INTRODUCTION TO THE LIQUID SCINTILLATION COUNTER

OBJECTIVE

To familiarize the student with the liquid scintillation counter.

THEORY

The problem solved by the apparatus to be described is that of detecting very low levels of sample light emission in the presence of background light. Figure 1 is a block diagram of a basic liquid scintillation counter. Referring to Figure 1, the top view of a sample vial can be seen between two phototubes. Most of the light emitted from the sample vial strikes the photocathode of the photomultiplier tube. Since the photon emission distribution from fluorescing molecules is approximately uniform in all directions, there is a high probability that photons will strike both photomultiplier tubes simultaneously. If this occurs, the coincident gate opens, which in turn permits the summing amplifier to sum the two phototube pulses and release its output to the discriminators. Depending on the discriminator's setting, the pulse from the summing amplifier enters a scaler and is thus counted. Accumulation of this count occurs for the duration of the count. The count may be read out at the end of the counting interval, or as the counts occur. It is clear from the preceding text that the coincidence requirement discriminates against single photon events, since a single photon cannot strike both phototubes simultaneously. Obviously, multiple photon events are not discriminated against. It is not yet clear then why a coincidence technique is used! By means of explanation, consider the phototube itself as a source of noise. If the noise of the individual phototube is measured in counts per minute, it would be found to be as much as 1×10^4. It turns out that this noise is random in nature and may be thought of as a series of single photon events. The coincident requirement then excludes tube noise!

On the other hand, it also excludes single photon events arising from β^--scintillation fluid interaction; that is, real sample counts. This is an unfortunate consequence of the coincidence counting technique. The low energy β^-s are the most effected by the coincidence requirement. For example, 28% of all tritium β^-s cause single photon events when they interact with the scintillation fluid, but are never counted because of the coincidence requirement. ^{14}C losses are only

~ 3% and ^{32}P's losses are insignificant. Recall that tritium's maximum energy is 0.018 MEV, ^{14}C's maximum energy is 0.155 MEV and ^{32}P's maximum energy is 1.710 MEV. It can be seen that as the maximum energy of the isotope increases, the coincidence losses decrease.

It should be mentioned that a large part of the radiation background of the environment cause single photon events and are thus eliminated from the counting data by the coincidence requirement. All multiple photon events, whether from the sample activity or the background, pass the coincidence requirement.

MATERIALS

1. 3 LS vials - 1" X 2 1/2" (Low K)
2. 45 ml scintillation fluid whose composition is:
   a. 8 gms/l PPO = 2, 5-diphenyloxazole
   b. 0.4 gm/l dimethyl POPOP = 1, 4 - bis[2-(4-methyl-5-phenyloxazolyl)]-benzene
   c. toluene to volume
3. Several microliters of the following labeled material:
   a. ^{3}H labeled toluene
   b. ^{14}C labeled toluene

PROCEDURE

1. Identify all of the electronic controls on the counter.
2. Examine and operate the sample introduction system.
3. Prepare 3 samples as described in Table I.
4. With a set of so called "unquenched standards" set the instrument's gain as described in the manual.

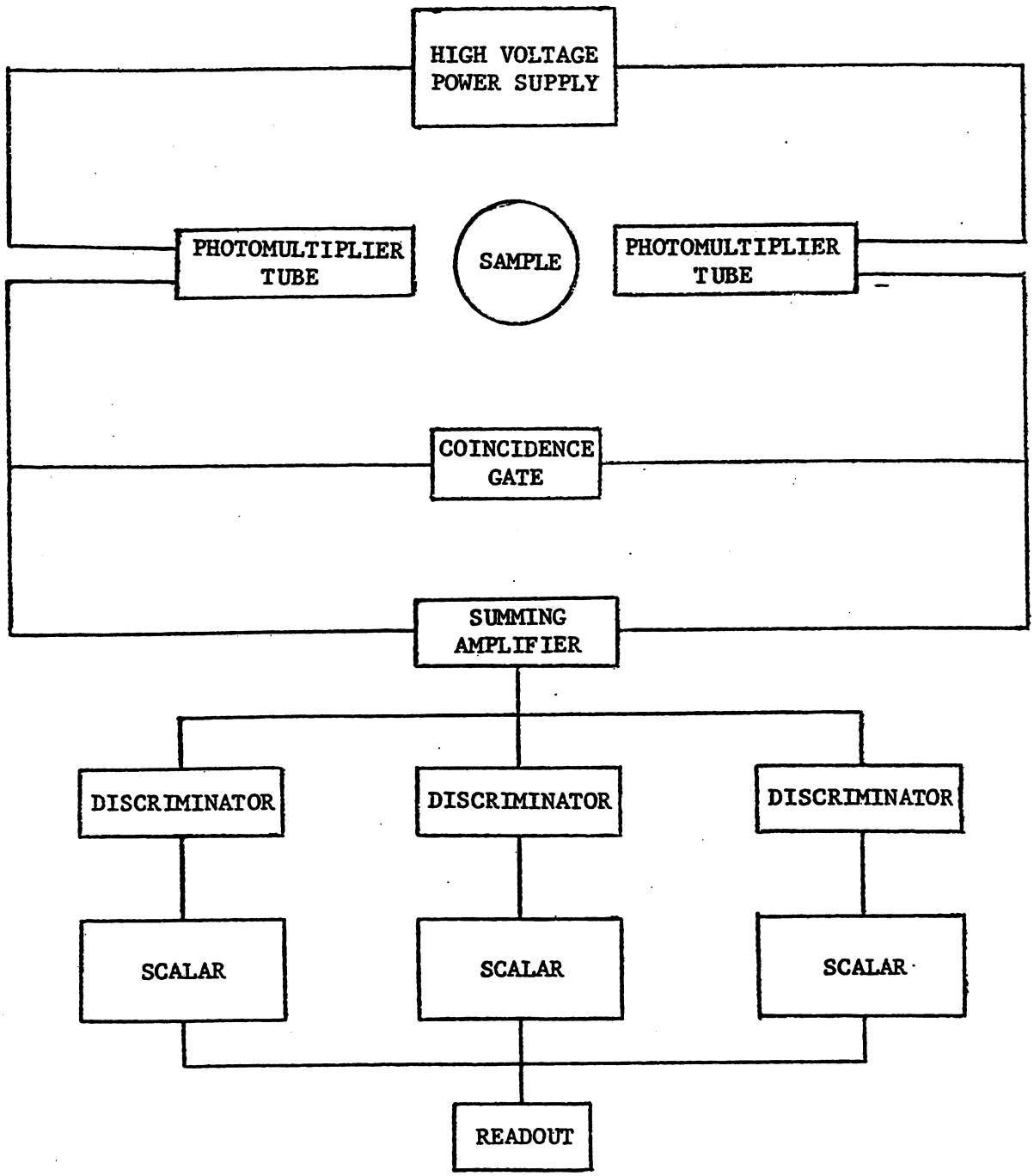
TABLE I

Sample #	Description	Volume Scintillation Fluid	Activity DPM	CPM
1	Bkgd.	15	0	( )
2	^{3}H	15	60,000	
3	^{14}C	15	60,000	

5. Count samples 2 and 3 for one minute in a wide open window, and record data in space provided in Table I.

6. Count sample 1, the background sample, in a wide open window for one minute. Repeat count 10 times and enter the average value for the background sample in the space provided in Table I.

Figure 1.


HIGH VOLTAGE POWER SUPPLY
PHOTOMULTIPLIER TUBE
SAMPLE
PHOTOMULTIPLIER TUBE
COINCIDENCE GATE
SUMMING AMPLIFIER
DISCRIMINATOR
DISCRIMINATOR
DISCRIMINATOR
SCALAR
SCALAR
SCALAR
READOUT

DETERMINATION OF β^- SPECTRUM

OBJECTIVE

To measure and display on two sets of coordinate axis a β^- spectrum and a background spectrum in the absence of a β^- spectrum.

THEORY

β^- particles emerge from the nuclei of atoms which are referred to as unstable. The primary reaction which leads to β^- emission is

$$n \rightarrow p^+ + \beta^- + \bar{\nu} \qquad (1)$$

where n = neutron in the nucleus

p^+ = proton

β^- = beta particle whose mass and charge are exactly the same as an electron

$\bar{\nu}$ = anti neutrino

The energetics of this reaction, in general terms, are represented as follows:

$$E_n = Ep^+ + E_{(\beta^- + \bar{\nu})} \qquad (2)$$

$$E_{(\beta^- + \bar{\nu})} = E_{\beta^-} + E_{\bar{\nu}} \qquad (3)$$

All three terms of (2) are invarient with respect to a given isotope; hence, any values of E_{β^-} and $E_{\bar{\nu}}$ are acceptable as long as their sum, $E_{(\beta^- + \bar{\nu})}$ is invarient. Since E_{β^-} may vary from $E_{(\beta^- + \bar{\nu})}$ when $E_{\bar{\nu}} = 0$ to zero when $E_{\bar{\nu}} = E_{(\beta^- + \bar{\nu})}$, a distribution of E_{β^-} vs. population characterizes each unstable isotope, which emits β^- rays.

A liquid scintillation counter does not count β^- rays, but the photons emitted as the result of β^- -solvent-fluor interaction. The following reaction diagrams will delineate the energy transfer process:

$$\beta^- + S_1 \rightarrow S_1^* + (\beta^- - E^*) \quad (1a)$$

$$S_1^* + S_2 \rightarrow S_1 \cdot S_2^* \quad (1b)$$

$$S_{n-1}^* + S_n \rightarrow S_{n-1} + S_n^* \quad F_1 \quad S_n + F_1^* \quad (1c)$$

OR

$$(S_{n-1} \cdot S_n)^* + F_1 \rightarrow S_{n-1} + S_n + F_1^* \quad (2)$$

$$F_1^* \rightarrow F_1 + h\nu_1 \quad (3)$$

$$F_2 + h\nu_1 \rightarrow F_2^* \quad (4)$$

$$F_2^* \rightarrow F_2 + h\nu_2 \quad (5)$$

S = Solvent
S^* = Solvent (excited state)
$(S_{n-1} \cdot S_n)^*$ = Solvent eximer
F_1 = Primary fluor (solute)
F_2 = Secondary fluor (solute)
$h\nu_1$ = 3500-3800 Å
$h\nu_2$ = 3900-4300 Å
E^* = Energy lost by β^- in interaction

Note that steps one through two are radiationless, while steps three through five involve electromagnetic radiation. The β^- energy transfer can be modified by variations in the mole fraction relationships which exist among the sample components. Hence, from one sample to the next, the instrument's response may vary; however, the shape of the β^- spectrum is invarient, where the β^- spectrum is an intensity vs. energy distribution. The background spectrum is quite different from the β^- spectrum. The components of the background spectrum are: crosstalk (between photomultiplier tubes), electronic noise, dark current (electronic noise in individual tube), Cerenkov radiation (cosmic ray striking glass, or solvent), ^{40}K in glass parts (tube envelope and glass vial), and radioactivity in the environment such as ^{222}Rn (gas) and others.

MATERIALS

1. Two scintillation vials - 1" X 2 1/2" (Low K)
2. 20-30 mls scintillation fluid whose composition is:
   a. 8 gms/l PPO = 2, 5-diphenyloxazole
   b. 0.4 gms/l dimethyl POPOP = 1, 4 - bis[2-(4-methyl-5-phenyloxazolyl)]-benzene
   c. Toluene to volume
3. Several microliters of an activity bearing solution:
   a. ^{3}H labeled toluene
   b. ^{14}C labeled toluene
   c. $H_3\ ^{32}PO_4$

PROCEDURES

Into each of two empty vials add 10-15 ml's of scintillation fluid. Into one of the vials add a volume of ^{3}H activity such that 1 X 10^{6} dpm are added. Place the sample in the counting well, count as indicated in Paragraph 1, and record data in Table I.

1. Using the window settings indicated in Table I, tabulate the number of counts measured in 1 minute, using the indicated discrimination levels or windows.

TABLE I

Window Settings in % of Spectrum*			
Lower Level	Upper Level	Total Counts ± Error	Background Counts ± Error
0	10		
10	20		
20	30		
30	40		
40	50		
50	60		
60	70		
70	80		
80	90		
90	100		

2. Plot the average of the lower level setting and the upper level setting vs. counts, and show the error zone allowed along the plot. Remove the sample containing the isotope and replace with the second sample which contains no activity. Proceed as directed in Paragraph 1 and measure the background in the same manner as the isotope containing sample. Plot the average of the lower level setting and the upper level setting vs. counts.

QUESTIONS

1. Criticize the β^- spectrum as plotted. Discuss any inconsistancies. (See any nuclear physics text for β^- spectra.)
2. Knowing the endpoint energy of the β^- emitter and the energy at which the maximum occurs, convert discriminator setting to approximate energy in mev and record the numberical relationship of E_{ep} to E_{max}.

* For ^{3}H, the array of settings will range from 0 to 350 on the variable discriminator in 10% intervals. For ^{14}C, the array of settings will range from 0 to 550 on the variable discriminator in 10% intervals.

3. What special information is the result of knowledge of the β^- emitter's maximum and endpoint energy?

OPTIONAL

Repeat this experiment with another radioisotope such as ^{14}C or ^{32}P. It will not be necessary to repeat the background measurement.

THE EFFECT OF VARYING AMPLIFIER GAIN

OBJECTIVE

To illustrate the manner in which gain influences the photomultiplier's representation of the β^- spectra.

THEORY

When we refer to gain, it should be remembered that a change in gain is a change in amplification and is reflected as changes in the pulse area. The electrical output of the phototube as a result of its photocathode being struck by a certain number of photons, is referred to as a pulse. Area and height are measurable characteristics of this pulse; and of particular interest is the pulse area since it is proportional to the number of photons which started the pulse. Since the number of photons emerging from the vial per β^- event is proportional to the energy of the β^-, the pulse area is proportional to the β^- energy. Each pulse results in the registration of a count. A distribution of the counts is made on the basis of pulse area and hence, β^- energy. Since pulse area is a function of β^- energy, changes in pulse area result in shifts in the count distribution or β^- spectral shape.

MATERIALS

1. Four scintillation vials - 1" X 2 1/2" (Low K)
2. 40-60 mls. scintillation fluid whose composition is:
   a. 8 gm/l PPO = 2, 5-diphenyloxazole
   b. 0.4 gm/l dimethyl POPOP = 1, 4-bis[2-(4-methyl-5-phenyloxazolyl)]-benzene
   c. Toluene to volume
3. Several microliters of activity i.e.,
   a. ^{3}H labeled toluene
   b. ^{14}C labeled toluene

PROCEDURE

1. Prepare the following samples in counting vials.

TABLE I

Sample #	^{3}H counts/min	^{14}C counts/min	mls Nitro Methane	Scintillation fluid
1	50,000	0	0	15
2	50,000	0	0.010	15
3	0	50,000	0	15
4	0	50,000	0.100	15

2. Place sample #1 in the instrument. Note: The gain should be in its normal position, as indicated in the instruction manual. A record of this position should be made.

3. Determine the endpoint position and the spectral maximum position by positioning a 5% window over them. (The endpoint position and maximum position were discussed in Experiment #2 and their meaning has not changed.) For the endpoint this is most easily accomplished by first counting with the counting window wide open, then with successive counts, increasing the value of the lower discriminator by small increments, say 5% per count until 1-2% of the original counts remain. The position of the lower discriminator is considered to be the spectral endpoint. The algorythm for determining the position of the spectral maximum for each successive gain setting will be left to the student! Proceed with the count as indicated on the following page.

TABLE II

Gain Settings in % of Normal	UNQUENCHED SAMPLES Discriminator Settings			
	Sample #1 ^{3}H		Sample #3 ^{14}C	
	Endpoint	Max	Endpoint	Max
-50%				
-40%				
-30%				
-20%				
-10%				
Normal				
+10%				
+20%				
+30%				
+40%				
+50%				

Gain Settings in % of Normal	MODERATELY QUENCHEED SAMPLES Discriminator Settings			
	Sample #2 ^{3}H		Sample #4 ^{14}C	
	Endpoint	Max	Endpoint	Max
-50%				
-40%				
-30%				
-20%				
-10%				
Normal				
+10%				
+20%				
+30%				
+40%				
+50%				

4. Plot gain vs. endpoint for all samples. Also plot gain vs. maxima for all samples.

QUESTIONS

1. List the ways in which proper gain setting enhances the count.
2. List the ways in which improper* gain setting enhances the count!
3. Outline a procedure for selecting the best gain setting.

* See C.H. Wang and D.L. Willis, "Radiotracer Methodology in Biological Science," Prentice-Hall on -- "flat spectrum counting".