A Methods for Non-destructive Radiological Characterisation by In Situ Spectrometry Measurements

Miodrag Milošević, Milan Petrović

Abstract - **A methodology for non-destructive radiological characterisation by in situ measurement were prepared to support the decommissioning planning effort for the RA shut down research reactor, and to manage existing radioactive waste. The geometry models of semiconductor HPGe and scintillation NaI detectors were developed for the MCNP-5 Monte Carlo code. Radiography was applied to ascertain the physical dimensions of these detectors. Scanning with a collimated source was utilised to estimate the ðdead layerð of HPGe detectors. Finally, the MCNP-5 code was used to determine the total inactive ðdead layerð of HPGe detectors, which are needed in order to obtain the minimum discrepancy between estimated and experimental efficiency. This paper describes all these steps, and summarises the strategy for the space activity measurements. Example application was given for radiological characterisation of contaminated HEPA filters.**

Index Terms - **HPGe and NaI detectors, efficiency simulations, MCNP-5 code, in situ measurement.**

I. INTRODUCTION

The current efforts, one connected to the planning of decommissioning strategy for the RA nuclear reactor, and others to the management of historical radioactive waste stored in hangars of Public Company Nuclear Facilities of Serbia, stimulate the development of methods and procedures that could provide radiological characterisations for these challenges. The main objective of this work was to harmonise developed methods and procedures with the recommendations of the International Atomic Energy Agency (IAEA), presented in Ref. [1] for decommissioning strategy of shut down nuclear reactors, and in Ref. [2] for strategy of radioactive waste characterisation. The method and results placed on radiological characterisation of neutron activated materials in the RA nuclear reactor (representing the major source when estimating total inventory) were presented in Ref. [3]. This paper describes the methods and strategy which are developed for radiological characterisation of contaminated components, structures and materials in order to support the RA nuclear reactor decommissioning planning effort, to provide nuclear safeguards and also to enhance the quality and accuracy of existing waste characterisation.

The starting point in this development was to provide the as much as possible detailed non-destructive analysis for radionuclides in all cases where a measurable amount of gamma photons or X rays are able to penetrate the package to be measured. Since this can be achieved by using gamma spectrometry, the main purpose of this work was to prepare the calibrations of *in situ* measurements, and to determine the measurement uncertainty according to ISO/BIMP ″Guide to the Expression of Uncertainty in Measurement″ (usually referred to as the GUM). This was accomplished by development of geometry models for semiconductor HPGe and scintillation NaI detectors using the MCNP-5 Monte Carlo code [4] that could accurately reproduce experimentally determined efficiencies.

This paper gives review of developed geometry models of the HPGe and NaI detectors for the MCNP-5 code, summarises the validation of used Monte Carlo simulations for calibrations of in situ measurements, and presents results of activity measurement for radionuclides in contaminated HEPA filters.

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II. METHODOLOGY

Presented in situ gamma spectroscopy methodology is based on application of two semiconductor coaxial high purity germanium (HPGe) detectors and two scintillation NaI detectors. The HPGe detectors were chosen for *in situ* measurements, because of their superior energy resolution, while the NaI detectors with lower resolution were selected for situations demanding robust performance, due to their a proven long term reliability and stability.

One of the used HPGe detectors, the PGT-IGC7 detector, was manufactured by Princeton Gamma-Tech (Princeton, New Jersey). This detector was connected to GBS multichannel analyser MCA-166, operated with the WinSPEC acquisition software (GBS) [5]. The second HPGe detector is the GEM20P4 detector, manufactured by EG&G Ortec (Oak Ridge, Tennessee), connected to the Ortec DigiDART multichannel analyser that uses the GammaVision 32 software (Ortec) [6]. These detectors were specified as having a 15% and 20% efficiency relative to a 3x3″ NaI(Tl) detector at 1.33MeV, respectively. Both HPGe detectors were prepared as mobile measurement systems, and were kept at room temperature when not used for measurements.

The selected portable scintillation NaI detectors are Model 802 and Model NAIS, manufactured by Canberra (Meriden, Conectitat). Model 802 uses GBS multichannel analyser MCA-166 and the WinSPEC acquisition software (GBS), while the model NAIS operates with Osprey™ (Universal Digital MCA Tube Base for Scintillation Spectrometry) supplied with the Genie-2000 gamma analysis software [7].

All selected mobile detectors can be used as bare or with 30° and 60° collimators (each with 50 mm thick lead shield), reducing interfering 1000 keV radiation by factor of 60. In addition, the GEM20P4 detector includes 30°, 90° and 180° collimators with 25 mm thick lead shield, providing the reduction factor of 7.5 (for 1000 keV radiation).

The i*n situ* gamma-ray spectrometry can be a powerful method that can identify and quantify radionuclides directly at the measurement site; however it is a complex technique especially when applying for historical radioactive waste with non-uniform distribution of radionuclides. In order to provide required efficiency for more complex geometries, difficult to determine experimentally (requires that the standard sources of radioactive mixture has the same counting geometry as measured one) or describe mathematically, the Monte Carlo simulation with the MCNP-5 code was employed. This approach however requires an increase in computational power. Consequently, there is a need to improve the numerical calibrations of in situ measurements, and to determine the measurement uncertainty according to ISO/BIMP ″Guide to the Expression of Uncertainty in Measurement″. The approach that allows accomplishing this purpose includes next steps:

- modelling of all selected spectrometers to match Monte Carlo calculated efficiencies with experimental ones using the MCNP-5 code,
- validation of Monte Carlo simulations to experimentally determined efficiencies, and
- determination of the combined uncertainty of *in situ* measurements.

A. *Geometry models*

The geometry models for Monte Carlo simulation were initially based entirely on detector dimensions provided by the manufacturers. It is also important to note that the Monte Carlo simulations of gamma ray spectrometers overestimate the detection efficiency compared to empirical measurements [8-9]. Such discrepancies were also found for the two HPGe and two NaI detectors used in the work presented in this paper.

For HPGe detectors, the uncertainty analysis has been focused on dead layer thickness (at N+ contact) and on germanium crystal dimensions. In order to verify the physical dimensions of the crystal, as well as accurately determine the actual position of the germanium crystal within the aluminium casing, the detector was radiographed. The actual crystal-to-end cap distances were taken from X-ray images. Attempt to measure the thickness of the dead layer was made according to Ref. [9]. A source collimator that could be positioned in two different ways was designed, so that the photon beam could hit the detector surface at two different angles of incidence relative the crystal surface, 45° and 90°. Full-energy peak count rates for both angles of incidence were then recorded for 59.54 keV photons from the ^{241}Am source. The thickness of the absorbing layers was then calculated from the relative change in the count rate between the two angles. Results confirm the important role of the dead layer thickness in the low energy range of the efficiency curve. In the high energy range (from 300 keV to 1800 keV) the main contribution to the absolute uncertainty stem from variations in the active volume. Finally, adjustment of the HPGe detectors geometry models were achieved by optimising the dead layer thickness in Monte Carlo simulations, adjusting that the calculated efficiencies matches the experimentally obtained values.

The sources used for measurements were a point type, cylindrical and Marinelli beakers calibration gamma-ray standards, covering the energy range between 59.54 keV and 1408.0 keV. The uncertainty of the experimental efficiency was estimated using the ISO/BIMP standard from certified activities and counting results. The used gamma-ray standards contain the following radionuclides: ^{241}Am, ^{137}Cs and ^{60}Co with specified uncertainty less than 1.5% and ^{133}Ba and ^{152}Eu, with specified uncertainty less than 3.5%.

After the all steps (the radiography, the scanning with the ^{241}Am source, and the optimisation of dead layer thickness), it was found that adjusted geometry model of the IGC7 detector, with diameter of 39.0 mm and height of 27.0 mm, must have a dead layer increased to 1 mm. The horizontal cross section of the IGC7 detector geometry model, used in the MCNP-5 code, is presented in figure 1.

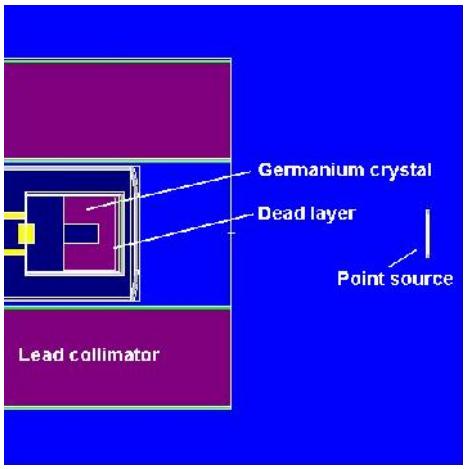

Fig. 1. Horizontal cross section of the IGC7 detector geometry model with lead collimator and point source

On the same way, for adjusted geometry model of the HPGe detector GEM50P4 (Ortec) the next values: diameter of 53.6 mm, height of 46.2 mm and dead layer of 1.3 mm were selected. The radius of the bulletizing of the crystal front was initially estimated to be 5 mm. Horizontal cross section of the GEM20P4 detector geometry model, used in the MCNP-5 code, is given in figure 2.

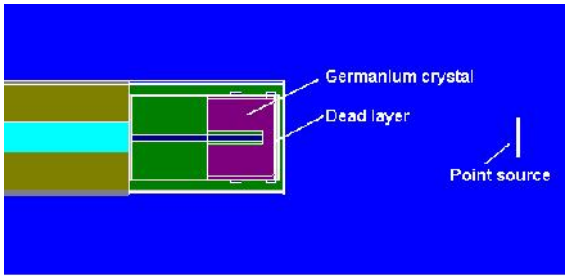

Fig. 2. Horizontal cross section of bare GEM20P4 detector geometry model with point source

For both NaI detectors (Canberra model s802 and NAIS) the parameters of geometry models were chosen based on the results of radiography measurements. These measurements

confirmed that NaI crystals in both detectors have diameter of 5.08 mm and height of 50.8 mm. The agreement between Monte Carlo simulation and measurement of detector efficiency was achieved by adjusting the thickness of reflector (initially chosen as MgO) placed around NaI crystal. It was estimated that thickness of MgO reflector for both NaI detectors has a value of 1.85 mm.

In Monte Carlo simulation of these detectors with the MCNP-5 code, photon and electron transport in the energy range from 1 keV to 2000 keV (phys:p and phys:e) were included. The F8 (Pulse Height Distribution) tally was used for photons and electrons, and the GEB (Gaussian Energy Broadening) card option was applied to provide a spectrum that can be compared with the experimental one in terms of resolution (FWHM and FWTM). The number of histories has been varied in order to obtain a relative error less than 0.1% at every peak net area. Finally, in order to avoid errors due to the net area calculation, the measured pulse height distributions (PHD) from all chosen spectrometers and Monte Carlo simulated PHDs for these detectors were analysed using the ANGES software [10].

B. *Validation*

Validation of presented geometry models for the IGC7 (PGT), GEM20P4 (Ortec), NaI model 802 (Canberra) and NaI model NAIS (Canberra) detectors by comparisons with experimental data are essential for the planned in situ measurements. In order to facilitate relevant comparisons between simulations and measurements, in all results an estimate of the measurement uncertainties (as the most significant sources of uncertainty) were included.

Using the adjusted geometry models, with dead layer thickness (in HPGe detectors) and MgO reflector thickness (in NaI detectors) based on optimisation process, the discrepancy between calculated and empirical efficiencies is reduced to below 5% for all standard sources, angles and energies tested. These findings about calculated and empirical efficiencies for the IGC7, GEM20P4 and NaI detectors (model 802 and model NAIS) are illustrated in tables I, II, III, and IV, respectively.

In parallel to this process, the testing of the ISOCS (Canberra) efficiency calibration software [11], obtained along with the NaI detector NAIS, was carried out. By combining the detector characterisation produced with the MCNP^{TM} code, mathematical geometry templates, and few physical sample parameters; this software provides ability to produce accurate efficiency calibrations of almost any sample type and size. The used ISOCS software contains the characterisation data not only for the NaI detector NAIS, but also for few HPGe detectors manufactured by Canberra. It was found that this software and characterisation data produced for Canberra coaxial HPGe detector with diameter of 50 mm gives for the GEM20P4 (Ortec) detector typically 10-20% lower efficiency than those found experimentally. Thedifference are the same as for the NAIS detector, for which this software was obtained. Such results enabled the possibility for another approach to solve efficiency calibration by simply accepting the difference between measurements and simulations, and correcting it simply by multiplying simulation results with a correction factor (sometimes referred to as an efficiency transfer function) [8,9]. This function should take the form of a geometry correction but includes a correction for the intrinsic detector efficiency, given the assumption that this correction depends only on photon energy, but not on the angle of incidence. The efficiency for a particular geometry, ε, is then given by

$$\varepsilon = \varepsilon_{ref} \frac{\varepsilon^{I}}{\varepsilon^{I}_{ref}} \quad (1)$$

where ε_{ref} is the experimental efficiency for a reference case, and ε^{I} and ε^{I}_{ref} are calculated (via ISOCS software) efficiencies for the geometry in question and the reference case, respectively.

TABLE I
DISCREPANCY BETWEEN SIMULATED AND MEASURED EFFICIENCIES FOR THE IGC7 DETECTOR AND POINT SOURCES AT 15 CM

Energy [keV]	ε^{mea}	$100(\varepsilon^{cal}/\varepsilon^{mea}-1)$
0.05954	$2.3497\cdot10^{-4}$	4.61
0.08100	$9.1733\cdot10^{-4}$	0.59
0.27640	$8.6121\cdot10^{-4}$	-1.21
0.30285	$7.5394\cdot10^{-4}$	0.46
0.35601	$6.1340\cdot10^{-4}$	0.13
0.38385	$5.5172\cdot10^{-4}$	1.14
0.66166	$2.9652\cdot10^{-4}$	-1.20
1.17323	$1.5908\cdot10^{-4}$	-0.72
1.33249	$1.3728\cdot10^{-4}$	0.87

TABLE II
RATIO OF MEASURED AND CALCULATED EFFICIENCIES FOR THE GEM20P4 DETCTOR AND PONIT SOURCES AT 15 CM

Energy [keV]	$\varepsilon^{mea}/\varepsilon^{cal}$	$\varepsilon^{mea}/\varepsilon^{ISOCS}$
0.05954	1.002	0.825
0.081	1.053	1.060
0.12178	1.053	1.122
0.2447	1.019	1.124
0.2764	1.038	1.154
0.30285	1.031	1.140
0.34428	1.017	1.112
0.35601	1.027	1.138
0.38385	1.040	1.150
0.44397	0.986	1.070
0.66166	0.999	1.071
0.7789	0.988	1.061
0.86737	0.980	1.034
1.08587	0.982	1.052
1.11207	0.995	1.066
1.40801	0.980	1.051

TABLE III
DISCREPANCY BETWEEN SIMULATED AND MEASURED EFFICIENCIES FOR NAI DETECTOR MODEL 802 AND POINT SOURCES AT 18 CM

Energy [keV]	ε^{mea}	$100(\varepsilon^{cal}/\varepsilon^{mea}-1)$
0.05954	0.00376	2.9
0.08100	0.00412	-0.1
0.12178	0.00375	2.4
0.35601	0.00238	-0.8
0.66166	0.00124	-1.6
0.96408	$8.53\cdot10^{-4}$	0.1
1.40801	$4.91\cdot10^{-4}$	1.1

TABLE IV
RATIO OF MEASURED AND CALCULATED EFFICIENCIES FOR THE NAI DETECTOR MODEL NAIS AND POINT SOURCES AT 10 CM

Energy [keV]	$\varepsilon^{mea}/\varepsilon^{cal}$	$\varepsilon^{mea}/\varepsilon^{ISOCS}$
59.54	0.945	1.329
81	1.047	1.534
356.01	0.946	1.013
661.66	0.968	0.944
1173.23	1.050	1.043
1332.49	1.042	1.035

Finally, the combined uncertainty of intrinsic detector efficiency calibrations for *in situ* measurements was estimated according to the uncertainties of the efficiency measurement what lead to discrepancies between the simulated and measured efficiencies. The uncertainty in the intrinsic detector efficiency was found to be less than 5% for almost every package in almost any environment.

C. *Strategy*

The strategy of prepared methodology for in situ measurements is based on the collimated detection geometry of presented (Monte Carlo calibrated) spectrometers and vertical and angular scanning of radioactive package. The activities of radionuclides of interest from the recorded spectrum or a set of recorded spectra of gamma emitters contained in the measured package, equal to the number of inserted segments (*n*) in the package, are calculated by solving the next system of equations

$$\sum_{j=1}^{n} r_{ij}\cdot A_j = c_i,\ i=1,2,...n,$$
$$A_j \geq 0,\ j=1,2,...n, \qquad (2)$$

where the element r_{ij} represents the simulated response of the detector at position *i* to the radiation coming from the uniformly distributed source of the activity of 1 Bq in segment *j* of the package; c_i represents the peak area of radionuclide (in cps); it has been measured in the *i*-th position of the detector; A_j represents the unknown activities of the measured radionuclide in segment *j* of the package. Numerically, the system of equations (2) with the restriction of non-negativity ($Aj > 0$) on the solution, is solved using the Least Squares with Linear Inequality Constrains Method (LSI method), described in [12]. Combined measurement uncertainty for radionuclide activity A_j is determined on the basis of solutions of system of equation (2) for parameters r_{ij} and c_i simulated by using the Box-Muller method, Ref. [13].

The main idea of planned in situ measurements is to provide optimal number of segments for vertical and/or angular scanning of examined package taking into account both - the accuracy of measurements and measuring time. This can be done on the basis of detailed measurements for some representative packages, using different number of vertical and angular segments. Starting from the estimation that total activity of package converges with increasing the number of segments, the parameter of optimal scanning for accepted uncertainty can be selected by using the difference between total activity for given number of segments and converged value. This idea is illustrated for the standard metal barrel (200 *l*) filled with radioactive concrete debris and soil, characterised with the collimated NaI detector Model 802, figure 4. According to the obtained results, given in table V, it is possible to select the minimum number of segment for given uncertainty due to spatial distribution of radionuclide in the barrel. In this particular case, for the overall uncertainty (connected to the non-uniform distribution of ^{137}Cs) less than 10% of results can be achieved with the 3 vertical segment by using stand for barrel rotation.

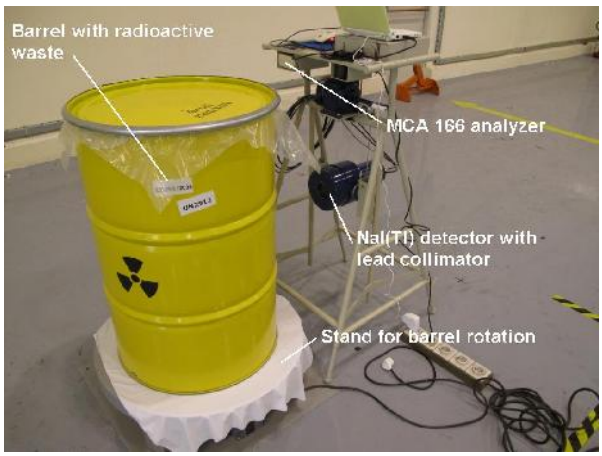

Fig. 4. Standard metal barrel with radioactive waste

TABLE V
ACTIVITY OF ^{137}CS AS FUVTION OF NUMBER OF SEGMENTS

Number of Vertical·Angular segments	^{137}Cs activity [Bq·g^{-1}]	$100(A_n/A_{32}-1)$ [%]
1·1	9.773±0.490	29.5
3·1	8.127±0.417	7.6
8·1	7.600±0.387	1.0
8·4	7.550±0.385	0.0

III. RESULTS

The proposed methodology is applied for three representative samples (designed as HP1, HP2 and HP3) for each of three set of contaminated HEPA filters, that should be characterised. According to the schematic view, presented in figure 5, one can see a non-uniform composition of the HEPA filters.

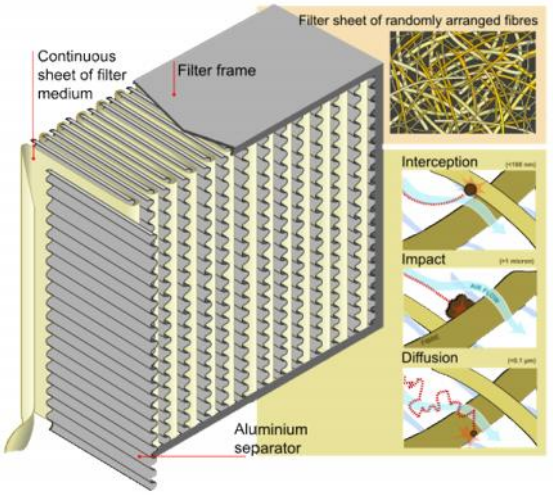

Fig. 5. Schematic view of measured HEPA filter

In order to determine an effective density and composition (volume weighting fraction of glass fiber paper and aluminium) of used HEPA filters, the transmission measurements for three different positions of the HEPA filter to the IGC7 detector (at three different angles, 0°, 22.5° and 45°) were carried out. For each position of examined HEPA filter to the IGC7 detector, the transmissions were measured for three selected gamma-ray peaks of ^{152}Eu (121.78 keV, 244.70 keV and 344.28 keV). Using the interpolation of prepared values of mass-attenuation coefficients for HEPA filter $\mu_a(E_i, v_{gfp}^{k-1}, v_{Al}^{k-1})$ as function of volume fractions of glass fiber paper (v_{gfp}^{k-1}) and aluminium (v_{Al}^{k-1}), the unknown parameters v_{gfp}^{k} and v_{Al}^{k} can be obtained by iterative approach based on solution of next three equations

$$e^{-\rho(v_{gfp}^{k}, v_{Al}^{k})\cdot\mu_a(E_i, v_{gfp}^{k-1}, v_{Al}^{k-1})\cdot d} = t(E_i),$$
$$\rho(v_{gfp}^{k}, v_{Al}^{k}) = \rho_{gfp} v_{gfp}^{k} + \rho_{Al} v_{Al}^{k} + \rho_{Air}(1 - v_{gfp}^{k} - v_{Al}^{k}), \quad (3)$$

where *k* denote current iteration, *d* is effective thickness of examined HEPA filter for selected angle, and $t(E_i)$ measured transmission for gamma-ray energy E_i. Parameters ρ_{gfp}, ρ_{Al} and ρ_{Air} are densities for glass fiber paper, aluminium and air, equal to 1.75, 2.7, and $1.25\cdot10^{-3}$ g·cm^{-3}, respectively. For numerical solution of equation (3), the LSI method was used. On the basis of results for three selected angles, the uncertainties for density and composition of each type of HEPA filters were estimated. Results clearly indicate that uncertainties for volume fraction of glass fiber paper and aluminium are much lower than uncertainty for density. The obtained results for density and composition (in term of weighting fractions) of HEPA filters HF1, HF2 and HF3 are given in table VI.

TABLE VI
DENSITY AND COMPOSITION OF HEPA FILTERS

HEPA filter	Description
HF1	Glass fiber paper (GFP) filter: 56.5 ·53.5· 14.3 cm^3, ρ=0.065375 g·cm^{-3} (±2.5%) w_{Al}= 0.20413, w_{gfp}= 0.77808, w_{air}= 0.01779
HF2	Glass fiber paper (GFP) filter: 47.2 ·46.7· 16.7 cm^3, ρ=0.061571 g·cm^{-3} (±2.5%) w_{Al}= 0.21918, w_{gfp}= 0.76188, w_{air}= 0.01894
HF3	Glass fiber paper (GFP) filter: 56.7 ·56.7· 26.5 cm^3, ρ=0.053711 g·cm^{-3} (±5.0%) w_{Al}= 0.24846, w_{gfp}= 0.72973, w_{air}= 0.02181

For checking of spatial distribution identified radionuclide ^{137}Cs, peak area of the ^{137}Cs were measured for each of examined HEPA filters (HF1, HF2 and HF3) with collimated IGC7 (PGT) detector. Measurements were carried out for 10 different position of detector (with 5 positions on each side of filter where air flowed), and for a minimum distance between the lead collimator and HEPA filters (0.5cm). Assays of measured spatial distribution of ^{137}Cs has shown that the maximum differences between 10 measured values for each of three HEPA filters were less than 2.5%. Based on these results, it was concluded that for ^{137}Cs measurements in these HEPA filters it is sufficient to use open geometry configuration. The open geometry configuration is setup in such a way that all parts of a package can contribute to the response of the detectors.

The obtained results for activity of ^{137}Cs in contaminated HEPA filters, are given in tables VII, VIII, IX, and X.

TABLE VII
RESULTS OBTAINED WITH COLLIMATED IGC7 (PGT) DETECTOR

HEPA filter type	Results Collimated IGC7 (PGT) detector and the MCNP-5 code geometry model
HF1	53837 ± 1723 Bq 19.1 ± 0.6 Bq·g^{-1} 24.5 ± 0.8 Bq·g^{-1} (paper)
HF2	13076 ± 420 Bq 5.8 ± 0.2 Bq·g^{-1} 7.6 ± 0.2 Bq·g^{-1} (paper)
HF3	14536 ± 514 Bq 3.2 ± 0.1 Bq·g^{-1} 4.4 ± 0.1 Bq·g^{-1} (paper)

TABLE VIII
RESULTS OBTAINED WITH BARE GEM20P4 (ORTEC) DETECTOR

HEPA filter type	Results Bare GEM20P4 detector and the MCNP-5 code geometry model
HF1	53564 ± 1964 Bq 19.0 ± 0.7 Bq·g^{-1} 24.4 ± 0.9 Bq·g^{-1} (paper)
HF2	13058 ± 488 Bq 5.8 ± 0.2 Bq·g^{-1} 7.6 ± 0.3 Bq·g^{-1} (paper)
HF3	14600 ± 495 Bq 3.1 ± 0.1 Bq·g^{-1} 4.2 ± 0.1 Bq·g^{-1} (paper)

TABLE IX
RESULTS OBTAINED WITH COLLIMATED NAI MODEL 802 (CANBERRA) DETECTOR

HEPA filter type	Results Collimated NaI detector model 802 and the MCNP-5 code geometry model
HF1	55981 ± 1777 Bq 19.8 ± 0.6 Bq·g^{-1} 25.5 ± 0.8 Bq·g^{-1} (paper)
HF2	13310± 409 Bq 5.9 ± 0.2 Bq·g^{-1} 7.7 ± 0.2 Bq·g^{-1} (paper)
HF3	14383 ± 509 Bq 3.1 ± 0.1 Bq·g^{-1} 4.3 ± 0.1 Bq·g^{-1} (paper)

TABLE X
RESULTS OBTAINED WITH BARE NAI MODEL NAIS (CANBERRA) DETECTOR AND ISOCS SOFTWARE

HEPA filter type	Results Bare NaI detector model NAIS and the ISOCS software
HF1	55675 ± 2832 Bq 19.8 ± 1.0 Bq·g^{-1} 25.5 ± 1.3 Bq·g^{-1} (paper)
HF2	12479 ± 644 Bq 5.5 ± 0.3 Bq·g^{-1} 7.2 ± 0.4 Bq·g^{-1} (paper)
HF3	14036 ± 731 Bq 3.1 ± 0.2 Bq·g^{-1} 4.2 ± 0.2 Bq·g^{-1} (paper)

These results show that difference between presented spectrometers in ^{137}Cs activity are less than 5%, i.e., less than total measurement uncertainty for each of them.

IV. CONCLUSION

On the basis of extensive validation of presented geometry models for two HPGe and two NaI detectors prepared for the MCNP-5 code, and selected strategy with vertical and angular scanning of radioactive packages, one can expect that the proposed methodology will be suitable and usable regarding the needs of the planned radiological characterisation.

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