Chapter 17

# Desktop Calculator-Assisted Liquid Scintillation Spectrometry of $^3H$ and $^{14}C$ with Special Consideration of Low Level Activities and Spectrometer Instability

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## SUMMARY

For the liquid scintillation measurement of $^3H$ and $^{14}C$ in single- and dual-labelled samples with the same spectrometer settings, which are desirable for routine work, the lower discriminator level of the $^{14}C$ channel has to be set down to a value for which the tritium crosstalk $\alpha$ amounts to about 15%. Under these conditions, the short time variations of $\alpha$ are important for the overall attainable precision. A simple method for the automatic computation of drift corrections based on the continuous measurement of a standard sample is presented. Furthermore, for low activities an appreciable improvement of the precision may be achieved by systematic re-use of the glass vials together with the cumulated measurements of their individual backgrounds. The precision of low activity measurements is also enhanced by correction of background counting rates for quenching.

In order to simplify operating conditions in routine work, it is advantageous to use the same measuring channels for single- and dual-labelled samples. In doing so a further advantage is gained: All single-labelled samples are also treated as dual-labelled and thus cross contamination is automatically detected. The considerations which follow were developed in order to meet this experimental situation; they apply to LSC coincidence spectrometers with external standardisation. Three "Mark I" (Nuclear Chicago, Searle) spectrometers with Ba-133 external standard were at our disposal. They showed very similar, but not identical, characteristics, and were operated with channel settings chosen to make them interchangeable in routine work, with proper consideration of their individual response at the data reduction stage. Data output was by teletype (TTY) on punched tape. Calculations were done off-line using a programmable calculator (Hewlett Packard 9820A) equipped with tape reader, plotter and TTY with tape punch. The improvements suggested are, however, not restricted to this instrumental set-up.

A general study of the overall efficiency, taking into consideration the prevailing working conditions (relative quota of samples of different types, as $^3H/^{14}C$, single/double, high level/low level, and so on) as well as basic principles of counting statistics, shows that, in order to optimise the output of the laboratory, about 15% of the $^3$H-activity has to be admitted in the $^{14}$C-channel of the spectrometers. On the other hand, as the required long-term consistency of the measurements is set at a fairly high level, say 2% for the r.m.s.-deviation of single measurement, it ensures that the instrumental parameters $f_T$ ($^3$H efficiency in $^3$H-channel), $f_C$($^{14}$C efficiency in $^{14}$C-channel), $\alpha$ ($^3$H in $^{14}$C-channel/$^3$H in $^3$H-channel) and $\beta$ ($^{14}$C in $^3$H-channel/$^{14}$C in $^{14}$C-channel), which are functions of $\gamma$ (external standard in standard channel/external standard in $^{14}$C-channel), must always be known precisely. Thus, the fitting of analytical functions to the experimental data with a sufficiently high degree of approximation necessitates the use of polynomials of up to the seventh degree. Under such conditions, special care has to be taken in view of the short-term instabilities of the spectrometers, which are variations of gain and discrimination, and affect mainly the stability of $\alpha$. A constant re-evaluation of this parameter would represent a prohibitive amount of work. It appeared, however, that the variations of $\alpha$ may be satisfactorily corrected by an appropriate shift of the whole $\alpha$-curve along the $\gamma$-axis (Fig. 1), which constitutes an important simplification of the situation. In practice, the correction method consists of determining the value $(\alpha, \gamma)_i$ of a $^3$H standard together with each group of samples to be measured. The calculator automatically finds by iteration the shift $\Delta\gamma_i$ of the function $\alpha$ which corresponds to the

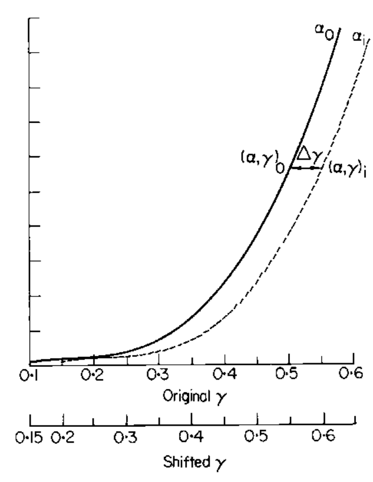


*Fig. 1.* Correction for drift of $\alpha$.

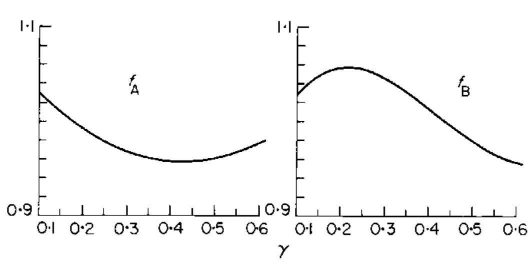


*Fig. 2.* Quench effect on background.

actual state of the spectrometer. This method of correction requires a minimal amount of work and measuring time. The component of error eliminated in this way amounts typically to about 1.5%.

In the search for further possibilities to improve the measurement of low activities by simple means, two points were found, by which some more precision may be gained. A first improvement consists in taking into account the dependence of the background on the degree of quenching of the samples. Figure 2 shows typical curves for the background in the $^{3}H$- and $^{14}C$-channels. A second improvement may be achieved by re-using systematically the same glass vials and calculating cumulated background mean values for each vial, which tend asymptotically to constant figures of higher statistical weight. The increment of precision gained thereby amounts typically to about 1%. Detailed computation programs are available upon request.