

# Efficiency calibration and optimization of a liquid scintillation spectrometer for low level $^{55}\text{Fe}$ and $^{63}\text{Ni}$ measurements

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## ABSTRACT

An experimental study on the counting efficiency, background, and Lower Limit of Detection (LLD) of a liquid scintillation spectrometer was carried out for establishing a measurement technique suitable for low level activity measurements of  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  in environmental samples. The spectrometer's response, using two scintillators (PerkinElmer Optiphase Hisafe 3 and Meridian Prosafe HC+) and glass and plastic vials, was studied. Colour and chemical quenching effects were also investigated for both scintillators and vials. Finally, a spectrum integration window selection method able to reduce the LLD through a Figure of Merit (FOM) optimization, was tested. Efficiency and LLD curves were obtained for both scintillators and types of vials with this technique.

## 1. Introduction

$^{55}\text{Fe}$  and  $^{63}\text{Ni}$  are two radionuclides known to have environmental impact in the vicinity of nuclear installations during operation and, specially, during decommissioning (Gudelis et al., 2010), (Skwarzec et al., 2006). They are mostly produced by neutron activation of stable Fe and Ni present in the construction materials of the nuclear installation.

$^{63}\text{Ni}$  is a weak  $\beta$  emitter with  $E_{\beta\text{max}} = 66.977$  keV and  $T_{1/2} = 98.7 \pm 2.5$  y (Erjun and Junde, 2001).  $^{55}\text{Fe}$  ( $T_{1/2} = 2.747 \pm 0.008$  y) decays through electron capture emitting consequently two Auger electrons, with energies 0.61 keV and 5.19 keV and five X-ray emissions, with energies ranging between 0.64 keV and 6.49 keV (Junde, 2008). In both cases, very low energy electrons are emitted, which make them particularly difficult to determine by means of conventional radiometric techniques.

Different systems have been used to measure these radionuclides in the past, including gas counters and solid-state detectors for  $^{55}\text{Fe}$  (Hou et al., 2005a), (Peng, 1966) and  $^{63}\text{Ni}$  (Verzezen et al., 2008), (Villa, 2003). Due to the low energy emissions of both radionuclides, especially in the case of  $^{55}\text{Fe}$ , an internal counting technique is needed in order to obtain a competitive detection efficiency. This has been done with gas counters (Rivera-Silva et al., 2019), (Douglas et al., 2016), which need an additional electrodeposition step during source preparation. The source also needs to be thin enough to allow the detection of the low

energy Auger electrons and X-rays, which is a limitation to the amount of iron in the sample. A similar difficulty is found for the measurement of  $^{63}\text{Ni}$  with Si semiconductor detectors, as only a thin source may be used due to the stopping of low energy electrons by thick sources. Liquid scintillation counting (LSC) is our radiometric technique of choice for convenience reasons, as it is an internal counting technique that doesn't show the disadvantages of an internal gas counter, making it sensitive and efficient enough to carry out studies on the environmental impact of  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  simultaneously, as well as determining their activity concentrations in nuclear waste (Hou et al., 2005b), (Gibson and Lally, 1971).

As a result of the sample matrix constituents and the radiochemical separation that must be performed to measure real samples, quenching will be present in variable amounts (Hou et al., 2005a). This is of crucial importance in LSC as colour and chemical quenching affect counting efficiency, so calibrations must be done prior to the measurement of a sample with an unknown activity (Peng, 1966).

Background is the other main parameter of study in this work as it is aimed to the measurement of environmental samples. Instrumental background may be used for the calculation of the Lower Limit of Detection (LLD). It is of our interest to reduce this parameter as far as reasonable for the purpose of environmental impact studies, particularly in the case of environmental restoration during the decommissioning of a nuclear installation. As the background spectrum is not homogeneous, it will vary depending on the choice of integration window. This results

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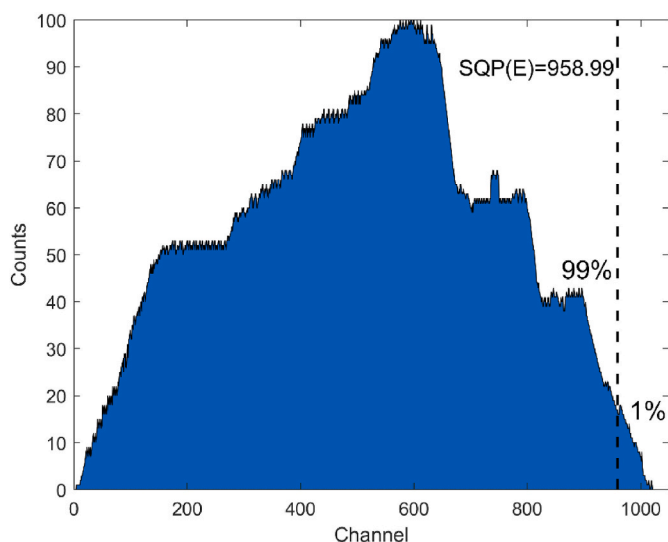


Fig. 1. Example of a Compton spectrum obtained with a  $^{226}\text{Ra}$   $\gamma$  source. 99% of the counts are located in the channel interval  $[1, \text{SQP}(E)]$ , while the remaining 1% is in the interval  $[\text{SQP}(E), 1024]$ .

in an opportunity for LLD reduction by choosing the appropriate integration window. A method for achieving a decrease of the LLD, through a Figure of Merit optimization, has been tested in this work.

Quenching, counting efficiency, background, and Lower Limit of Detection are known to be affected by the liquid scintillator (Verrezen et al., 2008) and type of vial selected for the measurements. With the objective of achieving a further LLD reduction, these dependences are well established for several different combinations of measurement materials and levels of quenching to obtain calibrations and compare the performance of every combination specifically for the case of environmental samples.

For the improvement of environmental measurements, this study was aimed at the reduction of the LLD without making improvements in the radiochemical procedure, but by scintillator, vial and summing window selection method choice. This is addressed in the following sections by the display and discussion of different experiments. The effect of quenching on LSC measurements has been studied in the past (Villa, 2003), (Rivera-Silva et al., 2019). However, our study includes some of the first data of the effect of quenching in  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  determinations with LSC as far as we are aware.

## 2. Experimental

Samples spiked with a known activity of  $^{55}\text{Fe}$  or  $^{63}\text{Ni}$  and different amounts of quenching agent were used to obtain different counting efficiencies, while blank samples were used for background measurement and LLD calculations. Each spiked sample had a respective blank sample with the same amount of quenching agent so LLD dependence on quenching can be established. This was done for the two scintillation cocktails and vials of choice.

### 2.1. LSC spectrometer

A Quantulus 1220 system has been used for this study. As it is known (Douglas et al., 2016), it is very well suited for low level counting with a passive shielding consisting of Pb, Cd and Cu and an active shielding with two photomultiplier tubes connected in anticoincidence with the main two photomultiplier tubes. The response of this system for  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  radioactive emissions is studied in this paper. In this spectrometer, the effect of colour and chemical quenching is evaluated using the SQP(E) parameter. It is determined by irradiating the counting vials with an external  $^{226}\text{Ra}$   $\gamma$ -source and acquiring a Compton spectrum, which is

expected to be affected by quenching. The SPQ(E) is a channel of the Compton spectrum defined in a way that the channel interval between 0 and SPQ(E) includes 99% of the total Compton spectrum area. The SQP(E) changes as the quenching effects appear (Peng, 1966). For that, it serves to evaluate their influence on the spectrum. The spectrum used for the determination of SQP(E) is illustrated in Fig. 1. The spectrometer also allows for consecutive measuring of sets of up to 60 samples.

### 2.2. Scintillation cocktails

Two different commercial liquid scintillators were studied. Meridian ProSafe HC+ and PerkinElmer OptiPhase HiSafe 3. Meridian ProSafe HC+, is an environmentally friendly liquid scintillator recommended for more concentrated samples, specifically those dissolved in  $\text{H}_3\text{PO}_4$ , as  $^{55}\text{Fe}$  samples typically are. PerkinElmer OptiPhase HiSafe 3 is a well-known liquid scintillator of general use. They were compared in terms of efficiency, LLD and behaviour for quenching effects.

17.5 ml of scintillator was added to each vial together with 2.5 ml of the tracer solution and different amounts of a (colour or chemical) quenching agent to a total of 20 ml scintillation cocktail with the objective of measuring in the same conditions as a real sample (Hou et al., 2005b).

For each spiked vial with different amounts of quenching agents, a blank vial was prepared at the same quenching conditions but with 2.5 ml of very low  $^3\text{H}$  activity water mixed with the same acid as that in the tracer solution. These vials were used to obtain the background and LLD.

### 2.3. Tracer dissolutions: $^{55}\text{Fe}$ and $^{63}\text{Ni}$

The tracer solutions were provided by the Laboratoire Etalons d'Activité (LEA) (France).  $^{55}\text{Fe}$  activity was  $(127.662 \pm 6.383)\text{Bq/g}$  as of 04/11/2020, being the serial number for the sample 797251/3.  $^{63}\text{Ni}$  activity was  $(209.945 \pm 7.348)\text{Bq/g}$  as of 04/11/2020, with a serial number 7767D1/2. Spiked samples were measured for 15 min, while each background vial was measured for 10h.

$^{55}\text{Fe}$  samples were dissolved in 1M  $\text{H}_3\text{PO}_4$  and their activities ranged from 61Bq to 50Bq, depending on the run. ( $^{55}\text{Fe}$  half-life is short enough to make necessary to correct the activity for decay). In the case of  $^{63}\text{Ni}$  the activities of the tracer solutions remained constant and around 105Bq over the time period this work was carried out. 3M  $\text{HNO}_3$  was used as the medium for  $^{63}\text{Ni}$ .

### 2.4. Quenching agents: colour quenching and chemical quenching

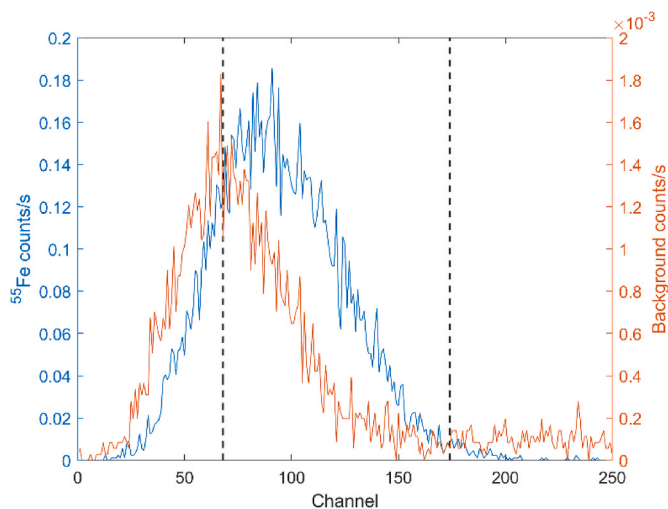
Different degrees of quenching were studied to obtain efficiency calibrations. Colour quenching was introduced using analytical grade  $\text{Na}_2\text{CrO}_4$  due to its availability and capacity to colour aqueous solutions. In real samples, quenching appears often due to elements present in the original sample or the different reagents used for radiochemical separation. This the case of colour quenching in  $^{55}\text{Fe}$  samples, produced by the addition of stable Fe (Rapkin and Gibbs, 1963). In this example, colour quenching will be produced by the total Fe inside the vial in real samples but introduced artificially with  $\text{Na}_2\text{CrO}_4$  for our calibrations. This difference in the origin of the quenching is not an issue due to the SQP(E) directly measuring the quenching, either colour or chemical, regardless of the cause of it.

$\text{CCl}_4$  (Gibson and Lally, 1971) was used for chemical quenching for its strong quenching capabilities and solubility in the scintillators. Efficiency and LLD curves were determined for every combination of vial type, scintillation cocktail and radionuclide.

$\text{Na}_2\text{CrO}_4$  amounts added were 0, 4, 8, 12, 16, 20, 40, 60, 80 and 100  $\mu\text{mol}$ , while  $\text{CCl}_4$  was 0, 10, 20, 30, 50, 70 and 90  $\mu\text{l}$ .

### 2.5. Vials

The effect of the selected counting vial in the response of the counter



**Fig. 2.**  $^{55}\text{Fe}$  and background spectra. The starting and ending channel of the window with the greatest FOM is represented with two vertical lines.

was studied. Glass and polyethylene vials provided by Meridian were used and their efficiencies, behaviour to quenching effects and LLDs compared.

Glass vials are known to yield higher backgrounds than polyethylene vials due to the presence of  $^4\text{K}$  in their composition (Rapkin and Gibbs, 1963).

## 2.6. Efficiency and LLD calculations

For efficiency calculations, total counts for each spiked sample are obtained by spectrum integration. To do this, an integration window must be chosen. Two window selection methods were compared: A continuum-wide counting window for maximizing counting efficiency, and a counting window which maximizes the Figure of Merit of the experiment that is expected to yield lower LLDs.

FOM is defined as (Oeschger and Wahlen, 1975):

$$\text{FOM} = \frac{(\varepsilon \cdot V)^2}{B} \quad \text{Eq. 1}$$

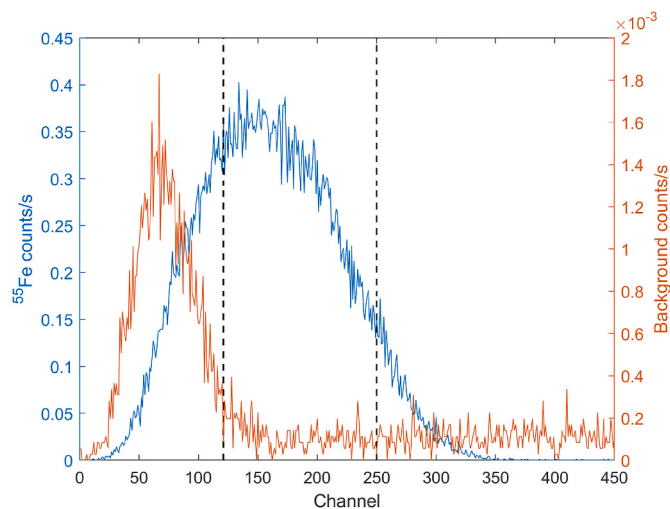
where  $\varepsilon$  is the counting efficiency,  $V$  the sample volume and  $B$  the background counts per unit time. The LLD is defined as (Currie, 1968):

$$\text{LLD} = 4.66 \frac{\sqrt{B/t}}{\varepsilon}, \quad \text{Eq. 2}$$

$t$  being the measurement time of our spiked sample.  $B$  and  $\varepsilon$  depend on the window selected to integrate the spectrum. Thus, FOM can be maximized by using an adequate choice of such window which in turn should minimize the LLD. It must be considered that  $B$  and  $\varepsilon$  depend on the degree of quenching. In other words, the optimization of FOM and LLD depends on the degree of quenching, and this is what we do in the following.

This optimal window determination was carried out by a script developed specifically for this task. It works as follows and was used for every spectrum obtained for this work:

1. Reading of a spiked sample spectrum file and its respective background spectrum, which was obtained by measuring a blank vial of similar SQP(E).
2. Calculation of FOM for every possible combination of starting and ending channel for count summing.
3. Determination of the greatest FOM obtained and its respective starting and ending channels. This establishes the optimal counting window for a certain SQP(E).



**Fig. 3.**  $^{63}\text{Ni}$  and background spectra, with the optimal integration window represented with two vertical lines.

**Table 1**

Comparison of the two integration methods for the measurement of  $^{63}\text{Ni}$  in polyethylene vials and the cocktail Meridian Prosafe HC+.

Sample name	Efficiency (%) (spectrum-wide integration)	LLD (Bq) (spectrum-wide integration)	Efficiency (%) (FOM optimization)	LLD (Bq) (FOM optimization)
Ni-51	55.4 ± 2.2	0.0914	36.9 ± 2.1	0.0517
Ni-75	56.0 ± 2.2	0.0904	37.3 ± 2.1	0.0511
Ni-76	51.7 ± 2.0	0.0865	33.8 ± 1.8	0.0599
Ni-77	53.2 ± 2.1	0.0841	36.6 ± 1.9	0.0579
Ni-78	48.2 ± 1.9	0.0797	32.2 ± 1.7	0.0649
Ni-79	45.5 ± 1.8	0.0692	27.1 ± 1.6	0.0635
Ni-80	42.7 ± 1.7	0.0738	23.8 ± 1.6	0.0690

4. Efficiency and LLD for this optimal counting window are determined and saved in a separate file.

Figs. 2 and 3 show the effect of applying this algorithm to  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  spectra respectively. A narrower window, where most of the background is left out, is automatically selected by the script.

In order to apply this method in the case of the measurement of a real sample with an unknown activity, a spiked calibration vial would be needed so that the optimal starting and ending channels can be determined for the degree of quenching present in the real sample. An alternative approach is to perform a calibration of the optimal integration window for different degrees of quenching by means of spiked vials with different amounts of a quenching agent. This is possible since from an experimental viewpoint the effects of quenching in the spectrum shift are independent of the quenching agent.

## 3. Results and discussion

### 3.1. LLD optimization by using FOM

With the previously described script, FOM-optimized efficiencies and LLDs were obtained. As an example, Table 1 shows a comparison between some results obtained with this method and those from the traditional beta continuum-wide window selection method to address the advantages of this technique.

The use of the spectrum-wide integration method gives higher counting efficiencies but also higher LLD for the combination of polyethylene vials and Meridian Prosafe HC + scintillator. As expected, the use of this FOM-optimization method for spectrum integration reduces

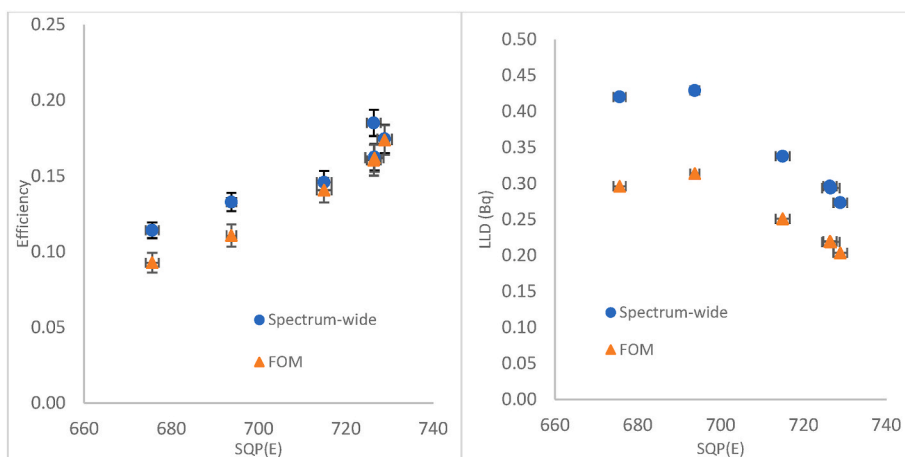


Fig. 4. Efficiency and LLD obtained for different degrees of quenching for <sup>55</sup>Fe in polyethylene vials and Meridian cocktail. The FOM-optimizing method provides a lower LLD by compromising some efficiency.

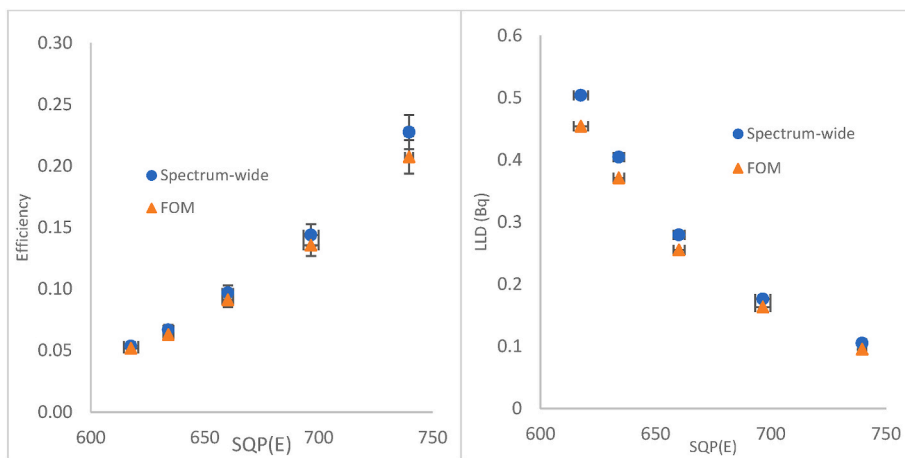


Fig. 5. Efficiency and LLD obtained for different degrees of quenching for <sup>63</sup>Ni in polyethylene vials and Optiphase cocktail. Switching from the traditional spectrum-wide integration method to a FOM-optimizing integration method doesn't affect neither efficiency nor LLD meaningfully.

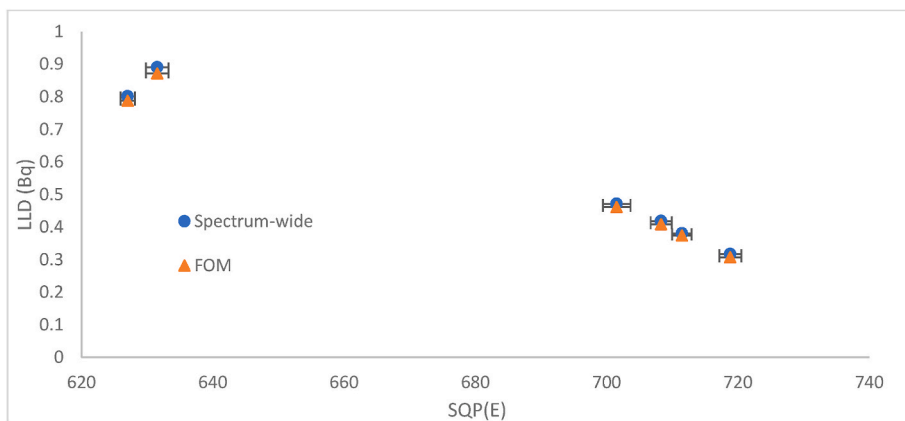


Fig. 6. LLD obtained for different degrees of quenching for <sup>55</sup>Fe in glass vials and Meridian cocktail. Switching from the traditional spectrum-wide integration method to a FOM-optimizing integration method doesn't affect neither efficiency nor LLD meaningfully.

the efficiency but minimizes LLD. This makes this method more convenient when measuring low activity concentration samples.

In the case of <sup>55</sup>Fe in polyethylene vials and Meridian Prosafe HC + scintillator, the results are revealed in Fig. 4. There is a significant lowering in LLD for all the studied quenching values when using the of

FOM optimization method. The counting efficiency also decreases in general but to a less extent in relative terms to the values found for the wide-spectrum integration method, the reason being the <sup>55</sup>Fe spectrum shape. Furthermore, no advantages can be observed when using the Optiphase scintillator and polyethylene vials (Fig. 5). Both integration

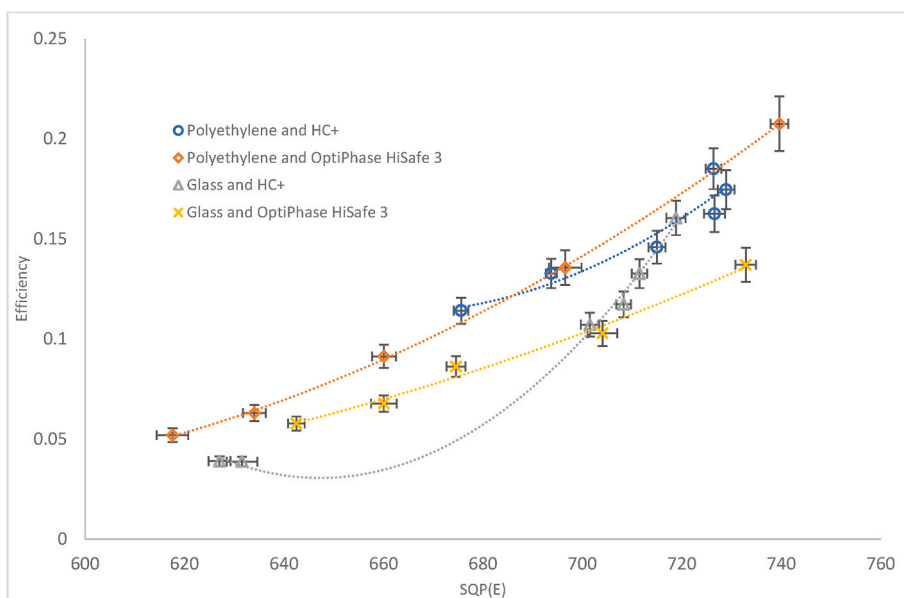


Fig. 7. Comparison of efficiencies and their quadratic calibrations for all <sup>55</sup>Fe combinations.

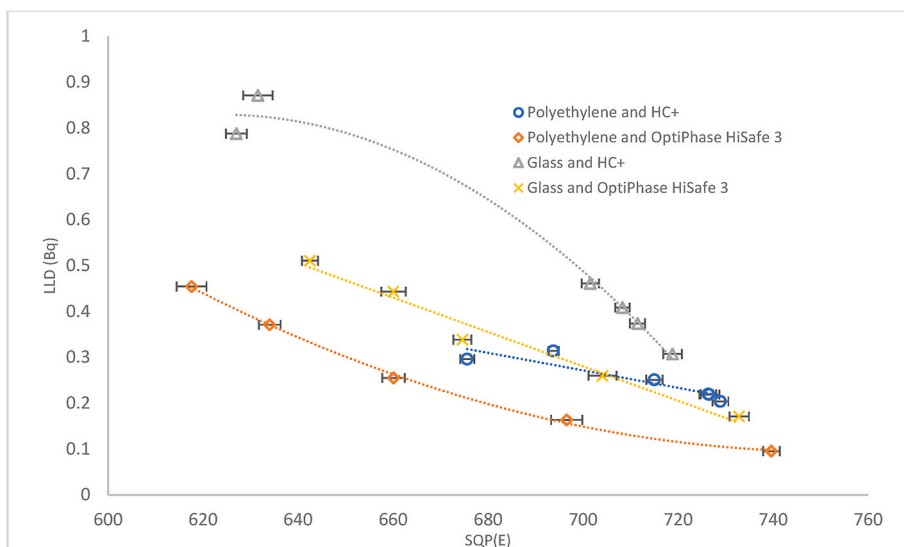


Fig. 8. Comparison of LLDs for all <sup>55</sup>Fe combinations and their quadratic calibrations except for polyethylene and HC+, which is linear.

methods give practically the same values for  $\epsilon$  and LLD. This is also the case when using glass vials and Meridian Prosafe HC + scintillator, as shown in Fig. 6. There is no difference among the observed values of the LLD over the analysed range of quenching values.

Summing up, an important reduction in LLD is achieved by using polyethylene vials and Meridian Prosafe HC + scintillator. None of the other vial and cocktail combinations show this property clearly, leaving the efficiency and LLD very close to the traditional spectrum summing method. The FOM-optimization method can be effective or not depending on the detecting medium and container.

### 3.2. Counting efficiency curves for the different scintillation cocktail and vial combinations

The FOM-optimization method was selected to find the counting efficiency calibration curves as function of the quenching effects as measured by SQP(E). Thus, all graphs shown in this part are plotted using FOM-optimizing calculations, as our calibrations will be used for

low-level samples in the future. It can be seen that most of the calibrations turn quadratic as usual in LSC, but in some cases linear fits are preferable due to lower uncertainties and acceptable values of  $R^2$ .

Fig. 7 through 10 show our results. As a general rule it can be seen how the use of glass vials increases the LLD independently of the SQP(E) value. For that, glass vials might not be used for measuring the two radionuclides of interest in the case of lower activities, as they produce a higher background, mainly due to the presence of <sup>4</sup>K in their composition as mentioned earlier (Rapkin and Gibbs, 1963).

Fig. 7 shows that higher efficiencies can be reached by using polyethylene vials for the measurement of <sup>55</sup>Fe. This is critical for environmental samples because counting efficiency for <sup>55</sup>Fe is usually low due to its low energy emissions. This low counting efficiency is an important limitation to LLD, thus the relevance of achieving higher efficiencies.

It is also important to note that quadratic fittings obtained with polyethylene vials are most often better because lower curvatures are achieved, which contributes positively to more precise calibrations, yielding lower errors when interpolating efficiency. In Fig. 8, a LLD

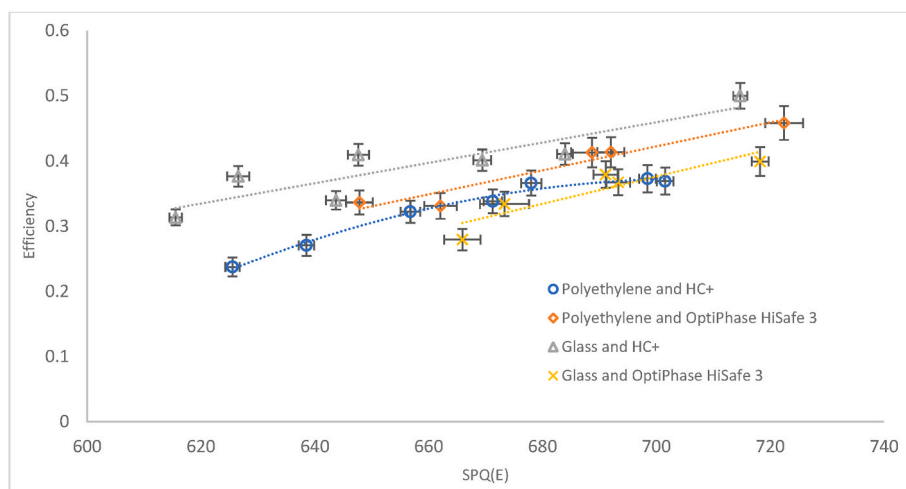


Fig. 9. Comparison of efficiencies for all <sup>63</sup>Ni combinations. All fits are quadratic except for glass and HC+, which is linear.

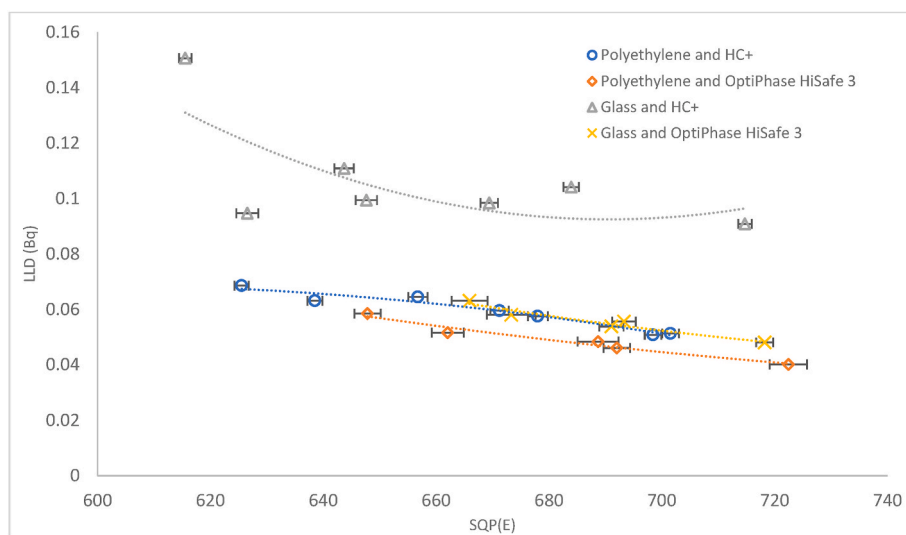


Fig. 10. Comparison of LLDs for all <sup>63</sup>Ni combinations and their quadratic calibrations.

calibration is shown. It can be seen that LLD is lower for polyethylene vials, and particularly while using the scintillator PerkinElmer OptiPhase HiSafe 3 even though this scintillator does not benefit particularly from FOM optimization.

As for <sup>63</sup>Ni results, all the differences between the two vials and scintillators are lower, probably due to higher energy electron emissions. In Fig. 8, glass vials may appear useful as they yield a very high efficiency, but the dispersion affects the fitting negatively, reaching a R<sup>2</sup> of 0.8044, which is not tolerable for a calibration.

In Figs. 9 and 10 we obtained a similar behaviour to that shown in Fig. 7, polyethylene vials are more convenient for achieving lower LLDs and better calibrations. On the other hand, the difference between both scintillators using polyethylene vials is almost negligible.

It is important to remark that all LLDs shown in this section are expected to be lower in the case of measuring real samples, as measuring times will be much greater than 15 min.

### 3.3. Response to quenching agents

We noticed that both scintillators were not affected equally by our quenching agents. This implies that their behaviour when exposed to quenching may be studied in order to improve this optimization. A

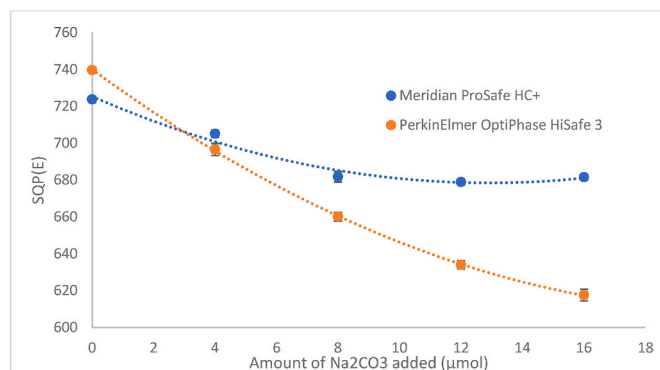


Fig. 11. Dependence of quenching on the amount of quenching agent for <sup>55</sup>Fe standards in polyethylene vials. Both calibration curves are quadratic.

scintillator less affected by quenching is desirable, as a greater SQP(E) may be reached for a certain sample, improving counting efficiency and LLD.

Fig. 11 shows the effect on the SQP(E) of adding different amounts of Na<sub>2</sub>CrO<sub>3</sub> to produce colour quenching in <sup>55</sup>Fe polyethylene vials for the

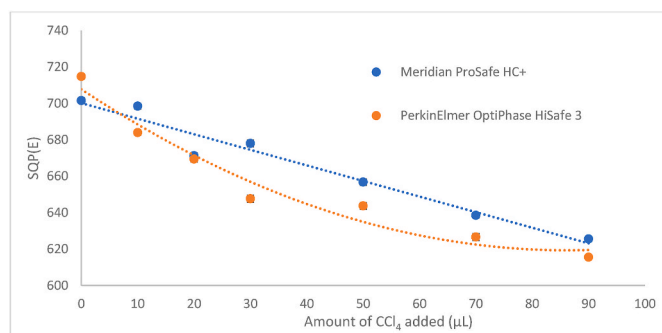


Fig. 12. Dependence of quenching on the amount of quenching agent for  $^{63}\text{Ni}$  standards in polyethylene vials. The Meridian ProSafe HC + curve is linear, while the PerkinElmer OptiPhase HiSafe 3 is quadratic.

two scintillators. It can be seen that Meridian ProSafe HC+ is less affected by colour quenching, making it more appropriate for the measurement of real environmental samples, especially in the case of  $^{55}\text{Fe}$  samples, which are affected by colour quenching.

Fig. 12 has a similar structure to Fig. 10, but it represents the effect of chemical quenching produced with  $\text{CCl}_4$  in  $^{63}\text{Ni}$  polyethylene vials. This time the difference is lower, but Meridian ProSafe HC+ is again less affected by quenching agents for the amounts of quenching most often present in real samples.

#### 4. Conclusions

In this paper, a study has been done on the calibration of a LSC spectrometer for  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  counting in environmental samples. Counting efficiencies have been determined as a function of colour and chemical quenching for two types of scintillators and two types of vials.

The determination of counting efficiency requires a precise method of spectrum integration. Thus, the conventional full-spectrum integration has been compared to a method based on the optimization (maximization) of the Figure of Merit (FOM) of the experiment and the subsequent minimization of LLD. For that, a computer program has been developed.

After comparing the two proposed integration window selection methods, we conclude that FOM-optimization based integration has benefits only in the case of measuring with polyethylene vials and with Meridian ProSafe HC + scintillator. In the other cases, it is better to use the traditional full-continuum integration.

As for the vial and scintillator choice, it is concluded that the use of glass vials should be avoided due to the high background they provide. PerkinElmer OptiPhase HiSafe 3 shows better Lower Limit of Detections (LLD) in general, while Meridian ProSafe HC + has better behaviour with regard quenching as it is less affected by colour or chemical quenching effects. This makes it the best choice for samples that have greater quenching like real environmental samples.

#### Author statement

J.L. García León: Methodology, Software, Validation, Formal

Analysis, Investigation, Data Curation, Writing – Original Draft, Visualization. M. García-León: Investigation, Resources, Writing – Review & Editing, Supervision, Funding Acquisition. G. Manjón: Conceptualization, Methodology, Resources, Investigation, Project Administration, Funding Acquisition.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data will be made available on request.

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