

LIQUID SCINTILLATION COUNTING OF LOW ENERGY β EMITTERS

B. N. AUDRIC

Chemical Research Laboratory, Teddington, Middlesex, England

At the Chemical Research Laboratory, Teddington, England two types of liquid scintillation apparatus have been constructed for counting low energy radioactive isotopes.

The original apparatus comprises a single 1 in. diameter cathode eleven stage E.M.I. photomultiplier, type 5311, and a 6 ml silica Beckmann cell. The phototube is inserted into a 6 in. lead cube for shielding and the silica cell is contained in a 2 in. diameter brass slide which slides into the lead block at right angles to the axis of the phototube. The slide is light-tight in order that samples may be easily changed. Unfortunately the arrangement does not give a good optical contact between the cell and the photocathode. The electronics are simple and comprise a power unit for EHT, a low gain amplifier, and scaling unit. The original phosphor was 5 g/l. *p*-terphenyl in benzene.

The optimum operating conditions give an efficiency of 40% for C^{14} with a background of 150 counts/min a large proportion of which is photomultiplier noise. No other photomultiplier, either E.M.I. or Du Mont, that we have examined has been found to give a comparably low noise count. Since its construction this apparatus has been used for many assays of organic compounds containing active carbon.

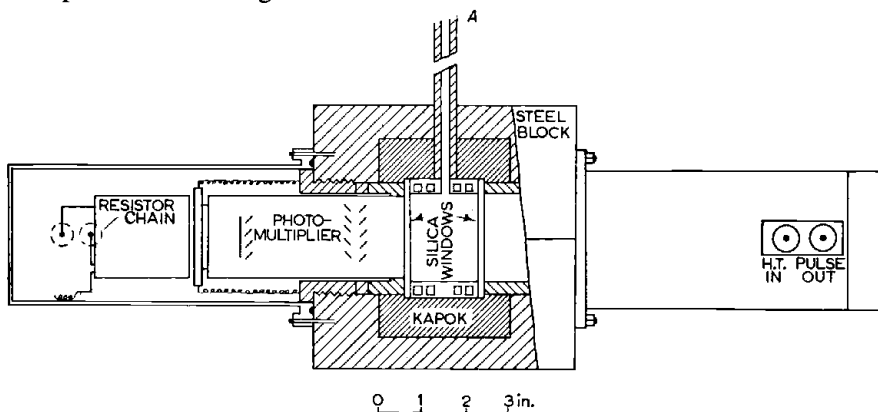


Fig. 1.

The apparatus for low activity counting by two photomultipliers in coincidence is shown in Fig. 1. The steel cell has a capacity of 90 ml, it is

silverplated internally and can be cooled to -70°C by pumping cold toluene-ethanol mixture through the annulus in the cell wall. The cell is vacuum tight and can be filled with phosphor solution through the tube A. The cell and photomultipliers are externally shielded with 4 in. of steel bricks and $1\frac{1}{2}$ in. of lead. Although it was realised that light interaction could probably be reduced by placing the photomultipliers at right angles to one another, the present arrangement was used for simplicity.

Figure 2 shows the arrangement of the electronics. These differ from the usual arrangement in that high sensitivity Kandiah discriminators have been used. The operating conditions are thus comparable with apparatus using

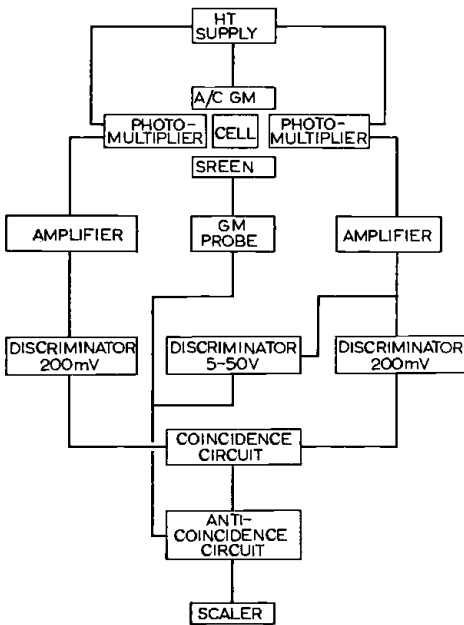


Fig. 2.

the conventional Schmidt discriminators where the pulse height would be in the range 5–0.5 mV at the photomultiplier corresponding to an amplifier gain of 10^3 – 10^4 with a bias of 5 V. In our case the conditions are about 2 mV at the photomultiplier with a gain of 100 and a bias of 0.2 V. The stability of the apparatus is good, no variation in total count rates of 20,000 in either sample or background have been found outside the amount expected from statistical variations.

The photomultipliers used are eleven stage E.M.I. type 6260 tubes. These have Pyrex windows and the envelope glass is low in potassium content and thus do not contribute appreciably to the background count. A recent check

on two of these tubes showed that when first assembled in the dark the noise count from a single tube was about 4000 counts/sec. After 48 hr this had dropped to about 150 counts/sec which gives a random coincidence rate of less than 1 counts/min. Thereafter the noise decreased more slowly and after two weeks reached about 100 counts/sec. This is at room temperature (20–25°C). No appreciable counting losses are introduced by the dead time of the Kandiah discriminators (150 μ sec). The coincidence resolving time is 0.4 μ sec giving a random noise count of $2 \times 100^2 \times 0.4 \times 10^{-6}$ counts/sec or 0.5 counts/min.

The background, with anticoincidence Geiger counters but no top level discriminator, will arise in part from the following sources, for which the estimated values are:—

$\alpha + \beta$	< 5 counts/min	
Cosmic rays	< 5 counts/min	based on known cosmic ray flux and actual cosmic ray coincidence rate observed with a phosphor.
Residual K ⁴⁰ in phototubes	< 3 counts/min	calculated from known K ⁴⁰ content of photomultiplier glass.
Random coincidence rate	< 1 counts/min	
Total	< 14 counts/min	
Observed rate	52 counts/min	with inactive phosphor in the cell
Difference	> 38 counts/min	

Again with anticoincidence shield operating the following values were observed:—

<i>Cell Filling</i>	<i>Count</i>
In active phosphor	52 counts/min
Empty cell	17 counts/min
Cell filled with opaque dye	< 1 counts/min

The count rate of 17 counts/min with the empty cell must therefore be due to some form of light interaction between the photomultipliers. It seems extremely likely that the count due to this cause will be higher than this figure with the phosphor in the cell since there will be much improved light coupling between the photocathodes. The discrepancy between the estimated background and the observed figure of 52 counts/min is therefore probably made up of light interaction pulses and gamma-ray pulses from contaminated shielding.

The curve of counting rate against EHT (Fig. 3) for a background solution shows a smaller slope than the corresponding curve for active carbon. It would be expected that a discriminator arranged to reject pulses above a certain energy would preferentially reject large background pulses. Such an

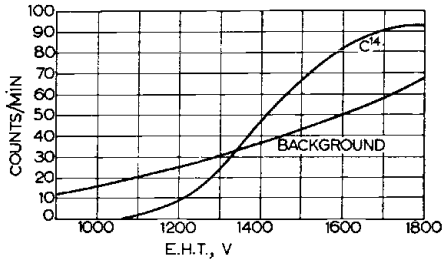


Fig. 3.

arrangement has been used successfully by Hayes. Figure 4 shows the results obtained when using such a discriminator. From the curve it will be seen that the top level discriminator begins to reject the longer C^{14} pulses when the level has reached 8.3V. At this point the background is 30 counts/min.

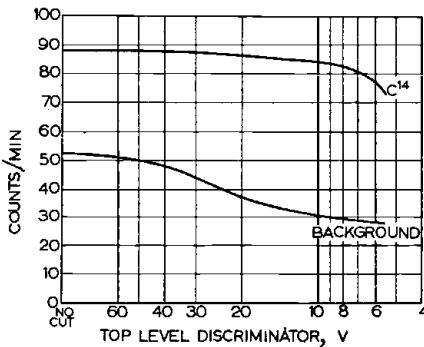
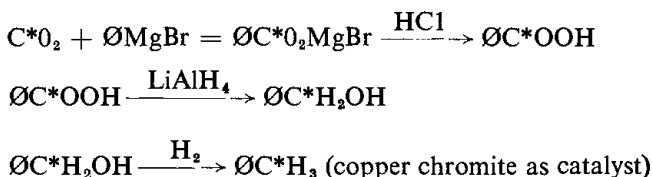


Fig. 4.

A comparison has been made between tubes having two different types of cathode, both manufactured by E.M.I. These have respectively red: blue sensitivities of about 1:9 and about 1:82 but no significant difference in background and C^{14} counting efficiency has been found. The noise levels of both sets of tubes at the optimum working voltage were approximately the same and the empty cell light interaction counts were almost identical.

Two methods of introducing labeled carbon into the apparatus for assays on very low specific activity materials have been examined. Originally acetylene, which could be prepared by the syntheses of Crathorn or Suess, was dissolved in the phosphor (4 g/l. diphenyl-oxazole in toluene with the

addition of 2% 'dead' ethanol to keep the solution clear at low temperatures). The phosphor was cooled to -70°C . It was found that 6 l. was the largest volume of acetylene which could safely be dissolved. This was equivalent to 7 g of labeled carbon and could be counted with an efficiency of about 45%. Because of the hazards involved in handling acetylene and the difficulty of storing large gas samples it was decided to introduce the labeled carbon as the toluene in the phosphor solution. The synthesis used is similar to that of Pringle:



Overall yield about 75%.

Using labeled toluene no cooling is necessary and 10.4 g of labeled carbon may be introduced into the cell and can be counted with an efficiency of 50%. The efficiency of the apparatus was determined by using as a standard the labeled polymethyl methacrylate issued by the Radiochemical Centre, Amersham, England. The activity of this material has been checked against the C^{14} standards issued by the National Bureau of Standards, Washington, D.C. Initial tests showed that the presence of the methacrylate in the phosphor did not depress the count rate for C^{14} .

Little experimental work has been carried out using tritium labeled compounds. Under the same counting conditions as those suitable for C^{14} (which are not necessarily the optimum conditions for H^3) the efficiency is 5–10%. A closer estimate of the efficiency was not possible since the specific activity of the tritium labeled compound used was not known accurately.

Acknowledgments—Finally I would like to acknowledge my indebtedness to my former colleague J. V. P. Long of the Cavendish Laboratory, Cambridge, with whom I have had many valuable discussions at all stages of the work. I also wish to acknowledge the assistance of Dr. H. S. Turner of the Chemical Research Laboratory for his assistance with the organic synthesis.