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Sub Title	
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Publisher	共立薬科大学
Publication year	1986
Jtitle	共立薬科大学研究年報 (The annual report of the Kyoritsu College of Pharmacy). No.31 (1986.) ,p.68- 69
JaLC DOI	
Abstract	
Notes	抄録
Genre	Technical Report
URL	https://koara.lib.keio.ac.jp/xoonips/modules/xoonips/detail.php?koara_id=AN00062898-00000031-0068

No. 31 (1986)

Absolute Liquid Scintillation Counting of Colored Samples of β-Emitters Using Integral Counting Techniques*

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The advantages of the use of integral counting techniques with a liquid scintillation spectrometer for the determination of the absolute disintegration rate of α-emitters have been demonstrated by Basson and Steyn and Wright, Steinberg and Glendenin, and for the determination of the absolute disintegration rate of β-emitters with a maximum energy higher than 200 keV by Steyn, Flynn and Glendenin and Goldstein. This technique involves measuring the integral counting rate as a function of pulseheight discriminator bias. Under the proper conditions, the extrapolated value is identical with the absolute disintegration rate.

In the work described below, integral counting techniques have been extended to the determination of the absolute disintegration rate for the colored sample of β-emitters by means of a spectral research program of a common piece of the latest apparatus, a Beckman Model 5801 liquid scintillation spectrometer. We have found that the extrapolation method is virtually unaffected by the presence of coloring agents for β-emitters with a maximum energy higher than 200 keV. Because the color quenching effect is one of the major problem in using liquid scintillators especially in biochemical and medical analysis, this information will be of value to users of liquid scintillation system interested in determination of the absolute disintegration rate of colored samples.

The absolute disintegration rate of ^{32}P, ^{35}S, ^{36}Cl, ^{45}Ca and ^{131}I was determined in a conventional 2π proportional counter mounting on a thin polyvinylidene chloride film. Then the same sample was dissolved in 0.5—1.0 ml water and introduced into a sample vial together with the film and 9 ml of Insta-gel Emulsifier (Packard Instr., Co.) and then 4 ml of liquid scintillator was added to make up a total volume of 13.5—14.0 ml solution. The liquid scintillator solution used here consisted of 4 g PPO (2,5-diphenyloxazole) in 1 liter of toluene.

In this work, two types of coloring agents were employed, methyl red (MR) and bromothymol blue (BTB). The MR solution consisted of 0.004 g MR in 50 ml ethanol, which was acidified with HCl. The BTB solution was prepared dissolving 0.003 g of BTB in alkaline solution of 50 ml ethanol. The color-quenched sample was prepared by adding the appropriate amount of the solution of the coloring agent to the sample scintillator solution until the color caused by BTB or MR was present.

* 本報告は *J. Radioanal. Nucl. Chem., Letters* **105** (4), 241-248 (1987) に発表

A commercially available liquid scintillation spectrometer, LS 5801, Beckman Instrument, Inc., Irvine, Ca., was used in this work. The sample was counted integrally at several pulse-height discriminator settings using the spectral research program of the liquid scintillation system. The pulse height-energy relationship of the integral counting curves was based on that measured by Quint.

The integral counting curves for ^{32}P, ^{35}S, ^{36}Cl, ^{45}Ca and ^{131}I are shown in Figs. 1 and 2. The extrapolated intercept value for the coloring-agent-free sample of the energetic β-emitter (i.e., ^{32}P, ^{36}Cl, ^{45}Ca and ^{131}I) agrees satisfactorily with the disintegration rate determined by conventional 2π β-counting of the sample. The results also indicate that in the case of ^{35}S, the extrapolation amounts to a correction of about 96.3%. These results confirm the work of Horrocks et al., Flynn and Glendenin and Goldstein.

The effect of the coloring agent is also clearly illustrated. The integral counting curve of the colored samples for ^{32}P, ^{36}Cl, and ^{131}I was a straight line which could be extrapolated accurately to the same point.

In the case of ^{45}Ca, not only the color-quenched curves but also coloring-agent-free ones show slightly curved portions especially near zero pulse height. However, the integral counting curves for ^{45}Ca can be extrapolated to the same point. The data for BTB show that the counting efficiency for the colored sample of these energetic β-emitters is close to 100% : $97.9\pm0.3\%$ for ^{32}P, $100.8\pm0.4\%$ for ^{36}Cl, $97.6\pm0.3\%$ for ^{45}Ca, and $100.4\pm0.4\%$ for ^{131}I.

The data for MR are also given. The counting efficiency of the colored sample of the β-emitters is also high : $96.0\pm0.3\%$ for ^{32}P, $101.8\pm0.6\%$ for ^{36}Cl, $97.1\pm0.4\%$ for ^{45}Ca, and $96.0\pm0.3\%$ for ^{131}I.

The slope, intercept and standard deviation of the integral counting curves are computed by a least-squares method. Precision is calculated as the sum of the maximum estimates of possible errors. The principal errors are due to : source preparation ($<\pm0.1$ %), estimation of the extrapolated value ($<\pm0.3\%$), counting losses due to dead time of the counting system ($<\pm0.1\%$), standard statistical error ($<\pm0.6\%$). Thus the total error adds up to about 0.7%.

Usually the quenched pulse height spectrum caused by color differs in shape from that by chemical quenching. However, it is apparent that under the present conditions the counting efficiency of the colored sample for β-emitters with a maximum energy higher than 200 keV is almost 100% with a precision better than 0.7%. Down to 0.1%, no essential obstacles against further improvements of precision and accuracy of this method inherent in the detector have shown up yet.