

OPTIMIZATION OF THE CHANNEL RATIO METHOD FOR COLOR QUENCHING CORRECTION FOR MEASUREMENT OF ^{90}Sr VIA CERENKOV COUNTING

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ABSTRACT. This paper presents a method for the determining ^{90}Sr via its β -emitting daughter, ^{90}Y , in environmental samples by Cerenkov counting. The method is based on the isolation of ^{90}Y by solvent extraction, using HDEHP as an extractor. The liquid scintillation (LS) analyzer Tri-Carb 3170 TR/SL has been employed. After radiochemical treatment, environmental samples might be colored, and this will lead to color quenching, which is one of the most important problems that affect Cerenkov counting. The channel ratio (CR) method has been employed to correct this effect. Thus, the A window, which includes the whole spectrum, and the B window, corresponding to the most energetic zone of the A window, are defined. The ratio between the net counts of these windows defines the CR. Most authors working with this method define the B window as starting from the channel with higher number of counts. However, no study justifies this selection. This paper presents measurements and criteria to optimize and justify the choice of the B window. Several "B windows" have been defined, and their respective efficiency versus CR curves has been determined. During a study of the fitting parameters for the curves, we evaluated how to choose the windows to establish the optimum curve that corrects the efficiency decrease due to color quenching. Results have been checked by taking part in an exercise of analytical intercomparison among a great number of Spanish laboratories for the measurement of ^{90}Sr in aqueous samples.

INTRODUCTION

^{90}Sr is an artificial radionuclide, a β emitter, generated by fission or neutronic activation and liberated in explosions or nuclear accidents. ^{90}Sr is interesting from a radioecological point of view for several reasons; on one hand, its relatively long half-life ($T_{1/2} = 28.6$ yr) assures its presence in the biosphere for a long time. On the other hand, due to the chemical similarity of Sr^{+2} - Ca^{+2} , it is possible to introduce ^{90}Sr associated to calcium into the food chain. Once in the organism, ^{90}Sr and calcium are fixed in the materials of the human skeleton. Bjørnstad et al. (1992) estimate that the biological period of semi-elimination of ^{90}Sr is approximately 50 yr; ^{90}Sr is able to provoke damage mostly due to the high energy of the β emission of its descendant ^{90}Y .

The objective of this work is the optimization of a liquid scintillation counter (LSC), model Tri-Carb 3170 TR/SL, for the determination of the concentrations of ^{90}Sr by Cerenkov counting in environmental samples. A radiochemical procedure is developed for the determination of ^{90}Sr through the Cerenkov radiation emitted by its descendant ^{90}Y . Due to their respective half-lives (28.6 yr for ^{90}Sr and 64.1 hr for ^{90}Y), we can assume that both radionuclides would be in secular equilibrium in the environment. ^{90}Y is preferred for Cerenkov measurement of ^{90}Sr due to its higher maximum β energy (2283 keV), which is superior to the Cerenkov threshold in pure water (263 keV).

The method used in this work is based on the one developed by Soumela et al. (1993). The main advantages of this technique are the simplicity of the chemical method and the rapidity of the measurements. On the other hand, to obtain an accurate result based on a Cerenkov measurement, the experimental work must be very rigorous because Cerenkov counting efficiency is very sensitive to color quenching (Fujii and Takiue 1988). There are 2 different kinds of quenching, chemical and color. Chemical quenching is caused by chemical substances that interfere with the energy transference from the solvent to the solute (Brooks 1979). Any absorbing material that colors the sample and reduces the number of photons transmitted through the scintillation medium causes color

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quenching (Baillie 1960). In both cases, the number of photons detected by the photomultiplier is lower than the one detected for unquenched samples, and this causes a reduction in the counting efficiency (Brooks 1979).

Environmental samples, after applying the radiochemical procedure, might be affected by color quenching; for this reason it is necessary to evaluate the quenching degree and correct its effects. To evaluate the color quenching when doing Cerenkov counting, it is not possible to use a gamma external source as in conventional liquid scintillation counting (LSC), and it is necessary to use alternative methods such as the channel ratio method.

In this work, we study the calibration curves for the decreased counting efficiency due to color quenching. The color quenching degree was calculated following the channel ratio method, which was obtained from the different windows of the spectrum. Furthermore, results from an intercomparison exercise for ^{90}Sr measurements are presented.

RADIOCHEMICAL PROCEDURE AND DETECTION

The Tri-Carb 3170 TR/SL is a liquid scintillation (LS) counter specially designed for detection of low-level alpha and beta radioactivity. Measurements were done using the TR-LSCTM (Perkin-Elmer, USA) device, which is the optimum counting mode for very low activity samples.

The method used in this work for separation and isolation of ^{90}Y has its origin in the one developed by Soumela et al. (1993). It is based on the property of yttrium that always behaves with valence 3+, forming stable complexes with diverse organic extractors. One of these extractors is the HDEHP, *bis* (2-ethylehexyl) hydrogen phosphate acid, employed to separate ^{90}Y from ^{90}Sr . Another extractor is the EDTA (ethylenediaminetetraacetic acid) used to obtain the chemical recovery of the process. The complete radiochemical procedure is shown in Figure 1.

CALIBRATION OF THE MEASUREMENT

Study of the Background

Proper control of certain parameters in the Cerenkov counting, such as the type of vial or the final volume of the samples in the vial, might reduce the background considerably (Vaca et al. 1998). For this reason, we have studied the dependence of the background on certain parameters and their influence on the counting efficiency, minimum detectable activity, and color quenching.

It is well known that polyethylene vials produce less Cerenkov background than glass vials, due to the presence of ^{40}K in the walls of the glass vials (Rapkin and Gibbs 1963); thus, we will only use polyethylene vials for our measurements. To study the influence of the final volume in the vial, 1 mL of HNO_3 70% was prepared, and increasing volumes of distilled water, from 0 to 22 mL, were added. The counting time was 10 hr, and the detector was always configured in low-level counting. Results are presented in Table 1. It can be observed that the background does not change significantly when the volume changes; the average value is 1.45 ± 0.19 cpm. Furthermore, the background was measured periodically and systematically for 2 yr; a relative stability can be observed, and the average value is 1.6 cpm for a volume of 20 mL.

Efficiency of ^{90}Sr - ^{90}Y , LLD, and FM

Given that the counting efficiency might depend on the final volume present in the sample, we have not only calculated the counting efficiency, but also optimized the final counting volume. To obtain

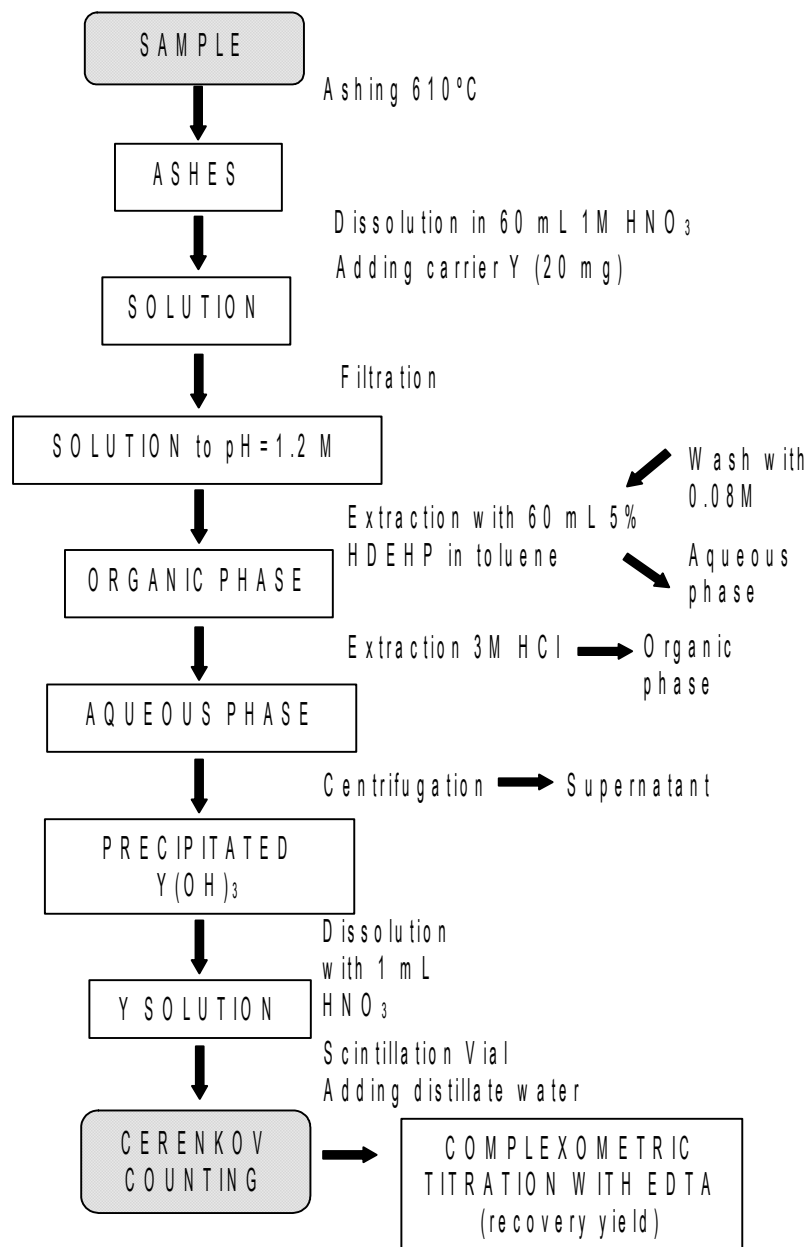


Figure 1 Radiochemical method for isolation and determination of ^{90}Y

these counting efficiencies, some samples have been traced with known amounts of ^{90}Sr - ^{90}Y . Eleven polyethylene vials were prepared with 1 mL of HNO_3 and 0.5 mL of tracer solution. In addition, different volumes of distilled water were added to the vials and measured using the Tri-Carb low-level counting program. The counting time was 600 min.

Table 1 Background, counting efficiency, lower limit of detection (LLD), and figure of merit (FM) for ^{90}Sr - ^{90}Y , depending upon the volume of the sample.

V (mL)	B (cpm)	ϵ	LLD (mBq)	FM (cpm $^{-1}$)
2	1.43 \pm 0.03	0.512 \pm 0.002	7.54	0.183 \pm 0.003
4	1.50 \pm 0.03	0.507 \pm 0.002	7.81	0.171 \pm 0.003
6	1.42 \pm 0.03	0.530 \pm 0.002	7.25	0.198 \pm 0.004
8	1.33 \pm 0.02	0.528 \pm 0.002	7.06	0.209 \pm 0.004
10	1.29 \pm 0.02	0.536 \pm 0.002	6.84	0.223 \pm 0.004
12	1.39 \pm 0.03	0.531 \pm 0.002	7.16	0.203 \pm 0.004
14	1.42 \pm 0.03	0.531 \pm 0.002	7.25	0.198 \pm 0.004
16	1.44 \pm 0.03	0.535 \pm 0.002	7.24	0.198 \pm 0.004
18	1.49 \pm 0.03	0.520 \pm 0.002	7.57	0.181 \pm 0.003
20	1.59 \pm 0.01	0.529 \pm 0.002	7.68	0.176 \pm 0.001
22	1.61 \pm 0.02	0.506 \pm 0.002	8.07	0.159 \pm 0.002

The counting efficiency has been obtained as follows:

$$\epsilon = \frac{n_T - b}{M_{(^{90}\text{Sr}-^{90}\text{Y})} \cdot A_{(^{90}\text{Sr}-^{90}\text{Y})}} \quad (1)$$

where n_T is the total counting rate; b is the background counting rate; $M_{(^{90}\text{Sr}-^{90}\text{Y})}$ and $A_{(^{90}\text{Sr}-^{90}\text{Y})}$ are, respectively, the mass (g) and the activity (Bq/g) of the added tracer solution.

We have used the figure of merit (FM) that, according to Schönhofer et al. (1991), is defined as

$$FOM = \epsilon^2 / B \quad (2)$$

and the lower limit of detection (LLD), defined according to Currie (1968):

$$LLD = \frac{2.71 + 4.65 \cdot (B)^{1/2}}{\epsilon \cdot \tau} \quad (3)$$

where B is the total background counts; τ is the background counting time, which, in this case, is the same as the sample counting time; and ϵ is the counting efficiency.

The efficiency uncertainty is $\sim 0.4\%$. The uncertainty of the standard solution is 0.25% , 160.8 ± 0.4 Bq/mL; uncertainties in the volume of the solution have been neglected. The results are shown in Table 1.

Usually, the counting efficiency chosen is the one that minimizes the lower limit of detection (LLD) or maximizes the figure of merit (FM). Taking into account these criteria, a final volume of 10 mL would be suitable to perform the measurements. Nevertheless, the differences between the different volumes are minimal, and thus the dependence of efficiency and background upon the final counting volume does not turn out to be a decisive factor.

For this reason, other factors must be taken into account in order to choose the suitable volume. In this case, color quenching has a major importance in the measurement of samples through Cerenkov radiation (Vaca 1998). The color quenching of the sample will be minimized if we dilute the sample in the vial. For this reason, we decided to measure all the samples with a final volume of 20 mL. From Table 1, for polyethylene vials and a final volume of 20 mL, we see that the counting efficiency for ^{90}Sr , in equilibrium with ^{90}Y , is 53% .

Efficiency of ⁹⁰Y

The procedure we use in this work for ⁹⁰Sr determination is based on measuring the concentration of its descendant, ⁹⁰Y. Consequently, it was necessary to determine the counting efficiency of the isolated ⁹⁰Y. For that purpose, 10 aliquots were traced with ⁹⁰Sr-⁹⁰Y solution of known specific activity. The usual chemical method was then applied to the traced samples. Once ⁹⁰Sr was separated from ⁹⁰Y, it was measured in polyethylene vials, completing the volume with distilled water up to 20 mL. Efficiency is obtained by the following:

$$\epsilon = \frac{\ln 2(N_t - N_B)e^{\frac{t \ln 2}{T}}}{V \cdot A \cdot T \cdot R_Q \left(l - e^{\frac{-\tau \ln 2}{T}} \right)} \quad (4)$$

where N_t is the total counts, N_B the background counts, τ the counting time (s), t the elapsed time (s) from the separation of ⁹⁰Sr-⁹⁰Y to the beginning of the measurement, V is the volume (mL) of the tracer dissolution added, A is the activity (Bq/mL) of the added ⁹⁰Sr, T is the ⁹⁰Y half-life (s), and R_Q is the chemical recovery. We have taken into account that the measurement time of the sample is of the same order of magnitude as the ⁹⁰Y half-life and also that some time has elapsed from the ⁹⁰Sr-⁹⁰Y separation to the measurement of ⁹⁰Y.

Results are shown in Table 2. The chemical recovery values obtained were relatively homogeneous, ~85%. From those data we have calculated the average Cerenkov counting efficiency, ϵ_0 , obtained for ⁹⁰Y, and the result was 51%. This value is assumed to be the counting efficiency for the samples not affected by color quenching. In the next section, we will evaluate the color quenching degree and correct its effects.

Table 2 Radiochemical yields and efficiency calculations for ⁹⁰Y standards. Average values and standard deviations are included.

Sample	τ (s)	B (cpm)	R_Q (%)	σ (R_Q)	ϵ_i	σ (ϵ_i)	$\epsilon_0 \pm \sigma$ (ϵ_0)
Std-1	36,000	1.59 ± 0.01	84	2	0.502	0.013	0.51 ± 0.02
Std-1			84	2	0.502	0.013	
Std-2			82	2	0.521	0.014	
Std-2			82	2	0.516	0.014	
Std-3			88	2	0.505	0.013	
Std-3			88	2	0.502	0.013	
Std-4			78	2	0.551	0.015	
Std-4			78	2	0.547	0.015	
Std-5			77	2	0.538	0.014	
Std-5			77	2	0.535	0.014	
Std-6			87	2	0.483	0.013	
Std-6			87	2	0.481	0.013	
Std-7			79	2	0.483	0.013	
Std-7			79	2	0.481	0.013	
Std-9			89	2	0.498	0.013	
Std-9			89	2	0.496	0.013	
Std-10			80	2	0.534	0.014	
Std-10			80	2	0.531	0.014	

Color Quenching

We used the channel ratio method to evaluate and correct the effects of the color quenching degree. This method is based on analyzing the shift of the spectra to lower channels when color quenching increases in the sample. To study the relationship between the shift of the spectrum and the efficiency, some standard samples were prepared. Different degrees of color quenching were achieved by adding increasing amounts of Na_2CrO_4 , the color quenching agent (Villa et al. 2003).

For the study, 12 polyethylene vials were prepared with 2 mL of carrying dissolution of stable yttrium, Y_2O_3 (for the radiochemical yield), and tracer dissolution of ^{90}Sr - ^{90}Y . ^{90}Y was isolated by applying the chemical procedure described and was then transferred to the vial. Increasing quantities of Na_2CrO_4 (from 10 to 1100 $\mu\text{mol Na}_2\text{CrO}_4$) and distilled water were added to every vial.

^{90}Y is measured by Cerenkov counting, and from the obtained spectra, 2 different windows are defined, A and B. The A window includes the totality of the spectrum, whereas the B window is placed in the higher energies of the spectrum. From these windows, we obtained the channel ratio (CR) factor:

$$CR = \frac{n_B}{n_A} \quad (5)$$

where n_A = net counting rate in the A window, and n_B = net counting rate in the B window.

In the Tri-Carb 3170, ^{90}Y Cerenkov spectrum is placed between the channels 0 and 47, so that the A window will include channels 0–47. For the B window, all the authors consulted in the literature define the second window as dependent upon the number of counts and try to find the one that presents most of the counts, but with no defined criterion (Fujii and Takiue 1988; Vaca et al. 1998; Villa et al. 2003). One of the aims of this work has been to choose the range of the B window while trying to justify its suitability. Thus, 7 B windows have been defined (B1, B2..., B7), from the interval of channels 4–47 (B1) to the interval 10–47 (B7) (Figure 2).

The channel ratio (CR) factor has been calculated for all the standards previously prepared. For every sample, CR has been obtained for the 7 windows, and CR will be related to the counting efficiency. To evaluate the decrease in the efficiency, a new parameter has been introduced in this work, the color factor (f). As is shown in Equation 6, using this parameter, the efficiency of a quenched sample is related to the reference efficiency (ϵ_0), which is the efficiency of a sample not affected by color quenching,

$$\left(f = \frac{\epsilon}{\epsilon_0}\right) \rightarrow (\epsilon = \epsilon_0 \cdot f) \quad (6)$$

We represent f versus CR; for every quenching degree, 7 curves have been obtained depending upon which B window was chosen.

In Figure 3, 7 curves of f versus CR have been represented, each of them dependent upon a different B window. All the curves have been fitted to a 2nd-order polynomial, using 25 points; every color standard was measured twice (24 points), and the last point corresponds to the average of the colorless standards.

In the graphics for f calibration, a study of several parameters has been made in order to choose the most suitable curve to correct counting efficiency, that is, in order to find the optimum B window.

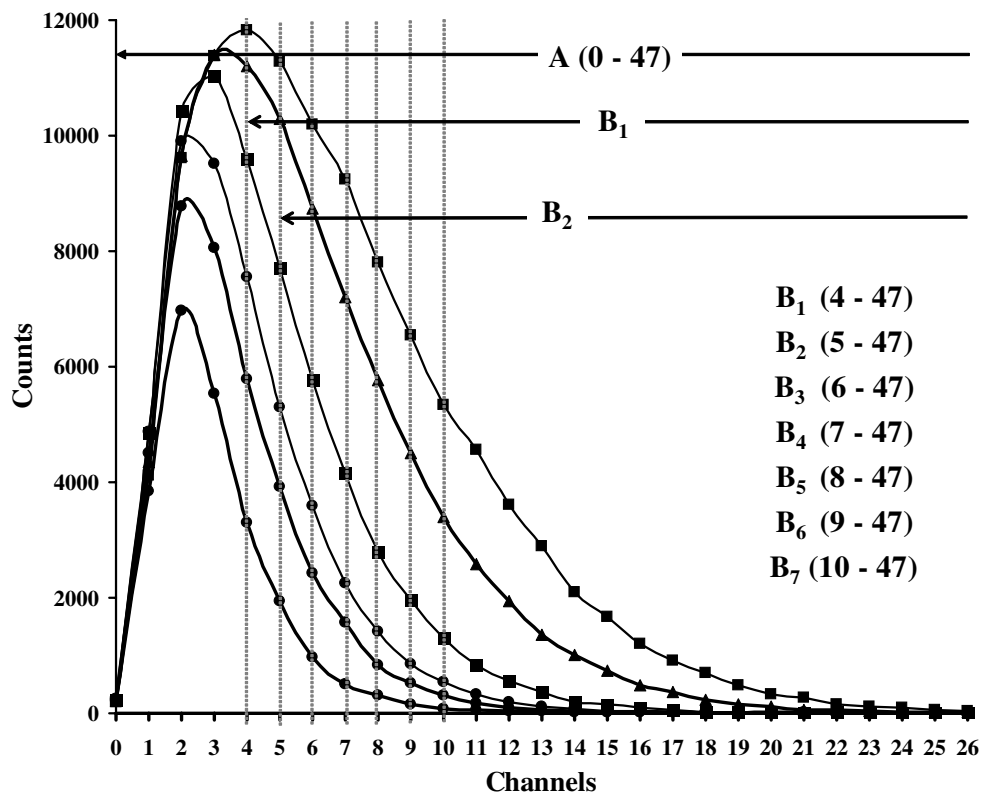


Figure 2 Scheme of a Cerenkov spectrum and description of the A window and B windows (B₁, B₂..., B₇)

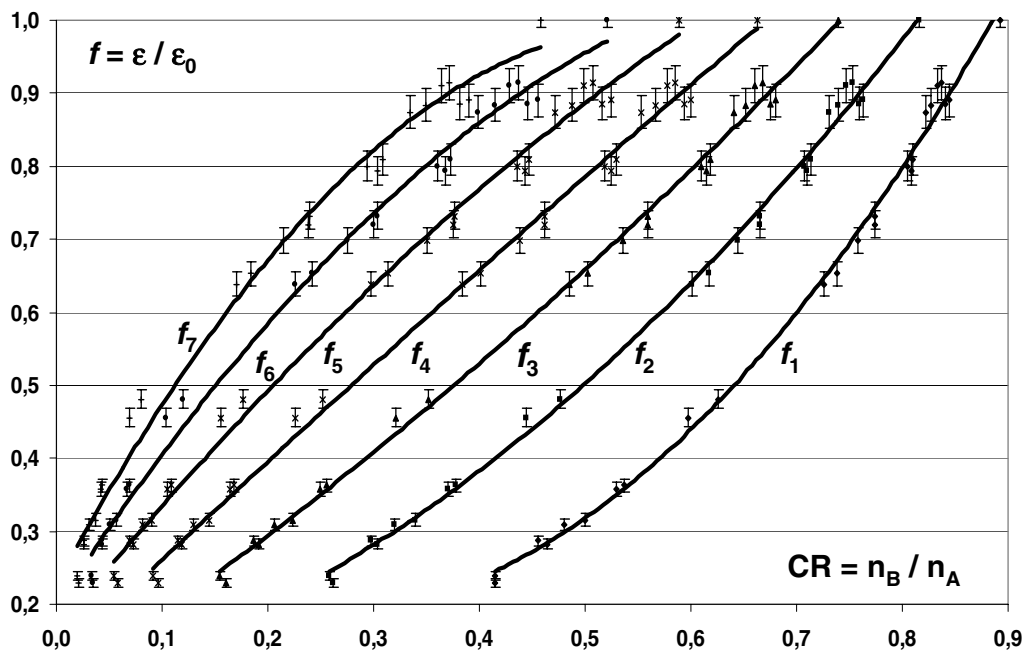


Figure 3 Calibration curves f versus CR for the different B windows

These parameters were chi-squared reduced, $\chi^2 R$ (Bevington and Robinson 1992); the coefficient of regression, R ; and the range of the CR values and efficiencies that are valid for every curve,

$$\chi_R^2 = \frac{1}{n - n_{param}} \cdot \sum_{i=1}^n \frac{(Y_i - y_i)^2}{\sigma(y_i)^2} \quad (7)$$

where n is the number of experimental data used in the adjustment (in this case, $n = 25$), n_{param} is the number of parameters of the adjustment, Y_i are the predicted values for the function of the second order fitted curve, y_i are the experimental values, and $\sigma(y_i)$ is the uncertainty associated with every experimental value.

The range of acceptance of the fitted curve is defined as a range of CR and, consequently, a range of efficiencies. To calculate the range, it is first necessary to obtain the fitting curve; then, we will only accept the range of CR that corresponds to color factors of f that, when applied to real samples, have an associated uncertainty under 20%. Otherwise, if the uncertainty is over 20%, the activities values would be too high to consider. As an example, in Figure 4, the range of CR that will be accepted in the window B_1 can be observed.

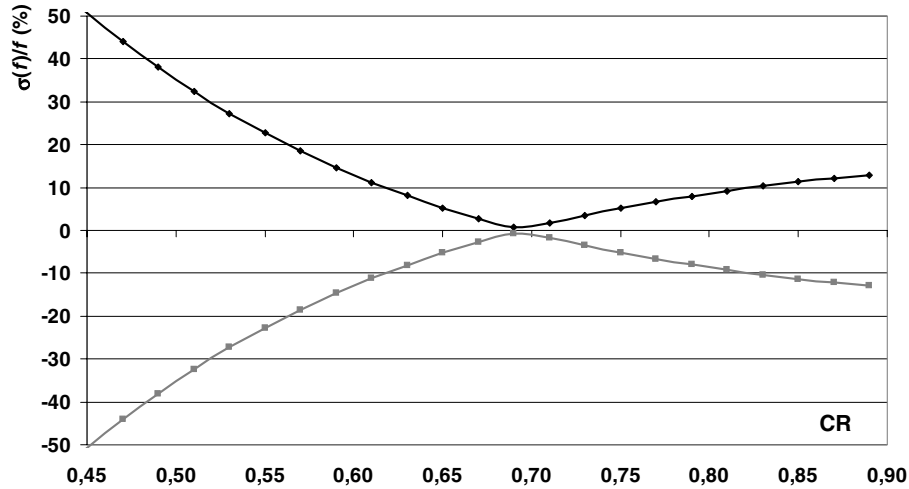


Figure 4 Range of acceptance of CR for the window B_1

From the results in Table 3, we can see the curves that give better results in relation to parameters f_2 and f_3 . In relation to the coefficient of regression, R , all the values are very similar, and it is not possible to obtain any definitive result from them. However, in relation to the chi-square, χ^2_R , values present, significant differences depending on the curve. The closest data to the tabulated data for a probability of distribution of 50% and 22 degrees of freedom ($\chi^2_R = 0.970$) (Bevington and Robinson 1992) is 1.24 from curve 2. However, if we take into account all the parameters presented in the table, we can see that for curve 3, χ^2_R is also close to the tabulated value; the experimental uncertainty value, $\sigma(y_i)$, is the smallest, and the accepted ranges for CR and efficiencies are quite wide. For this reason, curve 3 will be considered the optimum fitting curve, and the B_3 window (from 6 to 47 channels) will be the one that we will use to perform the color corrections.

Table 3 Equations for the fittings f versus CR for different B windows, with their respective fitting parameters.

	$f = c \cdot CR^2 + b \cdot CR + a$	R	χ_R^2	$CR_{MAX} \leftrightarrow CR_{MIN}$	$\epsilon_{MAX} \leftrightarrow \epsilon_{MIN}$
f_1 (CR = n_{B1}/n_A)	$c = 1.95 \pm 0.23$ $b = -0.93 \pm 0.30$ $a = 0.297 \pm 0.092$	0.9978	1.49	$0.89 \leftrightarrow 0.57$	$52 \leftrightarrow 20$
f_2 (CR = n_{B2}/n_A)	$c = 0.93 \pm 0.15$ $b = 0.36 \pm 0.16$ $a = 0.089 \pm 0.037$	0.9982	1.24	$0.82 \leftrightarrow 0.41$	$51 \leftrightarrow 20$
f_3 (CR = n_{B3}/n_A)	$c = 0.37 \pm 0.13$ $b = 0.96 \pm 0.11$ $a = 0.088 \pm 0.020$	0.9984	1.37	$0.74 \leftrightarrow 0.15$	$51 \leftrightarrow 12$
f_4 (CR = n_{B4}/n_A)	$c = -0.10 \pm 0.14$ $b = 1.37 \pm 0.10$ $a = 0.125 \pm 0.014$	0.9981	1.95	$0.66 \leftrightarrow 0.10$	$51 \leftrightarrow 13$
f_5 (CR = n_{B5}/n_A)	$c = -0.66 \pm 0.18$ $b = 1.78 \pm 0.11$ $a = 0.163 \pm 0.012$	0.9975	3.34	$0.59 \leftrightarrow 0.05$	$50 \leftrightarrow 13$
f_6 (CR = n_{B6}/n_A)	$c = -1.40 \pm 0.26$ $b = 2.21 \pm 0.13$ $a = 0.198 \pm 0.012$	0.9964	5.68	$0.52 \leftrightarrow 0.03$	$50 \leftrightarrow 13$
f_7 (CR = n_{B7}/n_A)	$c = -2.38 \pm 0.38$ $b = 2.70 \pm 0.16$ $a = 0.227 \pm 0.012$	0.9946	9.54	$0.46 \leftrightarrow 0.02$	$49 \leftrightarrow 14$

VALIDATION OF THE METHODOLOGY

To validate the method, 4 samples of eucalyptus bark, which were already measured by an alternative method, were chosen (^{90}Sr activity concentration was obtained following the method described) and compared to the results obtained in 1998 from a different detector, Quantulus 1220TM, from the validated method from Vaca et al. (1998).

The results are shown in the Figure 5. In order to obtain the results following our method, we have used the curves f_2 and f_3 . We have represented the ratio between the concentrations obtained following our method and the concentrations obtained in 1998. The results are very close, indicating that the method gives accurate results. We also observe that the 2 results, using window 2 and window 3 to correct color quenching, do not differ significantly from each other, as the results are the same within the error (σ).

Moreover, we calculated and compared the concentrations of 4 samples, where the counting efficiency is corrected following every curve f . Results are in Figure 6. If we compare the results obtained using window B_1 (curve f_1), the window that is usually found in the literature, and the results using the optimum window B_3 (curve f_3), we observe that the uncertainties using window B_3 are smaller than the ones obtained from window B_1 .

Finally, our laboratory took part in an intercomparison program among laboratories of environmental radioactivity (2004) organized by the CSN (Nuclear Security Spanish Council) for the measurement of ^{90}Sr in water samples. In this exercise, the activity concentration obtained by our laboratory was $195 \pm 14 \text{ Bq/m}^3$, using the curve f_3 to correct the counting efficiency. The mean value from all the laboratories participants is $195 \pm 29 \text{ Bq/m}^3$, and the reference value given is $187 \pm 37 \text{ Bq/m}^3$. Our results are equivalent within the uncertainty, which ensures the accuracy of our method.

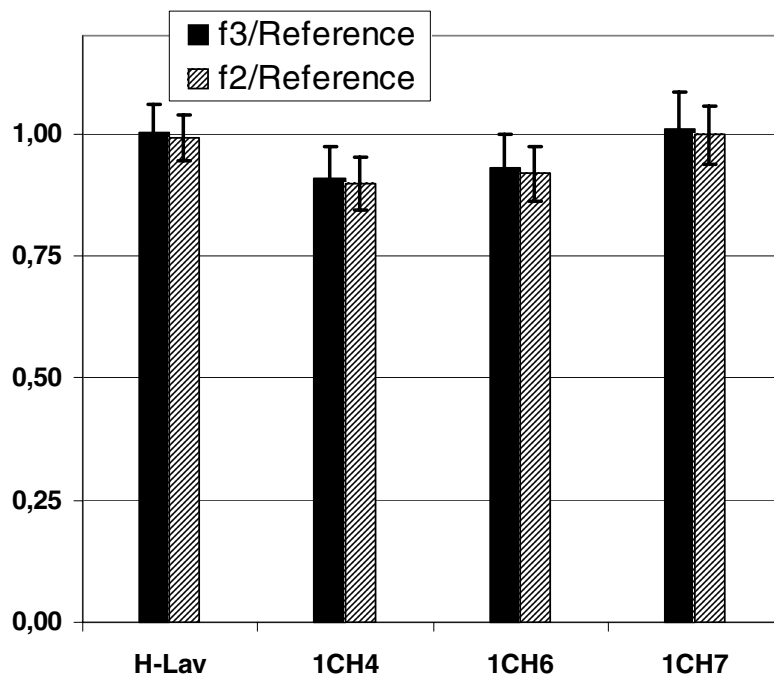


Figure 5 Comparison of activities determined in 1998 with the activities measured following our method. The uncertainty corresponds to 1σ .

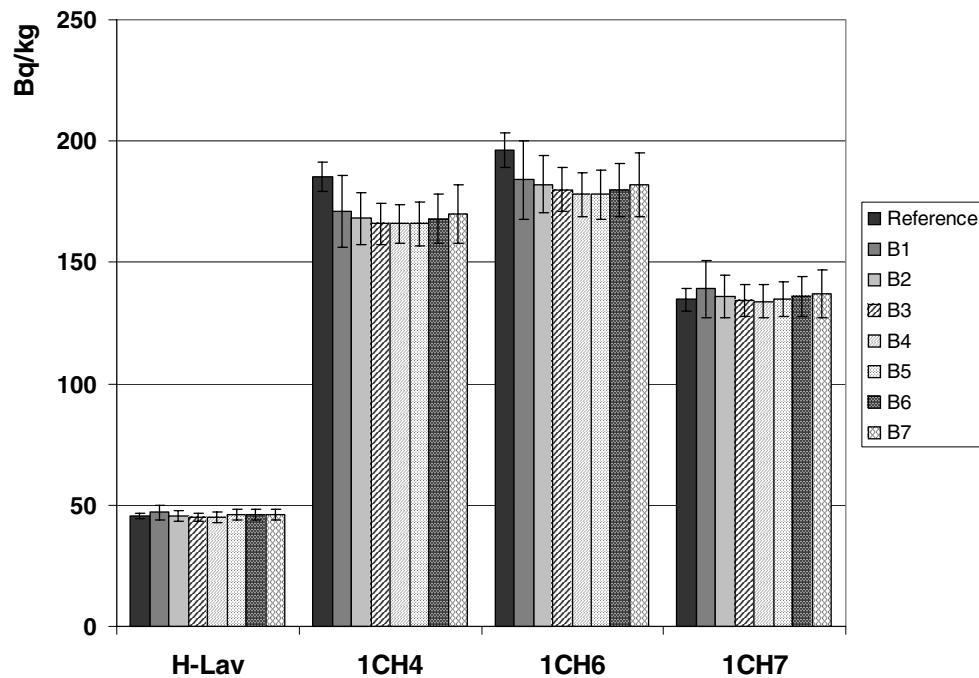


Figure 6 Comparison of concentrations of 4 samples, correcting the counting efficiency depending on every curve f

CONCLUSIONS

The detector parameters of Tri-Carb 3170 have been optimized for the measurement of ^{90}Sr through the determination of ^{90}Y by Cerenkov counting. A study of the background and counting efficiency using the Tri-Carb has been also included. Several studies to optimize the evaluation of color quenching via the channel ratio method have been done in order to fit the windows of measurement that give the best results. Finally, the procedure has been validated with an intercomparison exercise and the measurement of samples with known activity.

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