OI 001.912-2020 [15]

The proposed principle of RW characterization by ^{14}C and ^{36}Cl isotopes during the certification of packages with dismantled graphite blocks (Appendix B of OI 001.911-2020 and OI 001.912-2020 [14]) avoids mass measurements of samples under laboratory conditions. According to the principle laid down in Appendix B of OI 001.911-2020 and OI 001.912-2020 [15], ^{14}C inventory contained in the blocks is identified based on regression dependences between the elevation mark of the block in the cell and the power generation of the cell (for fuel cells) expressed in relative fractions of the maximum. For ^{36}Cl , the regression dependences are plotted based on the spatial distribution and the distribution function of graphite blocks according to the ^{36}Cl activity. Experimental data array on the ^{14}C and ^{36}Cl inventory present in the UGR stack is formed to plot relevant regression dependences under the comprehensive radiation survey. Figure 2 presents the flowchart explaining how the regression dependencies can be obtained and applied during the characterization of graphite RW by ^{14}C and ^{36}Cl isotopes in accordance with OI 001.911-2020 [14] and OI 001.912-2020 [15].

Practical evaluation of industry-specific instruction (method) application in the characterization of graphite stack blocks from RBMK-1000 type UGR

Physical features of ^{14}C accumulation suggest that the volume distribution of ^{14}C is with a high degree of correlation governed by the spatial distribution of thermal neutron fluxes both over the volume of graphite stacks and individual graphite blocks which is typical for Russian-built UGRs (including RBMK-1000 reactor units) with ^{14}C generation on gaseous nitrogen greatly contributing to its accumulation [17, 18]. Under the research performed, not only the cores sampled remotely from the

UGR graphite stacks were used in the experimental practical evaluation of the proposed methods: important statistics on the volumetric ^{14}C distribution was obtained regarding the graphite blocks removed from the graphite stack of PUGR ADE-5. Figure 3 shows the appearance of an extracted block, whereas Figure 4 presents the axial distribution of ^{14}C specific activity in this block.

Based on the studied spatial distribution, this block removed from the graphite stack of PUGR ADE 5 was certified by the Metrological Department of JSC VNIINM named after A. A. Bochvar. It was certified according to ^{14}C specific activity averaged over the block volume, as well as according to the volume distribution function for the ^{14}C specific activity. A Certificate for an Item Certified According

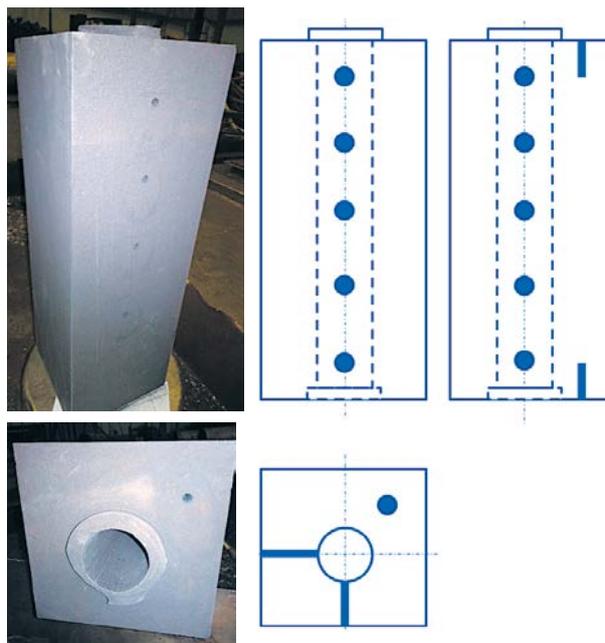


Figure 3. Graphite block removed from the graphite stack of PUGR ADE-5 after graphite sampling: side wall and end surface (left); graphite block sampling flowchart (right)

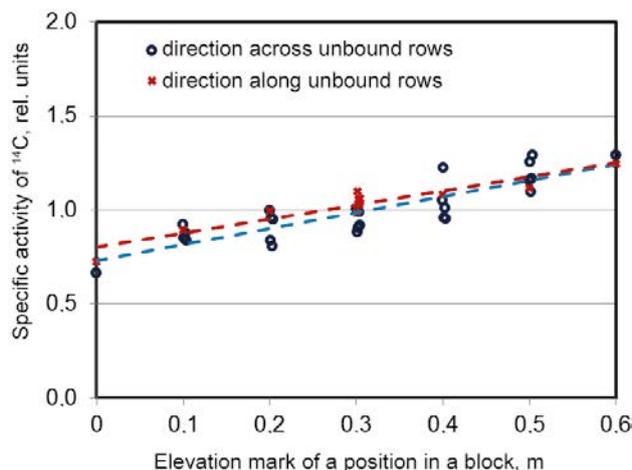


Figure 4. Relative distribution of ^{14}C specific activity along the height of a removed graphite block in two mutually perpendicular planes

to ^{14}C Activity (AO 95 505/531-27-2020) was issued. This block can be further used as a standard sample to check the correctness of already applied and newly developed measurement procedures (MP), to calibrate measurement tools, as well as to test possible options allowing express measurements of ^{14}C inventory. Previously, practical experience was gained during the development of Industry-specific Standard Samples. In 2018, interlaboratory comparative tests (ICT) allowed to certify graphite batches No. 1, No. 2 and No. 3 as Industry-specific Standard Samples based on the ^{14}C content [19]. Certificates for a Series of Items Certified Based for the Specific Activity of Carbon-14 Isotope (AO 95 505/532-3-2018; AO 95 505/532-3-2018; AO 95 505/532-3-2018) were issued following the certification. The certified items were included into the industry-wide register of certified items.

The flowchart for the certification of packages with graphite blocks according to ^{14}C isotope during the dismantlement of UGR graphite stacks using the OI 001.912-2020 method is based on the spatial distribution of ^{14}C (A_c) specific activity found in the graphite blocks over the volume of the graphite stack. This distribution results from a thermal neutron flux during UGR operation and the energy generation from the cells of the graphite stack.

Regression dependences allowing to identify ^{14}C specific activity in graphite blocks over the volume of the graphite stack are derived based on analytically processed experimental data from remote sampling (in accordance with Appendix A of the OI 001.912-2020) inside the UGR graphite stack volume and subsequent measurements of ^{14}C specific activity in the samples taken. Experimental studies focused on the spatial distribution of ^{14}C specific activity in the graphite blocks over the volume of the graphite stack are implemented

under comprehensive engineering and radiation surveys of the UGR graphite stacks after the final shutdown and fuel unloading. Appendix B of the OI 001.912-2020 presents a flowchart providing statistically representative arrays of samples taken from the graphite blocks over the volume of the graphite stack for the regression dependences.

Empirical regression dependences between ^{14}C specific activity in graphite blocks and the elevation mark of the graphite stack and the power generation of the cell are derived based on the processed data sets. Appendix B of the OI 001.912-2020 elaborates on the flowchart allowing to obtain such dependences for cells with a standard fuel load, control and safety system cells, as well as side reflector cells.

According to the algorithm laid down in Appendix B of the OI 001.912-2020, regression dependences presenting the ^{14}C inventory were obtained for the cells of graphite stacks in the reactor core areas of LNPP-2 and LNPP-3 accounting for their characteristic range of power generation. Figure 5 presents the regression dependences obtained by JSC PDC UGR based on an experimental data array from relevant studies on the accumulation of ^{14}C and ^{36}Cl in RBMK-1000 graphite stacks of LNPP-2 and LNPP-3 in accordance with OI 001.912-2020.

Figure 5 shows that the maximum deviations from the regression dependences obtained according to OI 001.912-2020 as regards the main share of the experimental points (more than 95%) do not exceed 25%. This fact testifies the correctness of the algorithms and recommendations of Appendix B OI 001.912-2020 suggesting the application of these regression dependences during the characterization of dismantled graphite blocks and certification of packages containing them as regards the ^{14}C inventory during the dismantlement of RBMK-1000 reactor graphite stacks.

The regression dependences for the ^{14}C inventory contained in graphite can differ significantly depending on the reactor type in question (RBMK, PUGR, AMB, EGP, etc.). Thus, for each specific type of reactor provided with identical grades of graphite and having similar operating parameters the regression dependences should be specified individually.

Appendix B of the OI 001.911-2020 methodology elaborates on the flowchart applied to certify packages with graphite blocks by the specific activity of ^{36}Cl isotope during the dismantlement of UGR graphite stacks.

^{36}Cl (A_{cl}) specific activity and in particular its spatial distribution over the stack volume heavily depends on the variation in the ^{35}Cl impurity content (impurity atoms in unirradiated graphite, from

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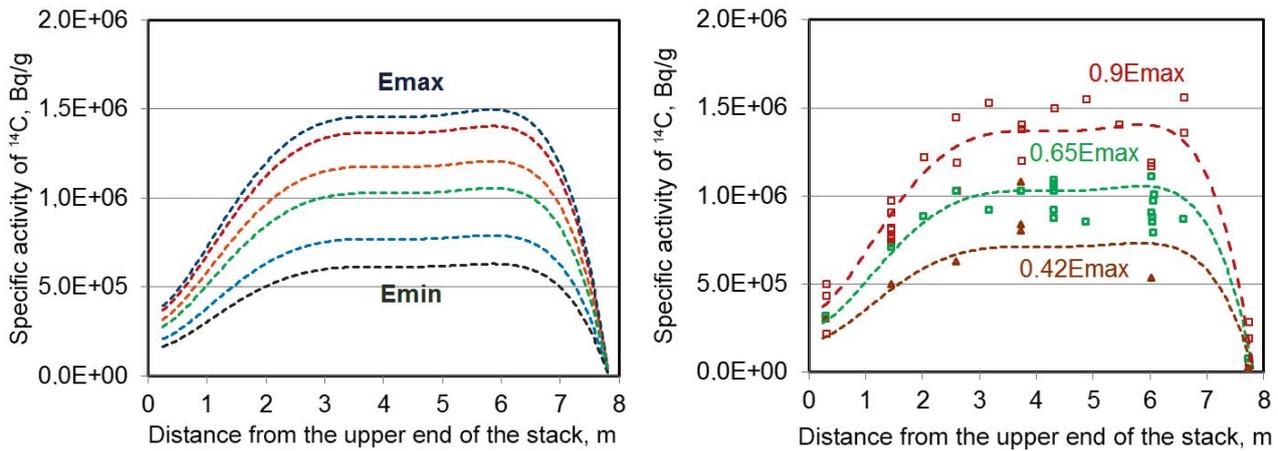


Figure 5. Regression dependences for the ^{14}C inventory obtained for the cells of graphite stacks in the reactor core areas of LNP-2 and LNP-3 accounting for their characteristic range of power generation: derived according to OI 001.912-2020 (left); with overlapping experimental points (right)

which ^{36}Cl is formed) distributed over the volume of individual graphite blocks, as well as from block to block. The research has demonstrated that general regularities and correlations are not typical for the ^{36}Cl distribution along the height of individual cells inside the UGR graphite stacks (Figure 6).

^{36}Cl isotope is distributed randomly over the graphite stack volume, but nevertheless its distribution stays within a certain range (Figure 6). Moreover, for the entire experimental data array on the height of the graphite stack, a tendency can be observed assuming its correlation with the altitude distribution of the neutron flux. However, the width of this range far exceeds the one being considered typical for the spatial distribution of thermal neutron fluxes in the stacks.

This phenomenon and its specific aspects are governed by several factors leading to a significant variation in its content [10, 18]. The functions

representing ^{36}Cl distribution over the entire array of graphite samples taken from the UGR stack (both PUGR and RBMK-1000) are similar in their main values and parameters and can be described by the Poisson distribution (Figure 7).

The width of the range showing the ^{36}Cl inventory amounts to over two orders of magnitude. Most of the blocks (~70%) are associated with a range having a width of an order of magnitude or 100–1,000 Bq/g. To characterize the blocks during package certification according to the OI 001.911-2020 method, the ^{36}Cl content is specified using the regression dependence of an axial distribution (Figure 6), as well as a function showing the distribution of the isotope content and relevant uncertainty indices identified for it (Figure 7). The resulting uncertainty in the identified ^{36}Cl specific activity is considered acceptable, since the ^{36}Cl specific activity found in graphite blocks does not affect the

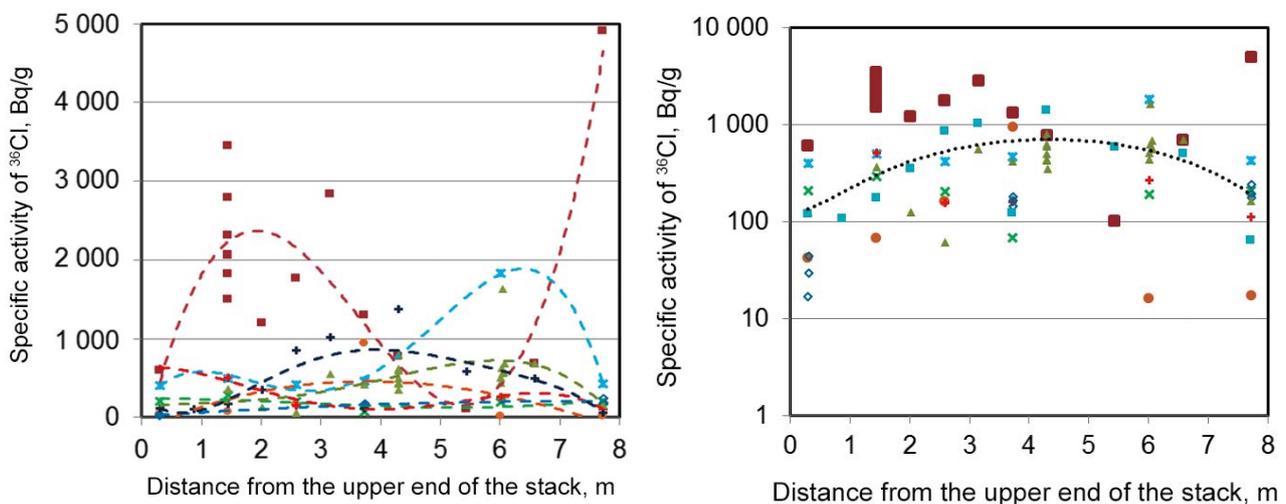


Figure 6. Distribution of ^{36}Cl content along the height of individual cells in the graphite stacks of RBMK-1000 reactors at LNP-2 and LNP-3 (linear scale (left) and logarithmic scale (right))

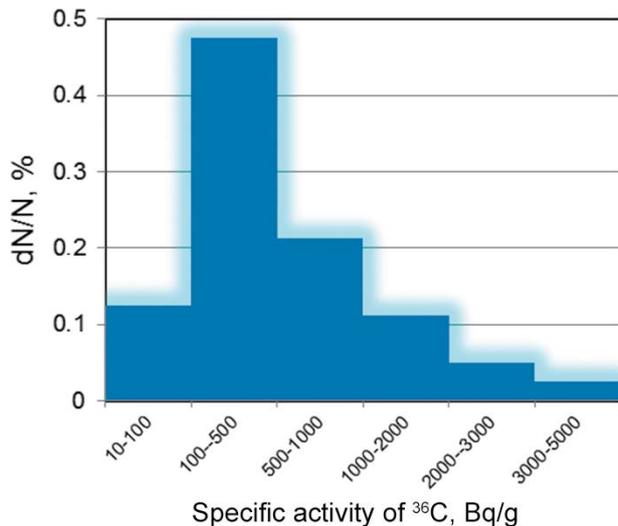


Figure 7. Distribution function for the ^{36}Cl content in RBMK-1000 graphite from LNPP-2 and LNPP-3

RW class. The class is established based on the ^{14}C isotope, the specific activity of which is on average 3–4 orders of magnitude higher than the one of ^{36}Cl . ^{36}Cl content in graphite typically does not exceed 5,000 Bq/g. Thus, even with the accepted measurement uncertainty of up to 90%, the average specific activity levels identified for the packaging will be definitely lower than the upper limit set for Class 3 RW (specific activity of 10^4 Bq/g). The actual uncertainty in the average specific activity of ^{36}Cl found in graphite will depend on the size of the block samples, i. e., on their number in the package. Considering some ~ 50 pcs. of blocks, the uncertainty in the average ^{36}Cl specific activity considered for a waste package will be less than 50%.

According to Appendix B of OI 001.911-2020, the spatial distribution of ^{36}Cl specific activity contained in graphite blocks over the volume of the graphite stack and the distribution function of the blocks by the ^{36}Cl inventory (Figures 6, 7) can be identified by remote sampling (in accordance with Appendix A of OI 001.911-2020) in the volume of the UGR graphite stack and subsequent measurements of ^{36}Cl specific activity during the KIRO (using the same array of samples as in case of ^{14}C).

During package certification, the specific activity of ^{14}C and ^{36}Cl found in the graphite blocks removed during the dismantlement of the UGR graphite stack from a certain cell and located at a certain elevation is determined in accordance with empirical expressions of regression dependences.

When the removed blocks are placed into a package (container), the total activity of the container according to ^{14}C or ^{36}Cl can be identified by the following expression:

$$A_{\text{C,Cl}}^{\Sigma} = \sum_{i=1}^L A_{\text{C,Cl}i} \quad (1)$$

where L is the number of removed blocks in a package (container); $A_{\text{C,Cl}}^{\Sigma}$ is the total activity of ^{14}C or ^{36}Cl in the removed blocks emplaced into a package (container), Bq; $A_{\text{C,Cl}i}$ stands for ^{14}C or ^{36}Cl activity of the i -th block in a package (container), Bq.

The average specific activity of the package (container) with waste is determined based on $A_{\text{C,Cl}}^{\Sigma}$ and the total mass of the blocks in a container.

Confirmatory measurements of samples taken from the blocks removed according to the present Methodology are recommended to be performed during the certification of packages (containers) in accordance with the procedure indicated in Appendices B of OI 001.911-2020 and OI 001.912-2020. Measurements are recommended to be performed for one or two blocks emplaced into a container. The blocks intended for control measurements are recommended to be selected among the blocks characterized by the maximum specific activity levels as regards ^{14}C and ^{36}Cl isotopes found in a package subject to certification.

Conclusion

Based the studies focused on the specific aspects of ^{14}C and ^{36}Cl accumulation and their spatial distribution in UGR graphite stacks, Industry-specific instructions (methods) OI 001.911-2020 and OI 001.912-2020 were developed providing the characterization of irradiated graphite by the specified isotopes. These instructions present:

- A technique allowing to measure the specific activity of radionuclides (^{14}C or ^{36}Cl) found in the irradiated graphite samples;
- A procedure for sample taking from graphite blocks;
- A procedure providing the certification of packages with graphite blocks by isotope (^{14}C or ^{36}Cl) during the dismantlement of UGR graphite stacks.

Thanks to the principle proposed for graphite RW characterization by ^{14}C and ^{36}Cl isotopes during the certification of packages with dismantled graphite blocks (Appendices B OI 001.911-2020 and OI 001.912-2020) to be implemented during the decommissioning of graphite stacks, mass measurements of samples under laboratory conditions can be avoided. The practical applicability of the developed techniques has been experimentally proven for graphite stacks of PUGR I-1, ADE-5, RBMK-1000 reactor units of LNPP-2 and LNPP-3. Practical testing has resulted in acceptable accuracy indicators as regards the measurements of ^{14}C and ^{36}Cl specific activity found in the graphite blocks of UGR stacks performed according to the proposed algorithms. These were recognized as metrologically reasonable. Industry-specific instructions (methods)

OI 001.911-2020 and OI 001.912-2020 were certified by the Metrological Department of JSC VNIINM named after A. A. Bochvar.

Regression dependences representing the content of ^{14}C and ^{36}Cl isotopes in graphite RW derived base on relevant provisions of OI 001.911-2020 and OI 001.912-2020 are considered applicable not only in the cases associated with the certification of packages with dismantled UGR graphite blocks. These can be used to specify the total ^{14}C and ^{36}Cl inventory found both in the dismantled graphite UGR RW during its disposal in RWDF of various types and in case of facility decommissioning according to the entombment option. These dependencies provide more accurate estimates (the results obtained are never underestimated) regarding the content of these radionuclides and avoid unreasonably (overly conservative) overestimated forecasts as regards the potential hazard of graphite RW when different disposal options are considered and their safety is evaluated.

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Processing, Conditioning and Transportation of Radioactive Waste

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