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Selectivity analysis of ^{99}Tc determination by LSC in the field of nuclear decommissioning

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Abstract
In this work, two Tc radiochemical isolation methods—a conventional method by Triskem TEVA® resin and a rapid method by Empore™ Tc Rad Disk—are compared in the field of nuclear decommissioning and ^{99}Tc assessment. The conventional method results more selective than the rapid one, being able to remove almost 100% of the main radiological interferers with the exception of the ^{90}Y; however, the rapid method obtains higher chemical yields (97% vs. 80%) and slightly lower detection limits (0.025 Bq vs. 0.030 Bq) than the conventional. Both methods are similar with regards to equipment and reagent costs.

Keywords ^{99}Tc determination · Nuclear decommissioning · Triskem TEVA® resin · Empore™ Tc Rad Disk · Selectivity analysis · Method comparison

Introduction

^{99}Tc is one of the most significant thermal neutron fission products of ^{235}U in nuclear reactors, with relatively high fission yield (6.1%) [1], and a half-life of 2.115×10^5 years [2]. It is one of the usual isotopes in the radionuclide inventory of nuclear power plants (NPPs) [3–6] and disposal of nuclear wastes; and therefore, must be considered in both the radiological characterization and the environmental impact of the nuclear installations under decommissioning and dismantling (D&D) processes.

Out of D&D processes, and due to its excellent properties as a low temperature superconductor and anti-corrosive material, ^{99}Tc is sometimes also present in carbon steel [7]. In the environment, it occurs naturally in small quantities in the Earth's crust [7] and it can be released from nuclear weapon tests and nuclear fuel cycle operations, especially those from nuclear reactors and fuel reprocessing plants [1].

Indeed, ^{99}Tc is mobile and soluble in oxygen conditions, and hence, it is present at trace level in solid or liquid form in different environmental compartments, such as seawater, sediments, soil, vegetation and aquatic and terrestrial organisms [1, 7]. Among its possible chemical forms (TcO_4^-, TcO^{2+}, $TcO(OH)^+$, $TcO(OH)_2$ and $TcO(OH)_3^-$), the pertechnetate ion (TcO_4^-) is the most widespread in nature, due to its high mobility between environmental components [8]. It is also a semi volatile element [1].

^{99}Tc is a medium-energy beta emitter (maximum energy of 293.8 keV, average energy of 94.6 keV) [2], so its measurement is commonly carried out by liquid scintillation counting (LSC), after sample treatment and Tc radiochemical isolation. Measurement is also possible by inductively coupled plasma mass spectrometry (ICP-MS), providing detection limits of two orders of magnitude lower than LSC (0.017 Bq for two counting hours), but is more expensive and therefore, less commonly-used for this purpose [1].

The sample treatment usually requires direct evaporation of water, ashing of organic matter or lixiviation or acid digestion of solid samples; and is carried out under specific conditions to avoid losses of volatile Tc [1].

The Tc radiochemical isolation implies different possible chemical techniques like ion exchange, liquid extraction, selective precipitation, extraction chromatography or combustion [1]. As there is no stable isotope of Tc to use as a carrier, chemical yield of the abovementioned Tc radiochemical isolation methods can be assessed using different tracers: stable rhenium (Re), electron capture ^{95m}Tc and gamma-emitting ^{99m}Tc [1].

However, it should be considered that Re and Tc could exhibit different chemical behaviours under certain

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conditions [1] and therefore Re will not always simulate the behaviour of Tc to be able to assess the chemical yield of the latter. Although ^{95m}Tc and ^{99}Tc are measured by LSC, ^{95m}Tc is a short-lived radionuclide, with a half-life of 61 days [9] and ^{99m}Tc requires a ^{99}Mo-^{99m}Tc generator and gamma spectrometry, so its use is complex and expensive. As a result, all these possible tracers have some constraints for laboratory use on a regular basis.

Among all these possibilities, in the laboratory of low activity measurements (LMBA) of the University of the Basque Country (UPV/EHU), the method applied for the ^{99}Tc determination in liquid and solid samples entails sample treatment by direct evaporation or acid digestion using a microwave oven, and Tc radiochemical isolation by ion exchange using Triskem TEVA® resin. To obtain the overall yield (the product of chemical yield and detection efficiency) we have decided to prepare two samples in parallel, one of them traced by ^{99}Tc, which can be applied to the other sample. It should be highlighted that currently this method is accredited by ENAC, the Spanish National Accreditation Body, under ISO 17025 [10].

Thus, in our routine environmental monitoring, radiochemical isolation by ion exchange on a TEVA®column provides pure ^{99}Tc spectra and high chemical yields. However, in the field of decommissioning other problems arise, not only because some of the samples are chemically highly complex, leading to low chemical yields, but also because some of the samples may contain high activities of other radionuclides potentially capable of interfering with ^{99}Tc during its measurement.

For these reasons, in this work an alternative Tc radiochemical isolation by using Empore™ Tc Rad Disk is studied and compared with the conventional one by using TEVA® resin, in terms of selectivity, chemical yield, detection limit and time and financial costs.

Rapid determination of ^{99}Tc using Empore™ Tc Rad Disks (Method RS551) [11] involves Tc radiochemical isolation with no prior preparation of the sample or the disk and measurement by a beta detector: a gas proportional counter, after drying and placing the loaded disk into a planchet, or a liquid scintillator spectrometer, after placing the wet disk into a vial. Therefore, it is a method with great potential to compare with the conventional method in terms of time, financial costs, and environmental impact.

In order to carry out this comparison, synthetic samples, traced by ^{99}Tc, have been prepared and analysed in the LMBA with a liquid scintillation spectrometer by following both methods in order to compare the chemical yields, detection limits as well as time and costs. With the aim of comparing the selectivity of both methods, samples containing different ^{99}Tc radiological interferers have also been prepared and then analysed in the LMBA.

Experimental

Material

In the analyses carried out, all the chemical reagents used have been of pro-analytical grade and the certified reference materials (CRM) used have been provided by the National Institute of Standards and Technology (NIST, USA), the National Physical Laboratory (NPL, UK) and the Isotope Products Laboratory (IPL, USA).

In this study, to measure ^{99}Tc, among the different liquid scintillation spectrometers at the LMBA, an ultra-low background liquid scintillation spectrometer 1220 QUANTULUS™, from PerkinElmer, has been used, which is able to detect low-, medium- and high-energy beta emitters as well as alpha emitters.

The liquid scintillation cocktail (Ultima Gold LLT) and 20 mL polyethylene vials for LSC have been purchased from PerkinElmer, TEVA® resin from Triskem International and Empore™ Tc Rad Disks from CDS Analytical.

Methods

Firstly, a review on the radionuclides present in light water reactor (LWR) and pressurized heavy water reactor (PHWR) plants [3–6], and therefore, susceptible to be measured in samples coming from D&D processes, has been carried out. These types of reactors have been chosen as they are presently the most common worldwide [12, 13].

Among the radionuclides obtained from this review, we have selected those radionuclides that are likely to interfere radiologically in the spectrum of ^{99}Tc obtained by LSC, when analysing D&D samples. In order to experimentally analyse these interferers, not all these radionuclides have been considered, but only those that meet the following three conditions: their expected activity should be almost > 0.1 ^{99}Tc activity; their decay mode would be beta or electron capture; and their half-life would be greater than 1.0 year (as decommissioning of NPPs takes, on average, from 15 to 60 years to be completed [3, 13]). Short-lived radionuclides have only been considered when being in secular equilibrium with their radioactive parent.

After establishing this list of interferers, synthetic samples containing these radionuclides have been prepared in order to study the level and impact of these radiological interferences in the presence of ^{99}Tc. In this regard, different 100 mL of 0.2 M HCl solutions have been prepared and each has been spiked with around 30 Bq of each interferer, an activity that is 1000 times greater than the expected ^{99}Tc detection limit (0.030 Bq). In addition, a similar

solution traced with ^{99}Tc has been prepared. The selection of HCl to avoid the attachment of Tc to the glass wall of a volumetric flask has been carried out considering the fitness for both studied Tc extraction materials: Triskem TEVA® resin [14] and Empore™ Tc Rad Disk [11].

Half of each dissolution has been passed through a column containing TEVA® resin, being finally added into a 20 mL polyethylene vial, together with 10 mL of Ultima Gold LLT scintillation cocktail from PerkinElmer, for LSC counting.

The other half of each dissolution has been filtered using an Empore™ Tc Rad Disk, at a flow rate of 5–10 mL min^{-1}. This value is around 10 times lower than the nominal flow rate of 50 mL min^{-1} [11]. However, according to D. M. Beals et al. [15], the best ^{99}Tc retention (almost 100%) is achieved with flow rates below 20 mL min^{-1}. Finally, the wet disk has been placed into a 20 mL polyethylene vial, according to Fons-Castells et al. [16], and mixed with 20 mL of Ultima Gold LLT for LSC measurement.

To conclude the process, vials have been stored for 6 h in the dark inside an ultra-low background liquid scintillation spectrometer 1220 QUANTULUS™ and measured by using low and high-energy measuring protocols (^{3}H and ^{14}C), to compare which is the most suitable for these types of measurements when considering sensitivity and figures of merit (efficiency^2/background). In the low-energy protocol, ^{99}Tc spectrum appears in the window defined between channels 25 and 600, whereas in the high-energy protocol, ^{99}Tc window is defined between channels 150 and 600.

Blanks and calibration samples are needed for both methods. Blanks have been prepared in the same way as samples; and calibration sources for ^{99}Tc and interferers, when necessary, have been prepared by spiking the resin and disks with a known amount of activity of ^{99}Tc and interferers from certified reference materials (CRM).

Test samples have been measured routinely for 6 h and blank samples for 12 h.

From the analysis of each sample traced with ^{99}Tc, following both the conventional and rapid methods, values for their detection limits, following the ISO standards 11929 [17], and chemical yields are obtained.

From the analysis of the samples traced with the interferers following both conventional and rapid methods, a sensitivity study has been carried out.

The interference in ^{99}Tc spectrum of each possible radionuclide interferer (i), I_i (%), has been calculated according to the following Eq. (1):

$$I_i = \left[\left(r_i - r_{0i}\right)/\left(A_i \cdot \varepsilon_i\right)\right] \cdot 100 \quad (1)$$

where: r_i is the gross count rate from radionuclide i in ^{99}Tc window, in s^{-1}; r_{0i} is the corresponding background count rate, in this window, in s^{-1}; A_i is the activity of the radionuclide i in the half of each dissolution, in Bq; $\mathcal{E}_i$ is the detection efficiency of radionuclide i in ^{99}Tc window.

To complete the study, an analysis of the financial costs of each method has been carried out. In the analysis, only costs of equipment and reagents used at the time of the study have been taken into account. In the equipment, the following are considered: the use of standard laboratory equipment, polyethylene vials and the liquid scintillation spectrometer, with its amortisation. Among the reagents used: acids, Triskem TEVA® resin or Empore™ Tc Rad Disk, Ultima Gold LLT and ^{99}Tc CRM. The analysis has been performed for the Spanish market and the results have been normalised to 100 for generalisation.

Results and discussion

According to the criteria described in the previous section and to cover a wide range of energy, from 5.8988 keV (^{55}Fe) to 2823.1 keV (^{60}Co) [2], the following radiological interferers in ^{99}Tc spectrum have been studied: ^{3}H, ^{55}Fe, ^{60}Co, ^{63}Ni, ^{90}Sr/^{90}Y, ^{137}Cs and ^{241}Pu.

Using the existing ^{14}C protocol for measurement in the 1220 QUANTULUS™ spectrometer, low-energy signals are neglected in the spectrum. Therefore, radionuclides with emissions of less energy than 66.980 keV (^{3}H, ^{55}Fe and ^{241}Pu) do not interfere in ^{99}Tc spectrum, where the spectrum window is defined between channels 150 and 600. Results of the tests carried out using this protocol are summarised in the following Table 1.

Table 1 Interferences obtained in ^{99}Tc spectrum (%) caused by ^{60}Co, ^{63}Ni, ^{90}Sr/^{90}Y and ^{137}Cs using TEVA® resin, Tc Rad Disk and ^{14}C protocol [2]

	^{60}Co	^{63}Ni	^{90}Sr	^{90}Y	^{137}Cs
Decay mode	β$^-$	β$^-$	β$^-$	β$^-$	β$^-$
Average energy (keV)	95.6	17.434	196	926.7	188
Half-life (y)	5.27	98.7	28.8	2.67 d	30.1
Interference using TEVA resin (%)	No	No	No	63.42 ± 1.33	No
Interference using Tc Rad Disk (%)	0.42 ± 0.01	No	0.14 ± 0.01	0.14 ± 0.01	0.16 ± 0.01

Data are provided with its standard deviation

No means that the interference is below 0.10%

As observed in Table 1, only ^{90}Y is retained in the resin, whereas ^{60}Co, ^{90}Sr/^{90}Y and ^{137}Cs are retained in the disk, but in much smaller amounts (< 1%). However, the shorter half-life of ^{90}Y allows interference to reduce over time. In Fig. 1, spectra obtained for 99 Tc and its interferers using 14 C protocol formeasurement are shown.

In contrast to ^{14}C protocol, when ^{3}H protocol is used, the entire range of energy is measured and hence, low-energy beta emitters (^{3}H, ^{55}Fe and ^{241}Pu) could interfere in ^{99}Tc spectrum, with a defined window from channels 25 and 600. Results of the tests carried out using this protocol are summarised in the following Table 2.

As we can see in Table 2, in the resin, apart from ^{90}Y, ^{55}Fe and ^{241}Pu are also retained; whereas in the disk, apart from ^{60}Co, ^{90}Sr/^{90}Y and ^{137}Cs, ^{55}Fe and ^{241}Pu are also retained, but in much smaller quantities (< 1%), except in the case of ^{241}Pu (around 10%).

These results are for defined activity concentrations of interferers. In the case of the resin, the results have demonstrated an aspect we already knew: its incapacity to adequately separate yttrium. In the case of the disk, the retention values for specific concentrations of interferers also show that data may vary with the concentrations of interferers; but, in all cases, the disks are not 100% selective for ^{99}Tc, especially for interferers with high activities, values expected in some of the samples from D&D. Thus, we believe that spectral deconvolution can be an appropriate tool, while still in development, for this set of problems.

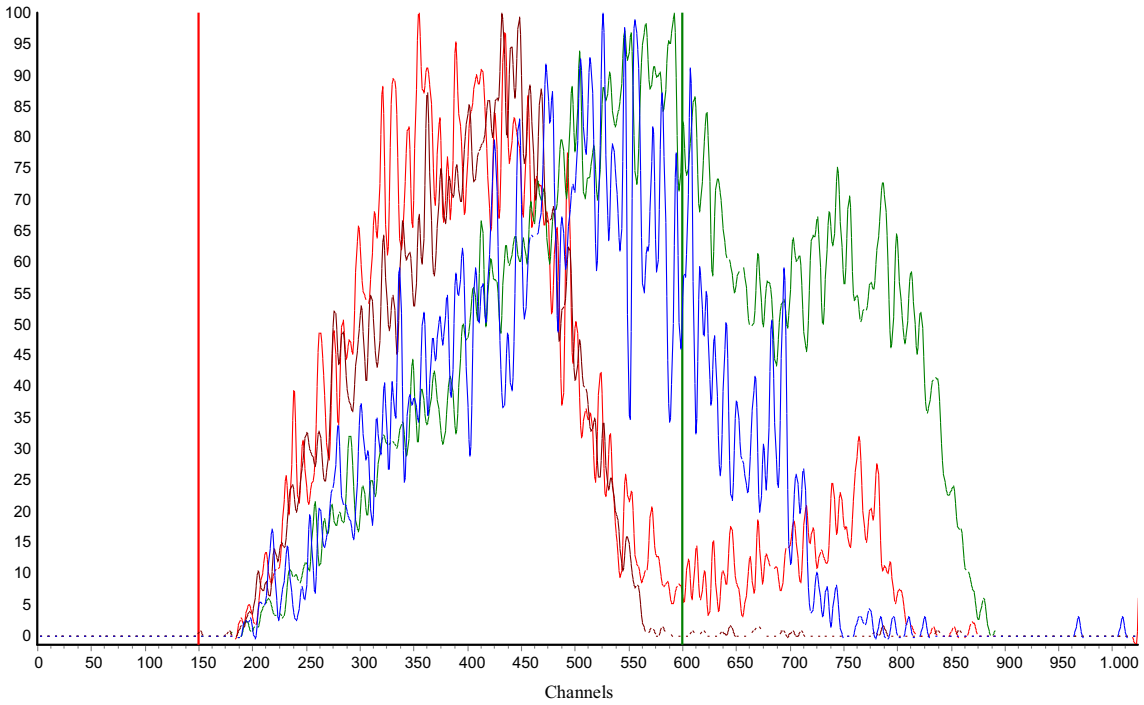

Fig. 1 Spectra of ^{60}Co (red), ^{90}Sr/^{90}Y (green), ^{99}Tc (brown) and ^{137}Cs (blue) using ^{14}C protocol. Window for ^{99}Tc is between channels 150 and 600. (Colour figure online)

Table 2 Interferences obtained in ^{99}Tc spectrum (%) caused by ^{3}H, ^{55}Fe, ^{60}Co, ^{63}Ni, ^{90}Sr/^{90}Y, ^{137}Cs and ^{241}Pu using TEVA® resin, Tc Rad Disk and ^{3}H protocol [2]. (Colour figure online)

	^{3}H	^{55}Fe	^{60}Co	^{63}Ni	^{90}Sr	^{90}Y	^{137}Cs	^{241}Pu
A	β^-	EC	β^-	β^-	β^-	β^-	β^-	β^-
B	5.68	–	95.6	17.434	196	926.7	188	5.8
C	12.3	2.75	5.27	98.7	28.8	2.67 d	30.1	14.3
D	No	10.60 ± 0.14	No	No	No	61.03 ± 1.19	No	14.30 ± 0.09
E	No	0.68 ± 0.01	0.31 ± 0.01	No	0.107 ± 0.001	0.107 ± 0.001	0.100 ± 0.003	9.86 ± 0.09

Data are provided with its standard deviation

A: decay mode (EC: electron capture), B: average energy (keV) for β^- emitters, C: half-life (y), D: interference using TEVA® resin (%) and E: interference using Tc Rad Disk (%). No means that the interference is below 0.10%

It is important to point out that, although ^{241}Am has not been considered as a possible interferer in ^{99}Tc spectrum according to the criteria established in this work, being an alpha-emitter [2], its retention has been assessed together with ^{241}Pu, taking advantage of its presence in ^{241}Pu standard solution.

Certainly, ^{241}Am is retained in both resin and disk with ^{241}Pu, but essentially does not interfere in ^{99}Tc spectrum due to the high energy (5637.82 keV) of its alpha particle [2].

In Fig. 2, spectra obtained for ^{99}Tc and its interferers using ^{3}H protocol for measurement are shown.

As shown in Fig. 2, when applying the ^{3}H protocol, there is also a clear overlapping of signals coming from the studied radionuclides. This highlights the spectral interference issue that, as mentioned before, may be solved by spectrum deconvolution or some additional chemical isolation steps.

In order to clarify which protocol could provide better results when measuring ^{99}Tc, they are compared in terms of sensitivity, efficiency, background and figure of merit (efficiency^2/background) in Table 3.

From the results in Table 3, it can be concluded that the ^{14}C protocol leads to spectra with fewer interferers than the ^{3}H protocol, with both radiochemical isolation method. Besides this, although ^{3}H protocol provides a better efficiency than ^{14}C protocol, the background is significantly higher by using it; and hence, the figure of merit with ^{14}C protocol is better than that with ^{3}H protocol, using both methods. Therefore, the ^{14}C protocol results more suitable than ^{3}H protocol for measuring ^{99}Tc.

Finally, focusing on the measurements carried out with ^{14}C protocol, the studied Tc radiochemical isolation methods are compared regarding sensitivity, ^{99}Tc chemical yield,

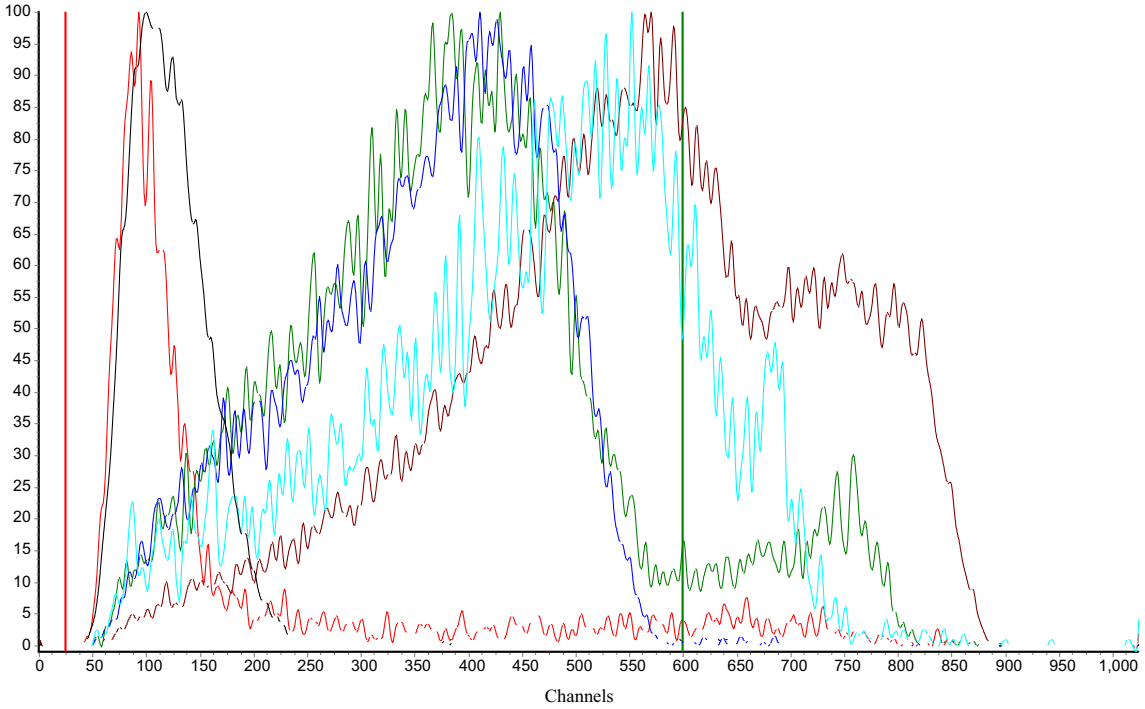

Fig. 2 Spectra of ^{55}Fe (red), ^{60}Co (green), ^{90}Sr/^{90}Y (brown), ^{99}Tc (dark blue), ^{137}Cs (light blue) and ^{241}Pu (black) using ^{3}H protocol. Window for ^{99}Tc is between channels 25 and 600

Table 3 Comparison of ^{3}H and ^{14}C measurement protocols using TEVA® resin and Tc Rad Disk

	TEVA resin		Tc Rad Disk	
Protocol	^{14}C	^{3}H	^{14}C	^{3}H
Sensitivity	1 interferer	3 interferers	4 interferers	6 interferers
^{99}Tc efficiency (%)	81.25 ± 1.19	97.32 ± 1.43	80.25 ± 0.35	96.12 ± 0.34
^{99}Tc background (cpm)	1.27 ± 0.06	2.48 ± 0.06	1.24 ± 0.08	3.08 ± 0.05
Figure of merit	0.52 ± 0.02	0.38 ± 0.01	0.52 ± 0.03	0.30 ± 0.01

Data are provided with its standard deviation

Table 4 Comparison of TEVA® resin and Tc Rad Disk using ^{14}C measurement protocol

	TEVA resin	Tc Rad Disk
Sensitivity	1 interferer	4 interferers
^{99}Tc chemical yield (%)	80 ± 1	97 ± 1
Detection limit (Bq)	0.030	0.025
Chemical isolation time	3 days	30 min
Relative financial costs	100%	106%

Data are provided with standard deviation

detection limit, chemical isolation time and financial costs (Table 4).

^{99}Tc chemical yield using TEVA® resin is around 80%, whereas using Tc Rad Disk is 97%, which leads to a 20% lower detection limit, using the same sample volume (50 mL) and measurement time (6 h) (Table 4). Apart from this, conventional Tc radiochemical isolation takes, on average, 3 days; but using disks, isolation reduces to 30 min. Regarding equipment and chemicals costs, both methods result similar, although the conventional method involves the use of more chemical reagents.

Conclusions

Two ^{99}Tc radiochemical isolation methods have been compared, a conventional one by Triskem TEVA® resin and a rapid method by Empore™ Tc Rad Disk. This comparison has been carried out in the field of nuclear decommissioning. In contrast to samples from routine environmental monitoring, samples from nuclear decommissioning are chemically very complex and may contain high activities of other radionuclides potentially capable of interfering with ^{99}Tc during its measurement. Among these potential interferers, ^{3}H, ^{55}Fe, ^{60}Co, ^{63}Ni, $^{90}Sr/^{90}Y$, ^{137}Cs and ^{241}Pu have been chosen after analysing radionuclide inventories of light water reactor (LWR) and pressurized heavy water reactor (PHWR).

After that, a set of samples containing these radionuclides and others containing ^{99}Tc have been prepared and ^{99}Tc radiochemical isolation following both methods have been performed. Then, a set of measurements using ^{3}H and ^{14}C protocols and an ultra-low background liquid scintillation spectrometer 1220 QUANTULUS™ has been carried out.

^{3}H and ^{14}C protocols have been compared in terms of sensitivity, efficiency, background and figure of merit; ^{14}C protocol being the most suitable for measuring ^{99}Tc, with fewer interferers and achieving higher figures of merit than ^{3}H protocol.

Focusing on the measurements accomplished with ^{14}C protocol, the conventional method by Triskem TEVA® resin results more selective than by Empore™ Tc Rad Disk. In the resin, only ^{90}Y is retained and its interference reduces over time due to its short half-life. However, in the disk, ^{60}Co, $^{90}Sr/^{90}Y$ and ^{137}Cs are retained, but in much lower amounts (< 1%). Possible solutions to reduce these interferences may be spectrum deconvolution or the addition of some chemical isolation steps. In any case, the use of the disk makes it a rapid method with high chemical yields for ^{99}Tc, which allows the application of lower detection limits.

Regarding equipment and chemical costs, both isolation methods result similar, although the conventional method requires more chemical reagents than the rapid one. Therefore, the rapid method has a lower impact on the environment than the conventional.

In conclusion, if applying other measurement techniques, it is known that there is no interferer in the sample, the Rad disk method is faster and presents lower detection limits than the resin method; but if there are interferers in the sample, the resin has to be chosen over the Rad disk as it is more selective.

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