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Inventorying the radionuclides in spent cartridge filters from the primary circuit of a nuclear research reactor by the dose-to-activity method

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ABSTRACT

The high activity of spent cartridge filters from the cooling water polishing system of a nuclear research reactor precludes, in routine work of a waste characterization program, the usual method of radiochemical analysis of filter samples. For this waste, the dose-to-activity method, using a gamma spectrometer and a dose rate meter, is an alternative for the determination of the activities of the gamma emitters with acceptable accuracy. The ratio of photon peak-areas from different radionuclides, corrected by the detector energy-dependent efficiency, allowed estimating the proportions between the activities of the different radionuclides present. Finally, the simulation of the counting geometry, using the point-kernel method to estimate the air dose rate resulting from an arbitrary concentration of those radionuclides and the comparison of the calculated with the measured air dose rate have yielded the needed estimates of the activity concentrations. The aim of this paper is to report the development of the method applied for the determination of the concentration of the gamma-emitters present in cartridge filters from the IEA-R1 Nuclear Research Reactor.

1. Introduction

Filter cartridges are used to retain particulate matter suspended in the water of the primary circuit of the IEA-R1 research reactor at IPEN, São Paulo, Brazil. These filters are part of the cooling water polishing system, which also includes beds of ion-exchange resin and activated charcoal. The radionuclides present in the reactor cooling system that are retained in the filters are the fission and activation products, and the transuranic elements suspended as solid particles in the cooling water. The filter cartridges used in the IEA-R1 reactor are made of expanded polypropylene with porosity equivalent to 10 μm. They have a cylindrical-annular geometry, 65 mm outer diameter, 25 mm inner diameter, 508 mm length. The dry, spent filter weights about 350g and the radioactivity content produce contact dose rates in the order of tens of millisievert per hour.

The IEA-R1 reactor is a pool-type research reactor, with operating power between 2 and 5 MW that uses water as the coolant, moderator and biological shield (Umbehaun et al., 2018). When filters become saturated and are unable to maintain the flow within the pre-established operational limits, they are replaced and disposed of as radioactive waste. After several weeks decaying in situ, they are sent to the Radioactive Waste Management Facility (RWMF), which is responsible for their treatment and interim storage as radioactive waste.

One of the first steps of radioactive waste management at the RWMF is the primary characterization, which is necessary to guide treatment processes and to establish the level of radiation protection required in the next steps. (IAEA, 2007). The characterization aims mainly at determining the radioisotope inventory of the waste. The inventory is needed for compliance with regulations and with the acceptance criteria for disposal (CNEN, 2002). Regulations require the radionuclide inventory being known with enough accuracy to ensure that the radiation safety objectives of the repository are met in the long term (IAEA, 2011).

The development of a primary characterization method, for determining the radioactive inventory of these filters is required for routine operation in the RWMF. One method of characterization is the radiochemical analysis of slices taken from each filter or from a representative sample. This method has the advantage that all radiologically significant radionuclides can be determined, as long as radiochemical methods with the required limits of determination, accuracy, and precision are available. This is particularly important for the determination of alpha or pure beta emitters in the waste, the so-called Difficult-To-Measure (DTM) radionuclides. However, the radiochemical method is used less frequently because of the high exposure and contamination risks incurred by the personnel, during sampling and analyses, and because of the high costs and the long time required

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for the determinations, both of which are serious difficulties in routine work of a waste characterization program.

An alternative method is the direct measurement of the gamma emissions of an individual piece of filter, or a whole waste packaging containing a number of filters, the estimate of the activity of the gamma emitters and the application of the Method of Scaling Factors (MSF) (IAEA, 2009). In this method, the concentrations of each DTM radionuclide are calculated as fixed proportions in relation to the concentrations of the measured gamma emitters in the filter element or whole packaging. These proportions, or scaling factors, can be calculated from concentrations determined previously in waste batches of the same type, from the same facility, operating under the same conditions (Geraldo et al., 2019). For the application of the MSF, the remaining problem is how to determine the concentration of each gamma emitter, for instance, by counting the photons reaching the detector in the measurement of gamma emission spectra.

The obvious solution is the 'calibration' of the measuring instrument, using a 'standard filter source' that contains the target gamma emitters with known concentrations, in a fixed source-detector geometry. In the routine work, the determination of the concentrations in the waste filters is based on the measurement of each filter in the same geometry and the comparison of the results with the 'standard'. However, 'standard filter source' was unavailable nor could be manufactured in our laboratory because not all calibrated solutions of the gamma emitter were available and because the hydrophobic nature of new filter elements impeded producing a homemade, homogeneous and reproducible standard source. All attempts to base the measurement process in a 'standard filter source' failed because of these difficulties.

The alternative way was to estimate the radioactive inventory of gamma emitters in waste filters by modelling the counting geometry of the gamma spectrometer and applying the dose-to-activity (DTA) method. It is a simple and easy way to estimate the radioactivity based on the measured dose rate from radioactive materials and has been employed as a reliable technique (Gedeon, 1996; IAEA, 2007; Ji et al., 2011; Ji et al., 2015) for waste characterization.

The method is not new, the original name was "dose-to-curie" (Helmholtz and Pyo, 1983; Stowe, 1987; Cline, 1993; Gedeon, 1996; Howell, 1999), which still appears in the literature with its original name (Rathbun and Anderson, 2010; Ji et al. 2011, 2015, 2017; Singledecker, 2012). The term DTA is here employed as preferable to dose-to-curie (DTC) or a more modern name, dose-to-becquerel (DTB) for this method.

DTA uses the dose rates measured in air with a portable radiation detector, the relationship between the measured photon emission rates evaluated by gamma spectrometry, and the combination of these data in a simulation of the counting geometry using point-kernel or Monte Carlo techniques to estimate the activity of each identified gamma emitter present in the waste.

The detectors used to measure the dose rates are the handheld survey meters commonly used in radiation protection monitoring, which are calibrated so as to indicate the effective dose rates, expressed in microsieverts per hour, that an individual would receive, as whole body dose, when exposed to the gamma radiation field in the same spatial point of measurement. The computational packages that are used to calculate the dose rates produced by gamma radiation sources can be set to estimate the same quantity, that is, the dose rate incurred by an individual at the point of interest in space. Consequently, measured and calculated dose rates are comparable and can be used in the method.

The gamma spectrometers can be fixed or portable. There are detectors with software packages in the market that uses the Monte Carlo N-particle (MCNP) to calibrate the gamma spectrometer in situ, but they are expensive. This work aims at presenting a procedure to estimate the activity of gamma emitters in filter cartridges, using the dose rates measured with handheld gamma detector, a fixed gamma spectrometry calibrated in energy and modelling of the measurement process by simulation with a commercial package for shielding calculations.

The ratios between the numbers of gamma rays of each energy detected by the spectrometer once known can be applied to estimate de proportion between the respective emitters, which avoids the necessity of calibrating the detectors for the measurement geometry. The deployment of a protocol for the primary characterization of the material is necessary so that the radionuclides and their respective activities are known with enough accuracy for the routine characterization work by the RWMD at IPEN.

2. Methods

2.1. Measurement of dose rates and gamma spectra

From a group of several dozen filters stored as waste, a sample of 15 old filters was selected with dose rates below about 0.03 mSv h^{-1}. This value was chosen to keep low the exposures of the personnel during the subsequent measurements and, at the same time, allowing the determinations with reasonable accuracy, for a sample of this size. Each selected filtration unit was individually wrapped in polyethylene bags and labeled.

The homogeneity of each filter was checked to assure that the modelling of dose measurements results in acceptable uncertainty in the results, which assumes that the distribution of activity is uniform. To this end, a shielding was constructed with lead bricks in the form of a tunnel, so that each filter could be completely shielded in the horizontal position (Fig. 1). The shielding was long enough to allow the filter to move along its axis without loss of shielding.

A beam-hole 1 cm in diameter in the central brick allowed the radiation detector to measure each turn the radiation emitted by a thin slice of the filter. The readings were taken at every 3 cm, totaling seventeen positions for each filter (Total length = 51 cm). According to IAEA (IAEA, 2007), waste can be considered homogeneous if the concentration of one radionuclide used as an indicator, in different parts of the waste, is within the range of ± 30% around the average concentration.

Subsequently, the dose rates were measured at the distances of 20, 40, and 60 cm from the surface of the unshielded filters in the median plane of the cylinder, using the Radiagem 2000 (Mirion, Radiagem 2000 Personal Portable Dose Rate and Survey Meter, USA), and the 6150 AD Automess detectors. The two detectors were used as a means of comparing results and detecting any discrepancies.

The filters were then positioned near a hyperpure germanium detector (Ortec, HPGe, model EPCG-15-190-R, USA), calibrated in energy. Each filter was measured during 600 s. The photopeak heights were corrected by the detector energy efficiency curve and the ratios between the peak heights were used as an estimate of the ratios of emission rates between any pair of photon energies of radionuclides in the filter. The ratios were input data for calculating the dose rates expected in the measurement positions, assuming that the proportions between the emission rates of photons of each energy are the same proportions that were detected. This simplification is acceptable since the differences in the self-absorption of radiation in the filters and the absorption in the air between the filter and the detector can be neglected, for the energies considered.

2.2. Simulation of the photon emission rates

Using the method of Point-Kernel described by Rockwell (Rockwell, 1956) and the MicroShield® software to simulate the counting geometry, and using the proportions of the detected intensities of each photon energy identified in the gamma spectrometry, the expected dose rates were calculated at each of the aforementioned measurement distances. In these calculations, the MicroShield® is used in the mode in which the individual photon energies and the emission rates are

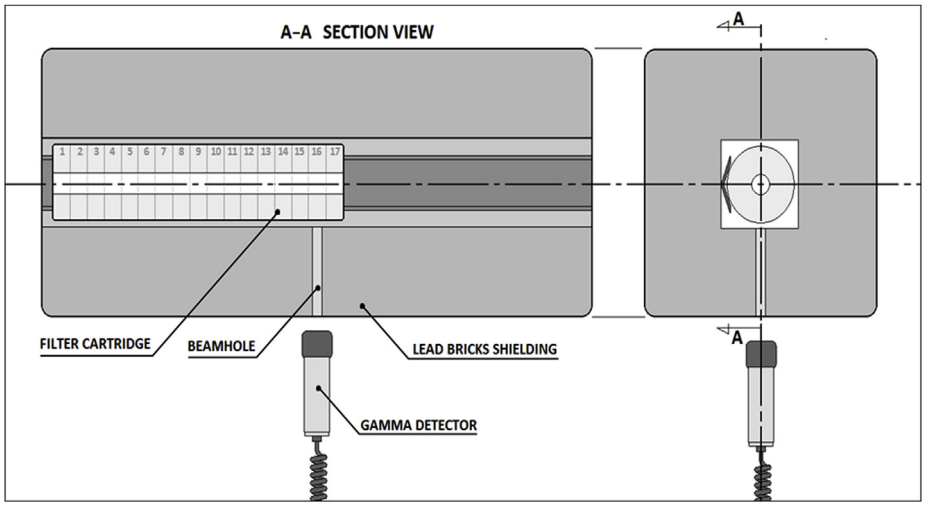

Fig. 1. Experimental arrangement for homogeneity test, using a survey meter (Automess GmbH, model 6150 AD, Germany).

specified.

Each photon contributes to the fluence at the measuring position, so that the dose rate$\dot{D}$, given in grays per hour, at a point P (Fig. 2), is given by the summation of the dose rates $\dot{D}_j$ produced by photons of each energy E_j. Fig. 2 presents, in an abridged form, the steps that are followed for the accomplishment of the filter characterization work.

In Fig. 2, B is the buildup factor, calculated for each energy j, Sv_j is the emission rate of photons of energy j, per unit of volume of the filter source, given in $s^{-1}\ cm^{-3}$, R_0 is the radius of the filter, given in cm; a is the distance from the measuring position to the filter surface, given in cm; Z_j is the self-attenuation distance of each photon of energy j inside the filter, given in cm; $F_j(\theta, b_i)$ is the integral expressed by Eq. (1).

$$F_j(\theta, b_j) = \int_0^{artg\left(\frac{h}{2a}\right)} e^{b_j \sec\theta} d\theta \tag{1}$$

where $b_j = \mu_{Sj}\ Z_j$, for μ_{Sj} the linear attenuation coefficient for the photons of energy j in the filter, in cm^{-1}.

The initial calculation of the dose rate at the measuring positions can be done with any values of Sv_j, as long as the proportions between the emission rates of any pair of photon energies agree with the detected rates determined previously and corrected by efficiency. Finally, the Sv_j values are adjusted so that the calculated dose rate coincides with the measured value. The adjustment is done by averaging the proportions between the values measured and calculated at the three distances from the filter and by multiplying the mean by the initial estimate of the Sv_j values. To check the accuracy of the method, the new calculated values of Sv_j of each energy are translated into the activity of the corresponding radionuclides, considering the yield of photon emission by decay. The activities are used to calculate the dose rate at the measurement positions, using Microshield. In these calculations, the software is used in the mode in which the radionuclides and their activities are specified.

Data input into the software was based on the selection of the "Cylinder Volume-Side Shields" modeling, with the following filter dimensions: source height = 50.8 cm, radius = 3.25 cm, calculated density = 0.207 g cm^{-3} and zero shielding. The energies and the number of photons per second calculated for each of the energies obtained by gamma spectrometry were entered.

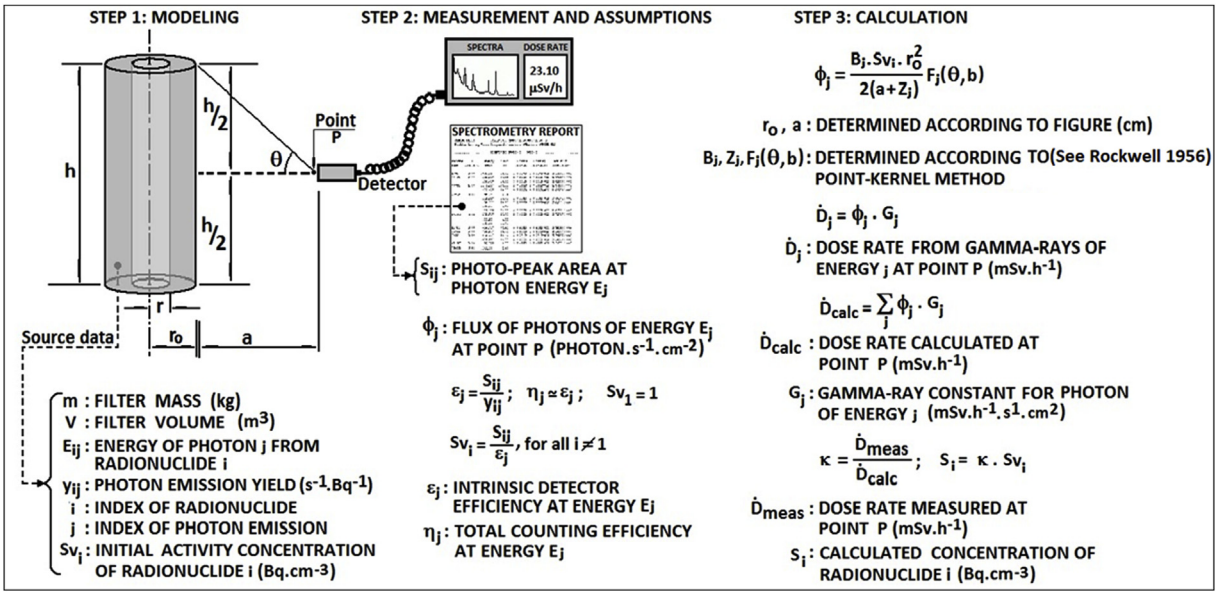

Fig. 2. Schematic representation of the method used in the primary characterization of filters.

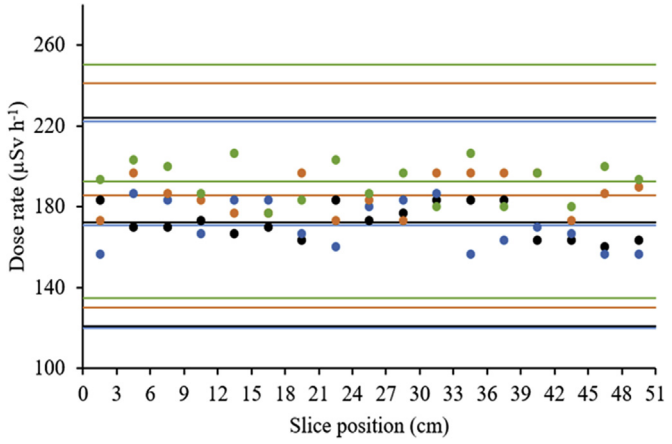

Fig. 3. Dose rates at the median plane of each slice. (orange) Sample A; (black) Sample B; (green) Sample C; (blue) Sample D. The colors of the lines coincide with the colors of the samples. Horizontal lines in the middle of the graph correspond to the average values, and the top and bottom lines correspond to each sample average ± 30%. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3. Results

The dose rate for each of the 17 parts (3 cm each) of the filter was measured using the Automess 6150AD (Canberra) detector. To decide whether the activity is homogeneously distributed in each filter, a limit of 30% variation around the mean was adopted as an envelope for all measurements, which is considered acceptable (IAEA, 2007).

Measurements on all samples provided results with a maximum coefficient of variation of ± 9.5% interval around the average values of the count rate obtained (Fig. 3).

3.1. Radionuclides identified

The gamma spectrometry using EPGC-15-190-R identified the same three gamma emitters in all fifteen filter-units measured, ^{60}Co, ^{108m}Ag and ^{110m}Ag. The radionuclides are activation products, shown in Table 1, with their nuclear properties and respective source of production in the reactor core, taken from Taddei (2013).

The fission product ^{137}Cs was not observed in any of the filters, although it is one of the main gamma-emitting radionuclides found in the reactor wastes. This indicates that the ^{137}Cs is fully dissolved in the cooling water and that its presence is undetectable either in precipitates or in molecules adsorbed onto the surface of the suspended particulate material in the water.

The list of other fission products that are gamma emitters and that are present in radioactive waste from a nuclear reactor is large (Taddei, 2013) but the radionuclides are either short-lived (^{86}Rb, ^{89}Sr, ^{91}Y, ^{95}Zr, ^{95}Nb, ^{99m}Tc, ^{103}Ru, ^{106}Ru, ^{125}Sb, ^{131}I, ^{132}I, ^{134}Cs, ^{140}Ba, ^{144}Ce, ^{141}Ce), or they are gaseous products (^{85}Kr, ^{133}Xe, ^{135}Xe), or they have a combination of long half-life with low fission yield and low gamma-emission rates (^{154}Eu, ^{155}Eu, ^{129}I, ^{94}Nb, ^{126}Sn) that makes them undetectable under the present measurement conditions. The same can be said about the activation products (^{24}Na, ^{28}Mg, ^{47}Ca, ^{46}Sc, ^{47}Sc, ^{48}Sc, ^{51}Cr, ^{54}Mn, ^{59}Fe, ^{57}Co, ^{58}Co, ^{65}Zn, ^{114}mIn, ^{115}Cd, ^{115m}Cd) that are short-lived, and (^{26}Al, ^{113m}Cd) that are long-lived but have low gamma emission yields.

These results are in agreement with those obtained before by Geraldo et al. (2019) who found only the same three gamma emitters, besides the DTM ^{63}Ni, ^{90}Sr, ^{234}U, ^{235}U, ^{238}U, ^{238}Pu, $^{239+240}$Pu, ^{241}Pu, ^{241}Am, ^{242}Cm, $^{243+244}$Cm. It is important to note that the radioisotope mass expressed as a summation, in the list above, indicates that the presence of the two isotopes are inferred but cannot be identified or quantified separately by the analytical method utilized. It is important to note too that other DTM may be present in the filters but were not analyzed by Geraldo et al. (2019).

3.2. Correction factor for the gamma emission rates

The source-detector counting geometry shown in Fig. 2 was simulated with the MicroShield® for three distances from the filter (20, 40 and 60 cm). An initial guess about gamma emission rates was tried and the results were compared with the dose rates measured with the Radiagen 2000 detector. The calculated dose rates result from guessed gamma emission-rates that obey the proportions obtained by comparing the peak areas of different radionuclides in the gamma spectrum, corrected by the intrinsic detector efficiency.

The correction factors presented in Table 2 are of the mean value of the ratios of the measured and the calculated dose rates for the three distances. Taking into account that the initial values of activity were the same in the simulations for all filters, the range of values for the correction factors observed in Table 2 only reflects the fact that the actual activities varied among the filters. All figures are of the same order of magnitude, as expected, because the filter sample was taken based on the initially similar measured dose rates of the filter sample, which is dose rates below about 0.03 mSv h^{-1}.

The dose rates were then recalculated using the initial guesses of gamma emission rates multiplied by the correction factors and the results were translated into the corresponding activity of each radionuclide by taking into account the emission yields of each gamma line. The calculated activity for each radionuclide in the filter units is presented in Table 3. The total estimated activity of the filters is in the order of a few megabecquerels.

Finally, the MicroShield® was used again to model the counting geometry presented in Fig. 2, but using as input the calculated activity in becquerels for each identified radionuclide, ^{60}Co, ^{108m}Ag and ^{110m}Ag. The calculated dose rates are presented in Fig. 4 together with the measured values, and it is noted that the measured and calculated dose rates are very close to each other, showing that the correction factors used and the calculated activity are good estimates of the true values. Fig. 5 shows the relative distance between measured and calculated

Table 1
Gamma-ray emitters detected in the filters.

Radionuclide	$t_{1/2}$	E_γ (keV)	I_γ (%)	Formation Reaction	Origin
^{60}Co	5.27 a	1173 1333	99.9 100	^{60}Ni(n,p)^{60}Co	Stainless Steel AISI 304 in the lining of the reactor pool
				^{61}Ni(n,np)^{60}Co	
				^{63}Cu(n,α)^{60}Co	Al-alloys ASTM 1060, 6262 and 6061 in the core structures
^{108m}Ag	418 a	723	91.3	^{107}Ag(n,γ)^{108m}Ag	Ag–In–Cd alloy of the neutron absorbing material in the control rods
		614	91.2	^{109}Ag(γ,n)^{108m}Ag	
		434	90.7	^{109}Ag(n,2n)^{108m}Ag	
^{110m}Ag	250 d	658	94.7	^{109}Ag(n,γ)^{110m}Ag	
		885	72.9	^{113}In(n,α)^{110m}Ag	
		938	34.3	^{110}Cd(n,p)^{110m}Ag	
		1384	24.3	^{111}Cd(n,np)^{110m}Ag	

Table 2
Correction factors of the proportions between measured and calculated dose rates.

Filter	Mean correction factors	Filter	Mean correction factors	Filter	Mean correction factors
1	173	6	169	11	232
2	291	7	212	12	206
3	223	8	218	13	215
4	201	9	239	14	196
5	251	10	183	15	199

Table 3
Estimated contribution of the identified radionuclide to the total activity of each filter.

Filter number	^{60}Co activity (Bq)	^{108m}Ag activity (Bq)	^{110m}Ag activity (Bq)	Total activity (Bq)
1	7.5E+05	1.8E+05	9.9E+05	1.9E+06
2	2.9E+06	2.1E+05	7.8E+06	1.1E+07
3	1.1E+06	2.5E+05	1.4E+06	2.8E+06
4	9.0E+05	2.2E+05	1.2E+06	2.3E+06
6	6.1E+05	1.8E+05	3.0E+05	1.1E+06
7	9.9E+05	1.0E+05	3.0E+06	4.1E+06
8	1.3E+06	1.3E+05	4.1E+06	5.5E+06
9	8.7E+05	2.6E+05	4.7E+05	1.6E+06
10	7.8E+05	1.9E+05	5.2E+05	1.5E+06
11	8.8E+05	2.1E+05	6.4E+05	1.7E+06
12	7.9E+05	1.9E+05	5.3E+05	1.5E+06
13	8.7E+05	2.4E+05	1.0E+06	2.2E+06
14	9.5E+05	2.7E+05	1.2E+06	2.5E+06
15	8.1E+05	2.5E+05	1.0E+06	2.1E+06

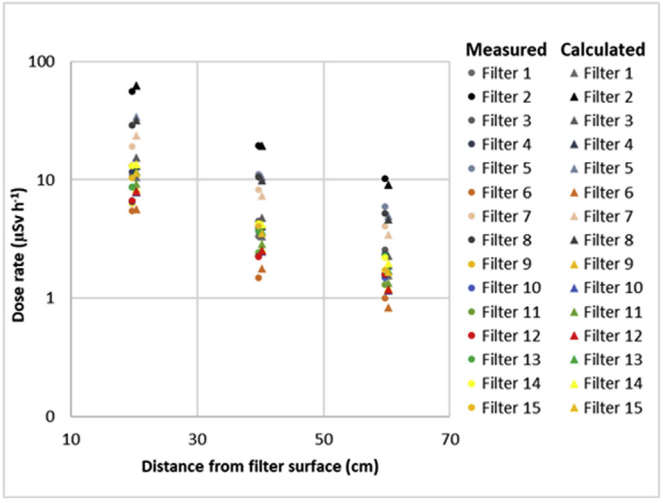

Fig. 4. Measured and calculated dose rates for all filters.

values. At 20 cm from the surface of the filters, the measured values are systematically below the calculated values, most probably because of the loss of detection efficiency as the incidence angle of photons emitted by the extremities of the filter. At 60 cm from the filter surface, measured values are systematically above the calculate figures, which can be explained by the averaging effect of the correction factors.

An estimate of the uncertainties in the results is yet unfeasible, as the process of calculation, using the method presented in Fig. 2 and (Rockwell, 1956), went through several steps of manual calculations based on interpolations in graphs whose errors still have to be estimated. The same occurs with the use of MicroShield® software, which does not provide an estimate of the uncertainties in the generated reports. Other sources of uncertainty are the measured dose rates that are accurate to 20% of the reading for the calibrated detectors, and the

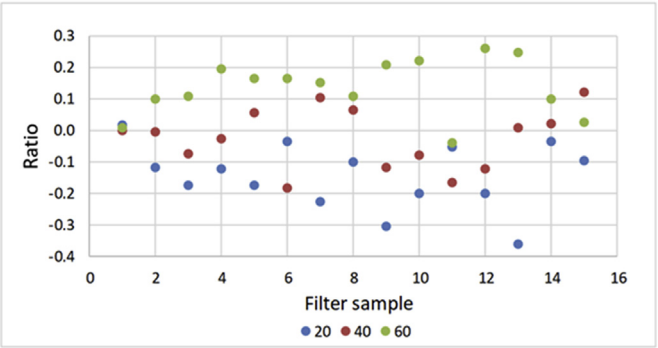

Fig. 5. Relative distance between measured and calculated values at the indicated distances: 20, 40, and 60 cm from the filter surface. Ratio means the difference between the measured and calculated values divided by the measured values.

attenuation of the gamma photons in the filter and in the air for different energies that were disregarded in the calculations of the proportions between two-photon peak areas.

4. Conclusions

The objective of this work was to develop a method to estimate the activity of the radioactive waste by measuring the dose rates and by capturing a gamma spectrum emitted by the waste, with handheld detectors and, through manual calculations or by using a software like MicroShield®, to estimate the activity of this waste.

As the handheld equipment was still unavailable by the time this work was performed, a fixed HPGe detector was used instead. This did not change the results as long as a portable spectrometer has an acceptable resolution in energy that allows recognizing individual gamma emission energies and measuring the areas of the photon peaks in the spectrogram. Portable equipment based on technologies that use HPGe or cadmium zinc telluride (CZT) detectors are examples of such instruments.

The method developed in this work is applicable for the cases in which sampling and laboratory measurement of the activity concentrations are less attractive because of high analytical costs, high exposure of personnel, or when it is not feasible to calibrate the source-detector counting geometry of whole packaging or individual waste objects with a standard source.

The method allowed to estimate the activity of spent cartridge filters collected as radioactive waste from the IEA-R1 nuclear research reactor. The purpose of this work was to contribute to the process of primary characterization of filter units in the routine waste management work of the RWMD of IPEN, where the IEA-R1 reactor operates.

One remarkable observation was the absence of ^{137}Cs in the cartridge filters, as initially expected, since this radionuclide is prevalent in all radioactive waste generated by a nuclear reactor facility. This may be due to the total dilution of the compounds of the element in the reactor cooling water or because any ^{137}Cs activity in the particulate matter suspended in the water is below the detection limit of the method.

The results obtained by both the manual calculation using the method presented and using the MicroShield® software were considered satisfactory. The calculated dose rates using the activity obtained by the method showed great congruence with the measured values. This indicates the possibility of using the method developed in the present work for the characterization of radioactive wastes in the operational routine of the radioactive waste management program.

CRediT authorship contribution statement

Ana Paula Gimenes Tessaro: Conceptualization, Methodology, Software, Validation, Formal analysis, Investigation, Resources, Data curation, Writing - original draft, Funding acquisition. **Leandro Goulart de Araujo:** Formal analysis, Data curation, Writing - original draft, Writing - review & editing, Visualization. **Roberto Vicente:** Conceptualization, Methodology, Formal analysis, Writing - review & editing, Visualization, Supervision, Project administration, Funding acquisition.

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