

**Ciemat** 675

Sp ISSN 614-087-X

**SPECTRAL INTERPOLATION AND UNFOLDING TO  
MEASURE MULTI-LABELLED SAMPLES BY LIQUID  
SCINTILLATION**

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**CENTRO DE INVESTIGACIONES  
ENERGETICAS, MEDIOAMBIENTALES Y TECNOLOGICAS**

**MADRID, 1990**

CLASIFICACION DOE Y DESCRIPTORES

440103

INTERPOLATION

SPECTRA UNFOLDING

LABELLED COMPOUNDS

SCINTILLATION COUNTING

LIQUID SCINTILLATORS

RADIOISOTOPES

EMISSION SPECTRA

BETA MINUS DECAY

BETA PLUS DECAY

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Este trabajo se ha recibido para su impresión en Abril de 1.991

Depósito Legal nº M-16117-1991  
ISBN 84-7834-104-8  
ISSN 614-087-x  
NIPO 238-91-002-3

IMPRIME CIEMAT



## CONTENTS

1. Introduction	1
2. Statement of the problem	2
3. Smoothing	2
4. Spectral function	3
5. Spectral interpolation	5
6. Characteristic spectral parameters that depend on quench	8
7. Spectral fitting	8
8. Spectral shift	10
9. Activity determination	11
10. References	12



## 1. Introduction

In tracer studies, it is of interest to use two or more radionuclides in the same experiment in order to obtain different functions of a system at the same time.

The main difficulties we find in these types of measurements are the continuous nature of the beta-ray spectra, the low resolution of the liquid scintillation spectrometer and the variation of the counting efficiency due to the presence of chemical quenching.

At present four procedures are generally applied to obtain the activity of each radionuclide in a double or triple labelled sample. These procedures are the following: double or triple window counting [1], counting rate at different times [2], application of different counting techniques [3] and different decay modes [4].

The method of using a double or triple window is the most general and the most frequently applied. Nevertheless, it has important limitations. As a consequence of the low spectral resolution of the scintillation system, the measurement of radionuclides with close energies is not possible, and in the case of very different energies the determination of the activity has large uncertainties when the quench is high. The other procedures referred to, are applied only in very special cases and their use is quite limited.

All the above procedures are based on counting portions of the pulse height spectra by pulse height discrimination. Because of the difficulties and limitations of the available methods, it is of interest to explore new methods based on the shape of the pulse height spectrum.

The aim of this paper is to develop a general method based on the spectral shape fitting in order to obtain the activity of each of the radionuclides in a mixture.

We will consider logarithmic spectra obtained for a constant gain and endowed with an external standard gamma-ray source for the determination of the quench parameter in scintillation samples.

## 2. Statement of the problem

The large difference in shape between the calculated beta-ray spectra and the observed pulse height spectra has limited liquid scintillation spectrometry. These differences are due to the low energy resolution of the liquid scintillation spectrometers, the existence of a finite probability for non-detection and the effect of chemical quench in the scintillation sample.

The chemical quench produces a deformation of the spectrum and a reduction in the counting rate. In general, when the quench value is reduced the pulse height decreases and therefore the spectrum shifts to lower height pulses. Fig.1 shows three spectra of the same nuclide with different value of the quench parameter. It is not possible to obtain the spectrum with a fixed quench value using ordinary interpolation methods. It may be possible to make a channel by channel interpolation only in channels between zero and the channel that corresponds to the maximum in the most quenched spectrum. The interpolation for values beyond the maximum will be inaccurate if not impossible.

A new procedure based on a dilatation, an interpolation and a contraction solves this interpolation problem. This, followed by the application of a least squares fitting method provides the activity of each one of the components of the sample.

Let us consider a mixture of  $n$  radionuclides: A,B,C... Our final objective is to find out the activity of each one of the components. For this, the following experimental data are required:

i) The spectrum of the mixture of radionuclides and the quench value  $Q_p$ , obtained by one of the external standard procedures: channel ratio,  $\#H$  number, spectral index (SIE), spectral quench parameter (SQP(E)) or transformed spectral index (tSIE).

ii) A set of spectra for each of the radionuclides of the sample. This set of spectra is obtained from a group of samples where the activity and quench value are well known. The quench parameter is denoted by  ${}^1 Q_1, {}^1 Q_2, \dots, {}^1 Q_i, \dots, {}^1 Q_n$  where  ${}^1 Q_1 > {}^1 Q_2 > \dots > {}^1 Q_{i-1} > {}^1 Q_p > {}^1 Q_i > \dots > {}^1 Q_n$  and where  $l=1,2,\dots,n$  are each one of the nuclides of the sample. The counts in a channel are denoted by  ${}^l y_{ij}$ , where  $j$  is the channel number and  $i$  is the spectrum number in the  $l$ -nuclide set of spectra.

## 3. Smoothing

The spectra obtained from a liquid scintillator spectrometer present statistical fluctuations because of the decay processes, light



detection height pulse analysis and gain variation of the photomultiplier and electronics.

It is useful to decrease these statistical fluctuations. The advantages of smoothing the spectra will be seen in the following section. The spectra shown in fig.1 do not have strong statistical fluctuations, thus they can be smoothed simply by using the expression:

$$y_{ij} = \frac{1}{4}y_{i,j-1} + \frac{1}{2}y_{ij} + \frac{1}{4}y_{i,j+1} \quad (1)$$

This equation is based on averaging over adjacent channels with a binomial distribution spanning three channels.

It is well known that this smoothing conserves the area of the spectrum. This area conservation is not important however for the method to be developed. We will centre our attention on the shape of the spectra.

The histograms obtained by application of this kind of smoothing to the experimental spectra of fig.1, are plotted in fig.2.

#### 4. Spectral function

The experimental spectra are pulse height distributions and have the inconvenience of being defined for discrete values of the channel spectra. The main difficulty for this kind of distribution is their mathematical manipulation. As we will see below, it is essential to have continuous mathematical functions defining the spectra to carry through the spectral interpolation. We call these functions spectral functions.

A simple way to obtain spectral functions is by fitting Fourier series to the experimental spectra. The spectral function  $f(\omega)$  is then obtained from:

$$f(\omega) = \begin{cases} a+b\omega + \sum_{k=1}^n c_k \sin \frac{k\pi\omega}{m-1} & 0 \leq \omega \leq \omega^* \\ 0 & \omega > \omega^* \end{cases} \quad (2)$$

where  $m$  is the number of experimental points and  $n$  is the number of Fourier harmonics. We have only considered the sine terms of the Fourier series due to the shape of the experimental spectra. The term  $a+b\omega$  has been added in order to avoid possible negative fitted values near  $\omega=0$ .

The first and last values of the spectra are  $\omega=0$  and  $\omega=\omega^*=m$ .

The constants  $a, b$  and  $c_k$  can be obtained from the following equations:

$$a = y_0 \tag{3}$$

$$b = \frac{y_{n-1} - y_0}{m-1} \tag{4}$$

$$c_k = \frac{2}{m-1} \sum_{j=0}^{m-1} y_j \sin \frac{\pi k \omega_j}{m-1} \tag{5}$$

where  $y_j$  is the number of counts in channel  $j$ , and

$$\bar{y}_j = y_j - (a + b\omega_j) \tag{6}$$

$$\omega_j = j = 0, 1, 2, \dots, m-1$$

The number of Fourier harmonics to be taken is not evident. When the number of terms is the same as the number of experimental data, the spectral function  $f(\omega)$  follows all the experimental points of the spectrum and consequently the statistical fluctuations are included. Thus, it seems advantageous to take a smaller number of terms in order to smooth the shape of the spectrum as much as possible. However, the spectral function  $f(\omega)$  can lose essential structures of the experimental spectrum if we take a very low number of harmonics. In our case the harmonic number is  $1/3$  of the experimental points. The results are shown in fig.3.

## 5. Spectral interpolation

The pulse spectra of the standard sample set for each radionuclide allows one to compute the corresponding spectral functions  ${}^1f_i(\omega)$ , where  $i$  denotes the spectrum and  $1$  the radionuclide.

The aim is to obtain the spectral functions  ${}^1f_p(\omega)$  which represent the computed spectra for the different radionuclides in the mixture when the quench parameter is  $Q=Q_p$ .

The idea is to shift all the functions  ${}^1f_i(\omega)$ , for  $i=2,3,\dots,n$ , in order to superimpose them on the function  ${}^1f_1(\omega)$ . The problem is to define the required mathematical transformations to obtain this superposition.

Three characteristic points are considered for the first spectral function  ${}^1f_1(\omega)$ , which corresponds to the least quenched sample. These points are: the zero channel of the spectrum, the channel of the maximum and the endpoint of the spectrum. The inflexion at the final region of the spectrum can be used instead of the endpoint of the spectrum.

The mathematical transformations oblige the other spectral functions to pass through these three points.

Let  ${}^1\omega_{i\alpha}$  be the channel number that corresponds to the maximum,  ${}^1\omega_{i\beta}$  the channel of the endpoint, and  ${}^1\omega_{i\gamma}$  the inflexion after the maximum of the spectral functions  ${}^1f_i(\omega)$ .

It is useful to divide the functions  ${}^1f_i(\omega)$  into two regions:

$${}^1f_i(\omega) = \begin{cases} {}^1g_i(\omega) & 0 \leq \omega \leq {}^1\omega_{i\alpha} \\ {}^1h_i(\omega) & {}^1\omega_{i\alpha} \leq \omega \leq {}^1\omega_{i\beta} \end{cases} \quad (7)$$

and discuss each one of these regions separately since the mathematical transformations to be applied are different.

A simple dilatation transformation for the first region of the spectrum is given by:

$$\omega' = a\omega + b \quad (8)$$

where  $a$  and  $b$  are parameters to be determined from the characteristic points of the spectra. The zero point of the spectra make  $b=0$ . The coincidence of the maxima gives:

$$a = \frac{{}^1\omega_{1\alpha}}{{}^1\omega_{i\alpha}}$$

(9)

Consequently, the transformation is :

$$\omega' = \frac{{}^1\omega_{1\alpha}}{{}^1\omega_{i\alpha}} \omega$$

(10)

Whenever this transformation is applied to each of the functions  ${}^1g_i(\omega)$ , they will connect zero and  ${}^1\omega_{i\alpha}$ , furthermore they will be defined in the interval  $(0, {}^1\omega_{i\alpha})$ .

The same dilatation transformation can be applied to the second spectral region. The coincidence conditions between  ${}^1\omega_{i\alpha}$  and  ${}^1\omega_{1\alpha}$ , and between  ${}^1\omega_{i\beta}$  and  ${}^1\omega_{1\beta}$  define the equation:

$$\omega' = \frac{{}^1\omega_{1\alpha} - {}^1\omega_{1\beta}}{{}^1\omega_{i\alpha} - {}^1\omega_{i\beta}} \omega + \frac{{}^1\omega_{i\alpha} {}^1\omega_{1\beta} - {}^1\omega_{1\alpha} {}^1\omega_{i\beta}}{{}^1\omega_{i\alpha} - {}^1\omega_{i\beta}}$$

(11)

Whenever this transformation is applied to each one of the functions  ${}^1h_i(\omega)$ , they will pass through  ${}^1\omega_{1\alpha}$  and  ${}^1\omega_{1\beta}$ , furthermore they will be defined in the interval  $({}^1\omega_{1\alpha}, {}^1\omega_{1\beta})$ .

It is possible to take the inflexion points instead of the points that represent the endpoint of the spectra. Then, it will be required that all the points  ${}^1\omega_{i\gamma}$  coincide with  ${}^1\omega_{1\gamma}$  and the maximum points coincide as well.

The transformation expression will be the same as eq. (11) where  ${}^1\omega_{i\beta}$  have been replaced by  ${}^1\omega_{i\gamma}$ . The spectral functions in fig.3 have been transformed by eq. (10) and (11) into the dilated functions showed in fig.4. In this case the points considered are  ${}^1\omega_{i\alpha}$  and  ${}^1\omega_{i\gamma}$ .

Let  ${}^1f_i^*(\omega)$  be the dilated spectral functions. A set of points  $\{{}^1Q_i, {}^1h_i(\omega)\}$  can be obtained for each channel and for each nuclide. These points can be fitted by least squares fitting in order to determine the number  ${}^1y_{ij}^*$  that corresponds to the quench value  ${}^1Q_p$ . Repeating this process channel by channel the dilated spectrum for a quench value  ${}^1Q_p$  is obtained.

The next step is to calculate the spectral function from the interpolated spectrum. Then a Fourier series will be fitted to the points again.

The new spectral function  $l_{f_p}^*(\omega)$  ( see fig.5) can be separated in to two regions:

$$l_{f_p}^*(\omega) = \begin{cases} l_{g_p}^*(\omega) & 0 \leq \omega \leq l_{\omega_{p\alpha}} \\ l_{h_p}^*(\omega) & l_{\omega_{p\alpha}} \leq \omega \leq l_{\omega_{p\beta}} \end{cases} \quad (12)$$

It is interesting to point out the importance of calculating the spectral function. It is essential whenever a dilatation is applied to have a function since the channel scale has changed. It is also essential to know the values of the maximum and inflexion for the contracted spectrum with quench value  $Q = Q_p$ . These values can be obtained from the curves showed in fig.6. Let  $l_{\omega_{p\alpha}}$ ,  $l_{\omega_{p\beta}}$  and  $l_{\omega_{p\gamma}}$  be these values, the contraction transformation required for the spectral function  $l_{g_p}^*(\omega)$  is:

$$\omega' = \frac{l_{\omega_{p\alpha}}}{l_{\omega_{1\alpha}}} \omega \quad (13)$$

In the same way for the spectral function  $l_{h_p}^*(\omega)$  we obtain

$$\omega' = a'\omega + b' \quad (14)$$

where

$$a' = \frac{l_{\omega_{p\alpha}} - l_{\omega_{p\beta}}}{l_{\omega_{1\alpha}} - l_{\omega_{1\beta}}} \quad (15)$$

$$b' = \frac{l_{\omega_{1\alpha}} l_{\omega_{p\beta}} - l_{\omega_{1\beta}} l_{\omega_{p\alpha}}}{l_{\omega_{1\alpha}} - l_{\omega_{1\beta}}} \quad (16)$$

The functions  $l_{g_p}(\omega)$  and  $l_{h_p}(\omega)$  form the function  $l_{f_p}(\omega)$  and consequently the required spectrum. In fig.7 the spectral function  $l_{f_p}(\omega)$  is compared with the experimental data with excellent results. The absolute differences are shown below.

## 6. Characteristic spectral parameters that depend on quench

It is possible to find many spectral parameters that depend on quench. Some of them depend on the whole distribution while others depend on a small portion of the spectrum. The first ones are called global parameters and the second local parameters.

Statistical parameters such as the spectral standard deviation or the average are global parameters while the position of the maximum, inflexion or end of the spectrum are local.

Whenever a dilatation or compression is performed the useful parameters are local.

An accurate determination of these characteristic points show statistical fluctuations and can be problematic.

The endpoint of the spectrum is not a well defined point. It is not easy to know the channel above which the information comes from the background or the spectrum. A reasonable criterion to define the end of the spectrum is to take the channel after which no channel has more than a fixed number of counts.

When one tries to define the maximum by one point we can also have difficulties. Due to statistical fluctuations the channel that corresponds to the maximum does not usually coincide with the channel that has the biggest count number. The channel of the maximum can be determined more precisely after smoothing and Fourier fitting. Therefore, it can be obtained with much more accuracy from the spectral function.

The inflexion point can be obtained by calculating the channel that corresponds to the half height of the maximum.

In fig. 7 the characteristic curves of the l-nuclide which give the position of the maximum, the inflexion and the end of the spectrum as a function of quench are shown. The value of these parameters, for a given quench value, can be obtained with higher accuracy from these curves.

## 7. Spectral fitting

When the spectral functions corresponding to the quench parameter of the experimental mixture have been computed, the

way to obtain the activity of each one of the components is a problem of least squares fitting.

Let  $y_j$  be the amount of counts in channel  $j$  for the mixture sample and  ${}^1y_j$  the amount of counts in channel  $j$  for the nuclide 1. Denoting by  $p_1$  the proportion of nuclide 1. We have:

$$y_j \equiv \sum_{l=1}^n p_l {}^l y_j \equiv \bar{y}_j \tag{17}$$

where  $n$  is the number of spectral components and  $y_j$  is the number of counts in channel  $j$  in the calculated spectra.

The counting rate for each one of the nuclides can be obtained from the condition of minimum for the quantity:

$$G(p_1, \dots, p_n) = \sum_{j=1}^m \omega_j [y_j - \bar{y}_j(p_1, \dots, p_n)]^2 \tag{18}$$

where  $\omega_j$  is the weight for channel  $j$  and  $m$  is the number of channels in the mixture spectrum.

The necessary and sufficient condition for the minimum of  $G$  is that the following equations hold.

$$\frac{\partial G}{\partial p_1} = \frac{\partial G}{\partial p_2} = \dots = \frac{\partial G}{\partial p_n} \tag{19}$$

These are equivalent to the system of equations:

$$\begin{aligned}
 p_1 \sum \omega_j ({}^1y_j)^2 + \dots + p_n \sum \omega_j ({}^n y_j) ({}^1y_j) &= \sum \omega_j y_j ({}^1y_j) \\
 \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots & \\
 p_1 \sum \omega_j ({}^1y_j) ({}^n y_j) + \dots + p_n \sum \omega_j ({}^n y_j)^2 &= \sum \omega_j y_j ({}^n y_j)
 \end{aligned}
 \tag{20}$$

which can be written in matrix form:

$$P Q = Y \tag{21}$$

where:

$$P = (p_1, \dots, p_n) \tag{22}$$

$$Q = \begin{pmatrix} \sum \omega_j ({}^1y_j)^2 & \dots & \sum \omega_j ({}^1y_j)({}^ny_j) \\ \dots & \dots & \dots \\ \sum \omega_j ({}^ny_j)({}^ny_j) & \dots & \sum \omega_j ({}^ny_j)^2 \end{pmatrix}$$

$$Y = (\sum \omega_j y_j ({}^1y_j) \dots \sum \omega_j y_j ({}^ny_j))$$

the  $p_1$  values are obtained from the equation:

$$P = YQ^{-1} \quad (23)$$

## 8. Spectral shift

Sometimes the calculated spectrum has a small shift when it is compared with the experimental spectrum. This shift is generally small and it has a value of a fraction of a channel.

This spectral shift is due to a non-accurate determination of the quench parameter for the samples. Then, the calculated spectrum does not correspond to the given quench value.

When the counting rates of each nuclide are more or less the same, the discrepancy due to a small shift is not very important. However, if the counting rate of one of the component nuclides is considerably greater than the others, a small spectral shift can produce large errors when the nuclides with less intensity are determined.

Once again we will make use of spectral functions to solve the problem. Let  $f(w)$  be the set of functions that represent the  $n$  radionuclides of the sample. Each one of these functions is translated until a minimum for  $D$  is reached, where  $D$  is:

$$D = \sum_{j=1}^m |y_j - \bar{y}_j| \quad (24)$$

This criterion is similar to the FOM given in ref [5,6].

When the required shift is greater than one channel, it means that the quench values of the samples have not been well determined. It is possible to do a new translation, but this time the



results will not be as good as before, since there will be also a large difference in shape.

## 9. Activity determination

The results of applying the least squares fitting, (see 8) are the quantities  $p_1$ . These values give the proportion of each one of the nuclides in the mixture. Let us denote by  $N_1$  the total number of counts for the 1 nuclide and by  $t$  the counting time in seconds for the mixture. The activity of each one of the nuclides is:

$$A_1 = \frac{p_1 N_1}{t \varepsilon_1} \quad (25)$$

where  $\varepsilon_1$  is the global counting efficiency for the quench value  $Q_p$  in nuclide 1.

The efficiency  $\varepsilon_1$  can be obtained from the counting efficiency curves using interpolation methods. If the samples used to obtain the spectra were standardized, the curve  $\varepsilon_1$  v.s.  $Q$  is straightforward. In case of using non standardized samples, it is just required to have the counting efficiency curve for  $^3\text{H}$  and apply the CIEMAT/NIST procedure to calculate the counting efficiency curve for every one of the radionuclides [7,8,9,10].

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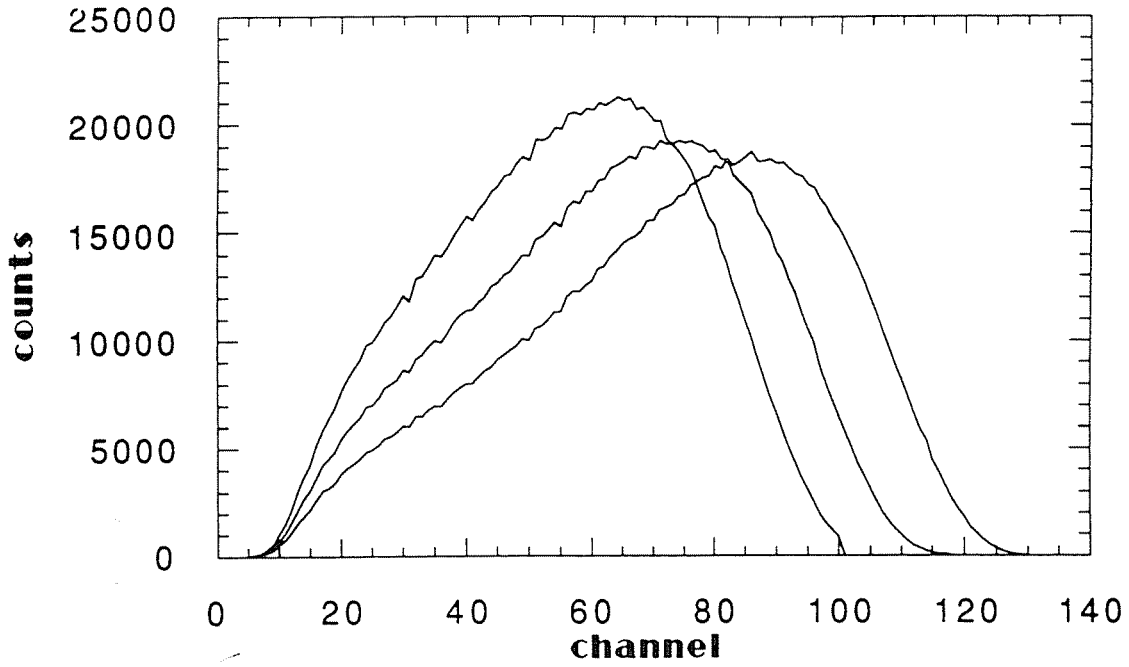


Fig. 1 Three pulse spectra of beta-ray emitter samples with different quench values.

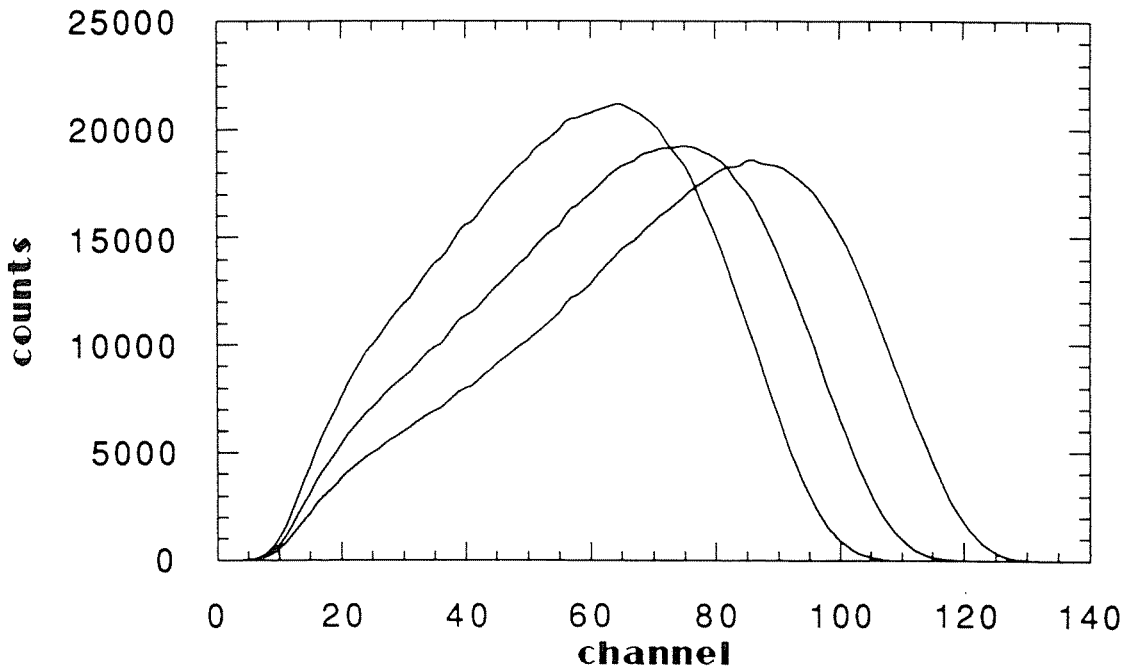


Fig. 2 The spectra of Fig. 1 after applying smoothing.

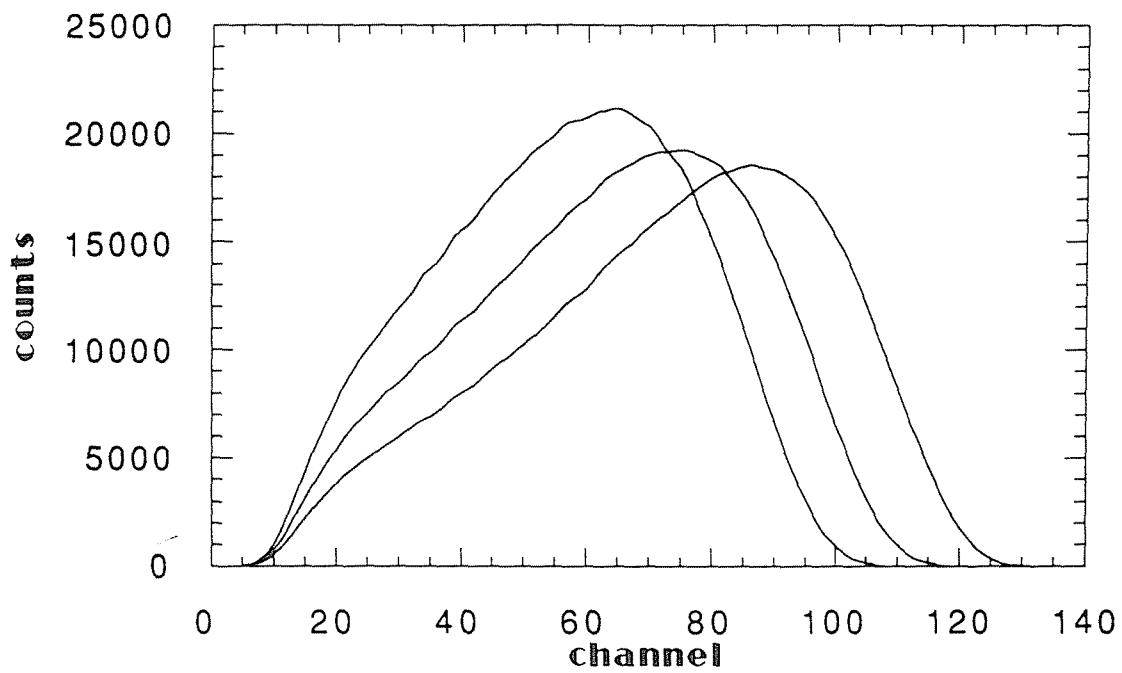


Fig. 3 Spectral functions obtained from Fourier series.

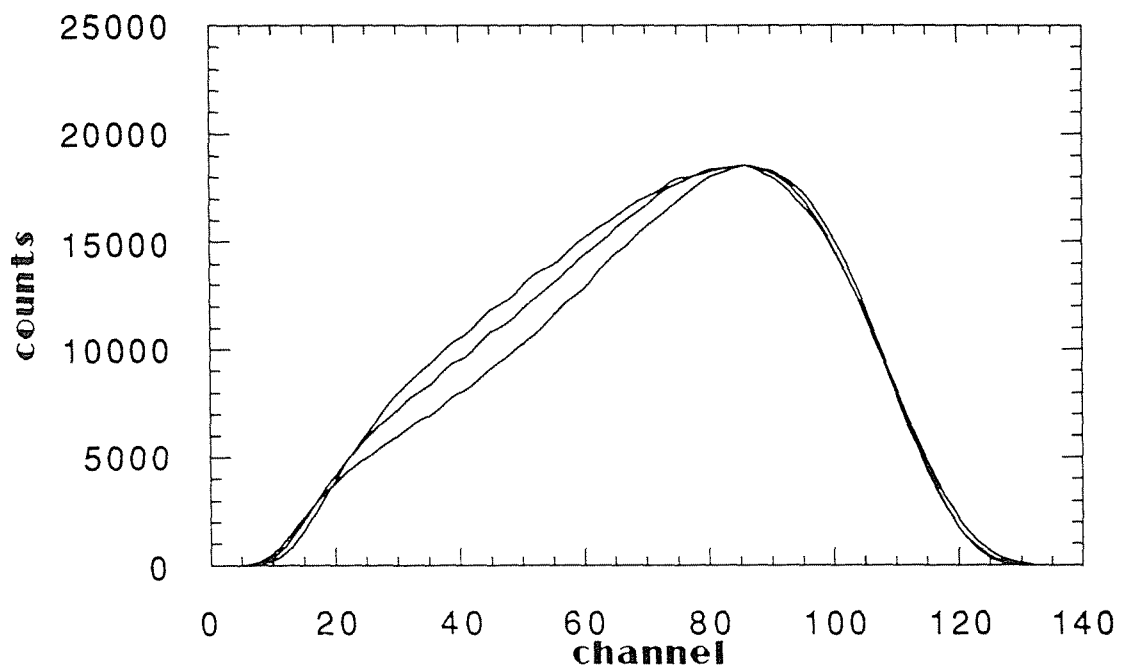


Fig. 4 Dilated spectral functions ready to apply an interpolation procedure.

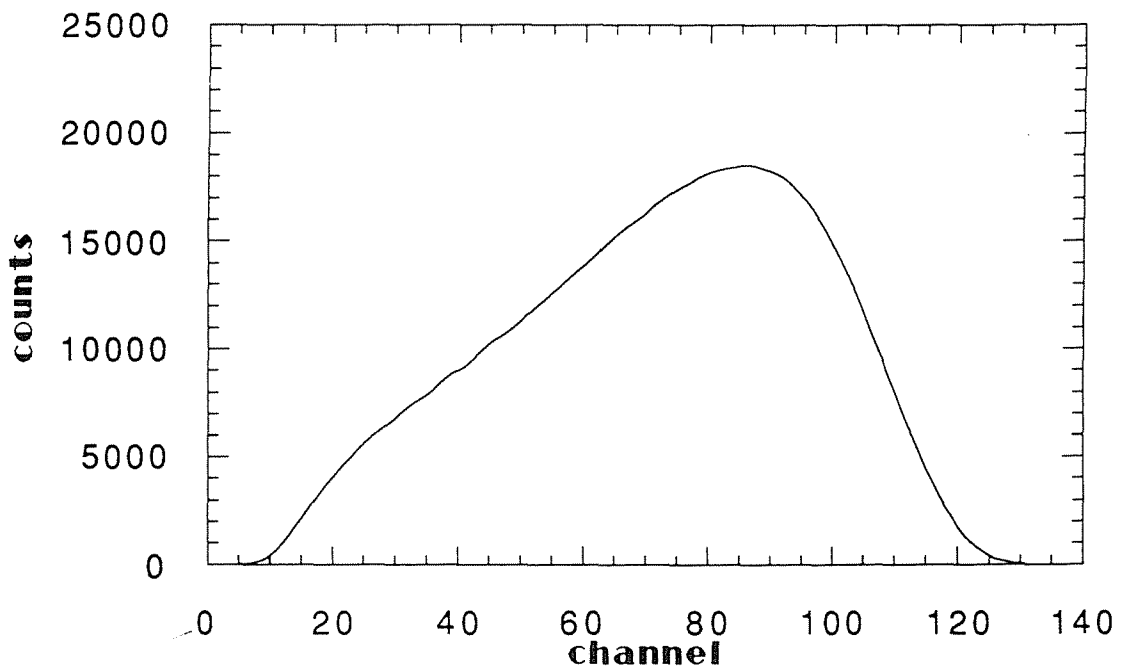


Fig. 5 Interpolated spectral function corresponding to a particular value  $Q_p$  of the quench parameter.

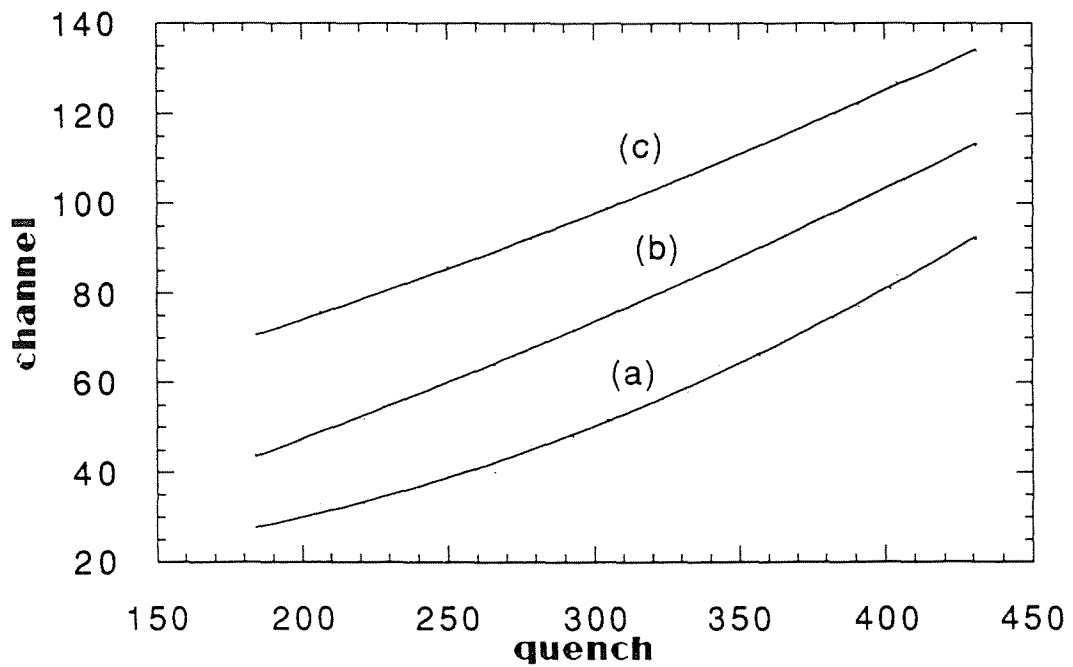


Fig. 6 Spectral parameters depending on the quench: (a) position - of the maximum, (b) inflexion point, (c) end of the spectra

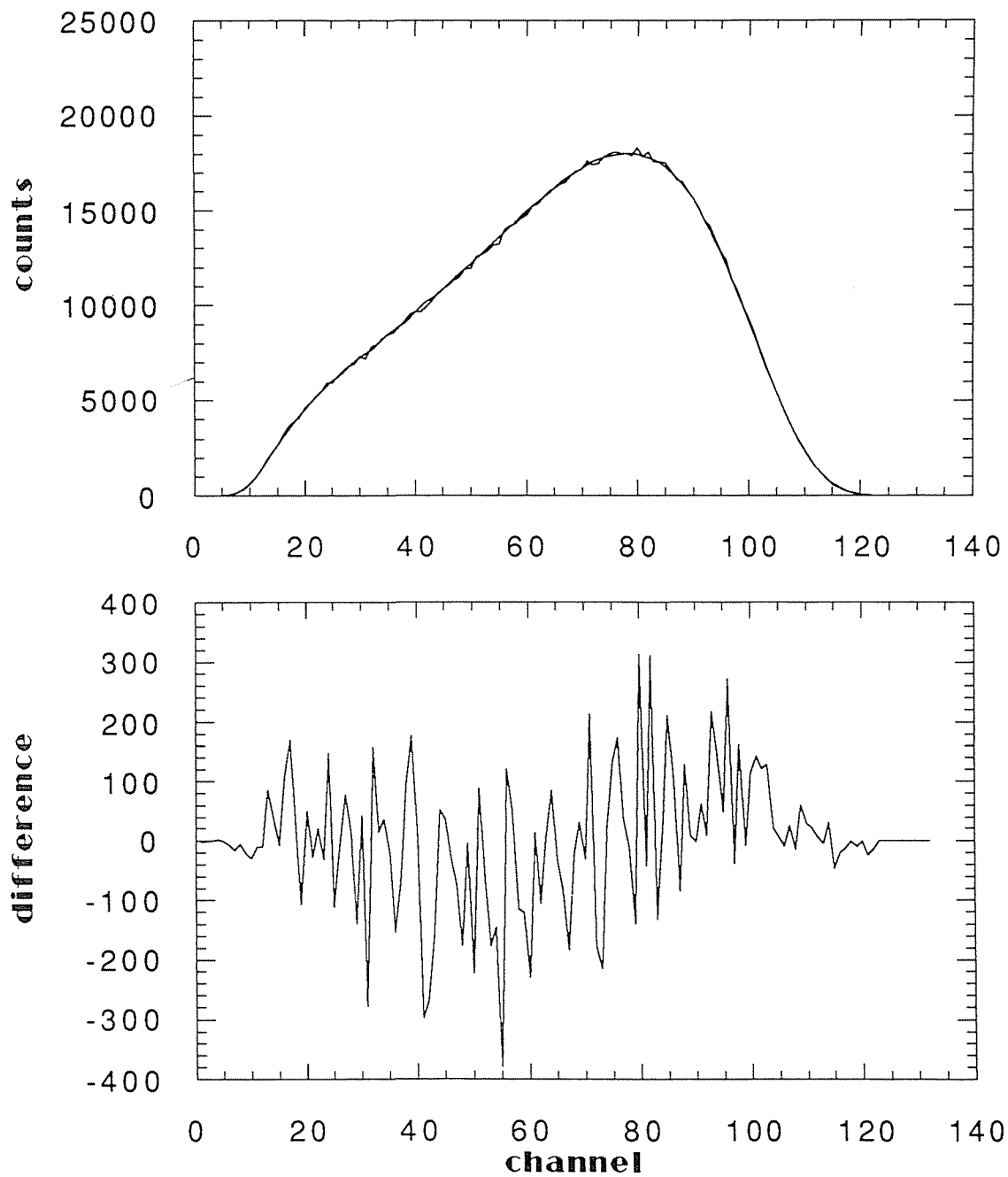


Fig. 7 The spectral function and the experimental spectrum for the same quench parameter are compared. The - difference spectrum corresponds to the statistical fluctuations.

## CIEMAT-675

Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas.  
Instituto de Investigación Básica. Madrid.

"Interpolación y descomposición espectral para la medida por centelleo líquido de muestras marcadas con varios radionucleídos".

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GRAU CARLES, A.; GRAU MALONDA, A. (1991) 17 pp., 7 figs., 10 refs.

Se describe en este trabajo un nuevo procedimiento para obtener la actividad de cada uno de los radionucleídos de una mezcla compleja. Para ello se aprovecha la información contenida en el espectro de amplitud de los impulsos de los espectros de centelleo líquido. A fin de obtener una buena descomposición espectral se describen los pasos necesarios para el ajuste de las formas elementales a los espectros complejos. Estos consisten en la dilatación, interpolación y contracción de las formas elementales. El procedimiento se puede aplicar a mezclas de radionucleídos que se desintegran por emisión  $\beta^-$ ,  $\beta^- - \gamma$ ,  $\beta^+$ ,  $\beta^+ - \gamma$ , EC, EC -  $\gamma$  y transición isomérica.

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## CIEMAT-675

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