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# A MANUAL ON THE MEASUREMENT OF RADIOACTIVITY

by

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#### FOREWORD

This manual was written in December, 1943, as an introduction to radioactivity measurements for chemists and others who were unfamiliar with this field. It is elementary and by no means critical, the aim being to provide fairly explicit operating instructions together with the minimum background necessary for intelligent use of the instruments and interpretation of results. It is restricted to instruments and techniques in use at the time of writing, and is further limited to those which were in use in the Section of the Metallurgical Laboratory with which the authors were associated. This section had as its primary task the development of processes for the chemical engineering scale separation of plutonium from uranium and fission product activities, and many other instruments and techniques in use in the laboratory but not applied to this problem are omitted from the manual.

In spite of these limitations and shortcomings the manual has continued to be of use for introductory purposes, and consequently is being declassified for informal distribution in its original form (corrected in March, 1944). The following comments indicate which of the material is now obsolete and what new methods and instruments are in general use.

Sample Preparation. The platinum dish and "lusteroid" techniques are now rarely used, whereas the evaporation and slurry-transfer methods have assumed greater importance. An important advance is the use of a ring of Zapon lacquer around the margin of the platinum plate, applied and dried before use, to prevent loss of samples. This is removed during the ignition process. Infrared heat lamps have proved more useful than hot-plates for sample evaporation and drying. In some cases good spreading can be achieved by adding a reagent to the solution on the plate which forms a precipitate with the carrier; this settles out uniformly during the evaporation and adheres well. Special methods, such as electroplating and sublimation, have been developed for preparing extremely thin and uniform films, but these are generally not applicable to routine assays.

Alpha Counters. The methane proportional counter has come into wide use for samples of high alpha disintegration rates and for alpha samples containing high beta backgrounds, as well as for ordinary measurements. Fast ionization chambers utilizing free-electron gases such as nitrogen and argon have also been developed for the same purpose but are not in as widespread use. The magnet chamber is obsolete, having been replaced by the two previously mentioned instruments. Similarly, the air-screen low-geometry counter, formerly used to bridge the gap between the atmospheric alpha counter and the vacuum low-geometry counter, is no longer used, since the methane and argon counters extend well into the range of the vacuum low-geometry counters. An instrument known as the alpha particle pulse analyzer, which sorts out electronically and counts separately alpha particles of different energies, is becoming of ever-increasing importance for the identification of alpha emitters, the measurement of alpha particle energies, and the analysis of mixtures of alpha active substances.

Beta and Gamma Measurements. Although only the mica end-window type of Geiger-Müller tube is described in the manual, thin-walled tubular counters are also used widely. For detection of very soft electrons, such as weak beta-rays and internal-conversion electrons, windowless Geiger-Müller counters have been developed. These are in a gas-tight enclosure which can be evacuated and filled with the counter gas after introduction of the sample. Provision is made for insertion and removal of absorbers without changing the atmosphere. For measurement of low-energy gamma radiation or of the X-radiation emitted by some radioactive substances, xenon-filled tubes are employed. A high-pressure ionization chamber filled with argon is frequently used for gamma ray measurements. A reentrant tube allowing samples to be placed in the center of the chamber gives high sensitivity, and test tubes containing samples in solution can be used directly without the necessity of preparing solid samples as described in the manual.

Statistics. The use of the term "reliable error" for the nine-tenths error has been discontinued, and the emphasis on the use of this and similar measures of the deviation other than the probable or standard error has been greatly diminished relative to the emphasis on the more conventional measures.

The list of references found at the end of this document, while by no means complete, may serve to supplement this manual with respect to its omissions and weaknesses. There are many other Metallurgical Project reports on the subject, and the published literature is extensive. The Plutonium Project Record, now in preparation, will summarize much of this information, particularly in Volumes 8 and 9.

# CONTENTS

Int	rod	uction	1
Se	ctio	n I. Alpha Counting	1
	Α.	The Pulse Ionization Chamber and Circuits	1
	в.	Operation and Care of Counters	2
•	C.	bumples	4 4 4 5 7
	D.	Backgrounds	8
	E.	Oscillograph Monitoring	8
	F.	1. Reason for the Difficulty	10 10 11 11 12
	G.	Coincidence Losses	12
	н.	Low Geometry Counter: Air-Screen Type	12
	I.	Low Geometry Counter: Vacuum Type ("Demultiplier")	13
S	ectio	on II. Measurement of Beta and Gamma Radiation	15
	Α.	General Principles	15
	В.	Geiger-Müller Counters  1. Description  2. Construction of Counter Tubes  3. Circuits  4. Operation  5. Background Corrections  6. Coincidence Corrections  7. Monitoring with Standards	16 17 18 19
	C	. Quartz-Fiber Electroscope	20
	D	. Ionization Chamber with FP54 Electrometer	2

	E.	Measurement of Beta Radiation	
		1. Sample Preparation	
		3. Interference by Gamma Radiation	
		4. Correction for Decay	
	F.	Measurement of Gamma Radiation	
		1. Sample Preparation	
		3. Counting Yield	
	~	Characterization of Activities	
	G.	Characterization of Activities	
		2. Absorption Curves	
Se	ctic	on III. Statistics and Counting	
	Α.	Reliability of Counts	
	В.	Combination of Results	
		1. Some Formulae	1
		2. Counting by Difference	
		3. Ratio of Two Counts	
	~	Packground Counting	)

# A MANUAL ON THE MEASUREMENT OF RADIOACTIVITY

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#### INTRODUCTION

The use of radioactivity measurement as an analytical technique is becoming of increasing importance. New techniques are constantly being developed and refinements in instruments and methods are continually being made. It is the purpose of this manual to describe the fundamental principles, the various instruments in use in this section, and the techniques of their use. Sufficient detail is given so that novices to the field of radiochemistry may intelligently operate the instruments and interpret the results.

Radioactivity is the phenomenon of the spontaneous disintegration of metastable atomic nuclei with the emission of energetic radiations. There are three principal kinds of radiations emitted by radioactive substances, designated as alpha rays, beta rays, and gamma rays. They differ in their physical nature, and from the point of view of their detection and measurement, they are distinguished principally by the manner in which they interact with matter to lose energy and produce ionization.

Alpha particles are helium nuclei (mass 4, charge 2) ejected from nuclei with energies of several Mev (million electron volts, the unit of nuclear energy; see Rasetti, "Elements of Nuclear Physics", p.13). They are densely ionizing and as a consequence are completely stopped by thin layers of solids, such as a piece of paper.

Beta particles are high-speed electrons with energies ranging from less than 0.1 Mev to several Mev. They are considerably more penetrating than alpha particles, but can be stopped by metal sheets of moderate thickness. Some radioelements emit positrons, which are electrons with positive charges, and are similar in other respects to negative beta rays.

Gamma rays are photons of high energies and consequently very short wavelengths. Their energies range from the X-ray region to several Mev. They are very penetrating and some can traverse several centimeters of lead.

Practically all instruments for measuring radioactivity depend on the ionization produced in a gas by the energetic nuclear particles. Two main types of instruments exist, those which respond to individual particles and those which average or integrate the effects of a number of particles. Instruments of the first type are called "counters". In general, counters have the highest sensitivity and give the most significant data, but instruments of the second type often have greater range and stability.

Instruments of either type can be used for measuring any of the various kinds of radiations. However, usage has shown that for specific applications, some instruments have definite advantages. Not all types of instruments are described in this manual, but only the ones which have proved most useful. The particular designs are not necessarily in universal use, but are the standard types used in this section.

#### SECTION I - ALPHA-COUNTING

# A. THE PULSE IONIZATION CHAMBER AND CIRCUITS.

The design of the usual type of chamber for counting alpha particles depends upon several important properties of the emitted alphas: (1) the alpha particle is very energetic and hence can ionize many molecules of air, (2) the ionization per centimeter path length (specific ionization) is very intense, so that all the ions generated by an alpha particle are found in a fairly small volume of space, and (3) because of this intense ionization, the range of an alpha is rather short—usually not more than 4 cm of air.

Because of these factors, the chamber (Figure 1) is quite small; it consists of two parallel disc-shaped electrodes, 5 cm in diameter and 8.4 mm apart. An electric field is maintained between the electrodes by an applied potential of 1250 volts, the exact voltage not being critical. The lower disc, which holds the sample, is mounted through an insulator onto a base plate. This screws by means of a breech thread into the cylindrical housing. The plate is lined up correctly by means of red indicator lines and then screwed in one-sixth of a turn; this connects the lower electrode to the 1250 V source through a wiping spring contact.

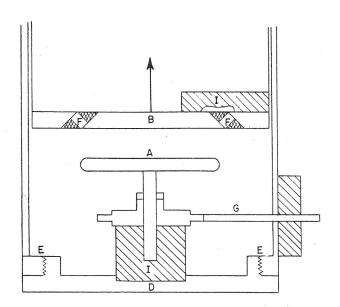


Figure 1.

- A. High voltage electrode
- B. Collecting electrode
- C. Guard ring
- D. Bottom plate
- E. Breech thread
- F. Paraffin insulation
- G. High voltage wiping contact
- H. High voltage inlet
- I. Amphenol insulator

The ions formed in the alpha-particle path are swept apart by the electric field and drawn towards the electrodes, thus causing a change in the voltage of the collecting electrode. This voltage change (or pulse), which is quite small (about 150 microvolts) is amplified 300,000 fold and then fed into a scale-of-eight circuit. For every eight pulses coming from the ionization chamber, only one emerges from the scaler and is counted on the mechanical register. (The register will not respond to a very rapid succession of pulses, but will respond if the number of pulses is cut down to one-eighth the rate). The procedure to be used in reading the register will be described below.

#### B. OPERATION AND CARE OF COUNTERS

While the chamber and circuits are designed to be used for routine counting and can be operated by personnel with no electronics training, they are not entirely foolproof and do require a certain degree of care. Certain of the vacuum tubes have very high plate voltages, which makes it desirable to allow the filaments to heat up before turning the high-voltage transformer on. In the oscillograph associated with each circuit, the electron beam reaches the screen before the sweeping mechanism has warmed up, and if the knob controlling the "On" switch of the oscillograph is turned far to the right (thus increasing the beam intensity) the electron beam subjects the center of the screen to a continuous intense bombardment; such treatment is detrimental to the screen.

The procedure used is as follows:

- 1. Turn on the "Master" switch (Type B Counter), the "Filament" switch (Type A Linear Amplifier) and the "On" oscillograph switch (the latter should not be turned beyond the switching position).
- 2. Wait about 30 seconds and turn on the "Kilovolt" switch (Type B Counter), the "Plate" switch (Linear Amplifier), and turn the intensity control of the oscillograph to the indicated position. The kilovolt meter should read 1250 + 50 volts; if not, it should be adjusted by a member of the service group. The gain control should be turned (if not already set) to the recommended value, which is posted on each amplifier.
- 3. After a few minutes, the counter is ready for use. During this interval of waiting, the oscillograph screen should be watched to see that the counter has settled down to equilibrium conditions.

To place the samples into the chamber, the base plate is unscrewed by one-sixth turn to the left. Care should be taken to keep one's fingers out of the cylinder when the breech plate is removed so as not to come in contact with the high voltage pin projecting on the inside. The sample is placed on the lower electrode, due care being taken to avoid contamination of the base plate and electrode assembly. The lower electrode is the part most commonly contaminated - usually by activity on the sample. To prevent such accidents, the lower electrode should be protected by a small circle or square of paper under the sample. Hard-finish paper rather than filter paper should be used to avoid getting fibers in the chamber. Samples are placed onto and removed from the electrode with uncontaminated forceps. It is also possible to contaminate the upper electrode by samples containing powders or samples which have not been dried thoroughly. Doubtful samples of this nature should not be put into the chamber. In all cases samples containing powders of precipitated carriers should be put into the chamber gently. Due care to avoid contamination cannot be overemphasized.

Small pieces of fuzzy material (such as hairs or fibers of paper or cloth) on a sample disc will often cause spurious discharges in the chamber, resulting in extra counts. These can usually be detected by examining the pulse shape on the oscillograph screen, and can be prevented by suitable care in preparing and introducing samples. Other types of difficulties will be discussed later under "Oscillograph Monitoring."

The base plate is screwed into the chamber cylinder and the counting started. The counting procedure is:

- 1. Turn the "Reset" switch on, then turn the "Count" switch on. Turning the "Reset" switch on sets the interpolation meter to zero and prevents pulses from passing through the scaler into the register.
  - 2. Set electric timer to zero.
- 3. Record the reading on the mechanical register; note that there are two small dials, reading in "Hundreds" and "Thousands".
  - 4. Simultaneously turn the "Reset" switch off and the timer on.
  - 5. When the count is finished, simultaneously turn both the "Count" switch and timer off.
- 6. Record the time of the count and the readings on the mechanical register and on the  $\underline{\text{interpolation}}$  meter.
  - 7. Total counts equals (net register reading x 8) plus interpolation meter reading.

When the counting is over, all the switches are turned off, including the oscillograph switch.

#### C. SAMPLES

Most of the alpha-emitting samples counted in this laboratory consist of tracer amounts of the active element ( $< 10^{-8}$ g) imbedded in much larger amounts (of the order of a mg) of "carrier", the latter being a compound which has properties similar to that of the active material.

## 1. Self-Absorption

Due to the intense ionization it creates, the alpha particle has a short range, making it impossible to use more than a small amount of carrier in a sample to be used for counting. The alphas usually encountered in the laboratory have a range of about 4 cm in air, i.e., the energy is completely dissipated after passing through 4 cm of air. It is usual to express the amount of an absorbing layer in terms of the weight per unit area, or surface density. Since the density of air at 15°C and 760 mm is 1.23 mg/cm³, an alpha having a range of 4.00 cm under the same conditions would be said to have a mass range of 4.92 mg/cm² in air. Since the carriers usually used have a mass stopping power less than one-half that of air, the mass range in the sample is greater, being approximately 10 mg/cm².

The pulse ionization chambers described are so efficient that a recordable pulse is generated when less than 10 per cent of the alpha particle's range lies between the electrodes. This does not mean, however, that it is permissible to use a sample whose surface density is, say, 9 mg/cm,² since the alpha particles coming off at an angle to the perpendicular must penetrate more than 10 mg/cm² and will thus be completely absorbed. It is true that the alpha particle causes ionization within the solid, but it is only when the ionization occurs in a gas (where the generated ions are mobile) that the ionization can be measured. For example, in a uniformly spread sample, when the surface density is equal to the range, just 50 per cent of the alphas are lost by absorption in the sample. Table I shows the amount of loss by "self-absorption" to be expected when the sample is uniformly spread on a flat platinum disc; the standard of comparison is the count that would be obtained if the sample were spread out in a layer so thin that self-absorption is negligible. Since the absorption losses quoted are approximate, and since most of the samples used in routine work are nowhere near ideally uniform, the values in the table should not be used as accurate correction factors, but simply as indications of magnitude.

Table I SELF-ABSORPTION LOSSES FOR VARIOUS SURFACE DENSITIES OF CARRIER

Calculated for polonium alpha particles—range 3.8 cm air ( $15^{\circ}$  C, 760 mm) and carrier with mass stopping power one-half of that of air.

Carrier Surface Density mg/cm <sup>2</sup>	0.05	0.1	0.2	0.3	0.5	1.0	5.0	10.0	20.0	50.0
Per cent of count lost	0.27 0.27	0.53 0.53	1.1 1.1	1.6 1.6	2.7 2.7	5.3 5.3	27 27	53 53	78 78	91 91

#### 2. Preparation of Samples

It is not feasible to mount samples directly onto the electrode; instead, counting discs of various types are used.

One of the more common types of counting disc is the flat circular plate punched out of .002 inch platinum sheet, usually  $\frac{1}{2}$  inch to 1 inch in diameter. If the amount of salt in the solution is small, the liquid containing the alpha activity is evaporated to dryness on one of these discs. Since it is not possible to manipulate the material with a micro-stirring rod, the amount of salt present should be so small that thick deposits of solid are not possible even if the residue tends to bunch. No more than .025 ml of solution should be used at a time—if more than this amount of liquid must be evaporated, it should be added in several steps. Evaporations are usually done by placing the platinum discs on a piece of asbestos sheet and placing them either on an electric heater or under an infrared lamp. During the evaporation, the surface tension of the drop decreases and the liquid tends to spread. Care must then be taken to prevent the sample from running over the edge of the disc. This difficulty is especially serious when acids are present. To avoid loss of sample and to provide for handling the disc, the edge is left free for a space of 1 to 2 mm.

When the amount of salt in the solution is too great for the straight evaporation technique, the alpha activity may be separated by precipitation with a suitable carrier. The precipitate is centrifuged, washed, centrifuged again, and the precipitate stirred into a slurry (after addition of a small amount of water) by use of either a micro-stirring rod or a fine wire platinum loop. The rotation of the loop in the precipitate is much like the action of an egg-beater, and is achieved by rolling the wire leading from the loop between the fingers. The slurry is pipetted onto the platinum disc, evaporated to a thick paste, spread smoothly over the disc with a micro-stirring rod, and evaporated to dryness. To avoid losses of solid on stirring rods or platinum loops, they should be rinsed onto the plates with a few drops of water. The evaporations of the slurry are done in the manner described above.

After drying the sample, it is important to heat it as hot as possible consistant with the volatility of the radioactive material (Po, e.g., volatilizes at  $700^{\circ}$ C). Where the radioactivity is non-volatile, it is desirable to ignite the platinum disc to red heat. Many salts precipitate as hydrates and do not lose all their water even on the hot plate; similarly, the  $\rm H_2\,SO_4$  residues from solution evaporation will not be removed except by high temperature ignition. Such ignition decreases the absorption losses and often increases the counting rate by 3 to 10 per cent.

Another type of sample plate in use is the platinum dish, which is made by raising the edges of the platinum disc about 0.5 mm. The samples are mounted in the manner described above. The flat disc is usually preferred, however, because with it the area over which the sample is spread is less critical.

Where a relatively large amount of solution (of the order of several tenths of a ml) is to be evaporated and counted, it is convenient to use 25 mm watch glasses. Because of the larger area, somewhat larger amounts of solids can be tolerated, but due to the concavity of the watch glasses, some solids tend to concentrate in the center, and must be spread with a micro-stirring rod during the last stages of the evaporation just as with platinum discs.

One of the widely used methods is the "lusteroid technique". Centrifuge tubes of lusteroid (a celluloid-like material) are prepared with flat bottoms. The alpha activity is precipitated with carrier; the precipitate is centrifuged and washed and the tube turned upside down to drain for a short period. This procedure is applicable only to flocculent types of precipitates which adhere to the bottom of the tube; for granular precipitates, it is unsatisfactory. The tube is cut around with a razor blade  $\frac{1}{12}$  inch above the bottom, the precipitate spread over the center section with a micro-stirring rod, and then evaporated to dryness. Care must be taken not to heat the lusteroid to the point of ignition. The sides are then cut away with a razor blade so that the sample is left on an almost flat disc. The precipitate must be kept away from the edges to avoid losses during the final cutting operation.

## 3. "Counting Yield" and "Relative Counting Efficiency" in the Standard Chamber

The fundamental characteristic describing the amount of a given type of alpha-emitter is the disintegration rate, usually expressed in disintegrations per minute. The number of alphas counted per minute is the

counting rate (counts/min.). The ratio of the counting rate to the disintegration rate is called the counting yield, which is determined by several variables:

$$Y = (G) \times (S) \times (V)$$

where Y = counting yield (after correction for background and coincidence losses)

G = geometry factor

S = scattering factor

V = self-absorption factor

When only comparative results with a single radioelement are needed, and all samples are prepared and counted in the same way, the value of the counting yield need not be known. However, for more accurate work, some or all of the above factors must be considered. The significance of the various factors is explained in the following:

Geometry Factor. The geometry factor is the fraction of the alpha particles emitted by the sample which start out in the direction of the sensitive volume of the counter. If, in a standard parallel plate ionization chamber, an infinitely thin sample were placed on a smooth plate and counted, the fraction counted of the total number emitted would be very close to one-half provided back-scattering is neglected; hence, the standard chamber is said to have 50 per cent geometry, and G = 0.500. The value of G for this type of counter and for the demultiplier can be calculated from geometrical considerations alone, and will soon be compared for cross checking.

Only when the sample is spread on a flat plate is G = 0.500, and with this type of chamber, 0.500 is the largest possible value of G. If the samples are mounted in a different manner, the geometry factor is less—e.g., the watch glass and platinum dish both have raised sides which cut off some of the alpha particles. The irregular surface of a lusteroid will also tend to decrease G. As a rule, the influence of type of sample mount on the value of G is not clearly defined except for flat plates, and samples other than the flat plate type are not used for absolute measurements of disintegration rates but only for relative measurements.

The geometry factor for chambers in which the sample is mounted outside of the sensitive region (magnet chamber, low geometry chambers) is always less than .500, often considerably less. Values of G must be determined by standardization, against the standard chamber. Only when G is very small (as in the "demultiplier") can it be easily calculated from geometrical considerations alone.

Stattering Factor. The value G = 0.500 implies that half of the emitted alpha particles are lost due to the fact that they are emitted in a downward direction and are absorbed in the substrate material, none of them being back-scattered. It is now known, however, that there can occur a certain amount of scattering of downward moving alpha particles back into the ionization chamber. The amount of such scattering increases with the atomic weight of the scattering material, and the back-scattering on a platinum base may be as high as 4 per cent,i.e., S may be 1.04 and Y may be as high as 0.52. For relative determinations, this factor is not important, but must be considered for accurate absolute determinations.

Self-Absorption Factor. For samples on flat platinum discs, V usually varies from .95 to .995.

Relative Counting Efficiency. An infinitely thin sample, mounted on a smooth, flat platinum plate is said to have a 100 per cent relative counting efficiency. This ideal is approached if a sample with very little carrier is evaporated onto a flat platinum plate. A sample of this type is used as a basis of comparison when considering the relative counting efficiency of other samples. They may have lower efficiencies due to decreases either in the geometry, the self-absorption, or the scattering factors.

For various reasons, the types of samples described above do not all have 100 per cent relative counting efficiencies; the counting efficiency will vary somewhat with the technique of the individual preparing the 12-209-p10-bu

sample, but the figures in Table II are typical. These values apply only when the error due to self-absorption in the carrier is not large. (See Table I).

Table II
RELATIVE COUNTING EFFICIENCY FOR VARIOUS TYPES OF SAMPLES

Sample Type	Evaporation on Flat Pt Plate	Ppt. with Carrier Mount on Flat Pt Plate	Ppt. with Carrier. Mount on Pt Dish	Evaporation on Watch Glass	Lusteroid Technique
Relative Per Cent Counting Efficiency	100	95-98	90-95	90	80-92

Several types of losses enter into the lowering of the relative counting efficiency from 100 per cent. For the watch glass and the platinum dish, the raised sides cause the solid angle within which alphas can enter the chamber to be less than 2  $\pi$  (one-half the total) and hence the geometry factor is less than 0.50. The irregular surface of the lusteroid bottoms may cause similar geometry losses. The precipitate mounted on flat platinum gives rather good results, the losses being due primarily to lack of complete carrying and slight mechanical losses in transfer. Back-scattering on materials of low atomic weight, such as glass and lusteroid, is lower than from platinum, thus decreasing the relative counting efficiency of samples mounted on such materials.

In the case of the "lusteroid technique", the large losses and large variations of efficiency with different technicians result from a different kind of phenomenon. Apparently the lusteroid softens during heating and allows some of the precipitate to "soak in", thus increasing the self-absorption. Overheating the lusteroid will increase this error. In addition, since it is not possible to ignite lusteroid or even to heat it very hot on the hot plate, the absorption losses arising from excess water of hydration may be appreciable.

Despite the variation of relative counting efficiency for different kinds of sample mounting, any of the methods will work where the ratios of the radio-activity in a number of samples is desired rather than the absolute amounts. A given technician can usually standardize his technique so that he can get reproducible results with any one of these methods. To make comparisons where several types of samples must be prepared, the relative efficiencies must be standardized by the technician making the analyses. Where many analyses must be made and where the volumes of solution to be handled are large, the lusteroid technique seems to be more convenient. Where greater precision and reproducibility is required for analyses involving precipitation with carrier, the transfer to a flat platinum plate is more desirable.

#### 4. Special Methods for Preparing Uniform Samples

For certain purposes, it is desirable to restrict the region of the platinum plate over which the activity is spread. For this purpose a lacquer called "Zapon" is used to paint a ring defining this region. After the lacquer is dry, the sample is pipetted onto the plate in the form of a solution, and is then evaporated to dryness. Solutions containing high-boiling acids such as  $H_2SO_4$  or  $HClO_4$  cannot be used, since these acids attack the Zapon. When the sample is dry, the plate is ignited, completely removing the lacquer.

Samples spread by this technique are subject to the same difficulties encountered in any evaporation technique—the solid material tends to clump in rings unless adequately dispersed by use of a micro-stirring rod

The most satisfactory method for preparing uniform samples of radio-active material has been to evaporate a water solution of the substance with a small amount of a high-boiling organic liquid which will lower the surface tension of water. The salt to be spread should be slightly soluble in the organic liquid, while the liquid should be quite soluble in water.

Thus far, in this laboratory, this technique has been successfully applied only to uranyl nitrate solutions, spread by the use of tetra-ethylene glycol (TEG). The Pt plate is first painted with Zapon as described above. After the uranium nitrate drop has been pipetted onto the plate,  $20\text{--}50\,\mu$  l of TEG is added. This causes the solution to spread over the whole confined area. Very slow heating with an infrared lamp, lasting for more than an hour, results in the deposition of a very uniform layer of powdered material. Too rapid heating causes the formation of crystals rather than a fine powder, due to the increased solubility of uranyl nitrate in the hot TEG. The slight solubility of uranyl nitrate in TEG allows the salt to remain in solution until most of the water is gone and the subsequent deposition as powder as the solvent evaporates.

#### D. BACKGROUNDS

Every substance investigated thus far has been found to contain within it small amounts of alpha-emitting radioactivity—known as the natural contamination. Not all substances are alike—Pb, e.g., is a bad offender— but even when substances of very low contamination are used for construction, an ionization chamber will always have an irreducible background of alpha counts. For small chambers, this may be as low as a small fraction of a count per minute.

A background this low is adequate for the counting done in this laboratory. Larger backgrounds are caused by the careless contamination of the interior of the chamber by brushing radioactive material onto some surface. Very small amounts of such contamination will raise the background to 2, 3, or 4 counts per minute. It is possible to use the counters with such backgrounds, but it is preferable to keep the contamination low. With due care to avoid touching the inside of the chamber by anything which may carry alpha activity, the backgrounds may be kept quite low. When the background does get high, the contamination may often be removed by wiping the lower electrode with a piece of lintless absorbent paper (removing all fuzzy hairs afterwards). If this does not suffice, call the difficulty to the attention of a member of the service group. Above all, care should be taken in putting samples into the chamber.

If the background is low and the count high, the background may be neglected for all but the most accurate work. However, if the background is greater than the statistical error of the sample count, it should be taken into account. During the interval in which backgrounds are taken, the oscillograph screen should be closely checked to see that spurious effects are not occurring (See Section I,E)—in this interval the spurious counts will stand out more clearly, not being "hidden" by the true counts.

In general, a short background count should be taken before starting the sample counts to check on the amount of contamination present. After counting the sample, if a more accurate background is needed, this count should be repeated. If the background is appreciable and the sample count is low or if the sample must be counted accurately, the background should be determined with a definitely uncontaminated blank sample plate. If many samples are to be counted, background counts should be interspersed several times between the sample counts.

For the time to be devoted to background counts, see the Statistics Section (III, C). It is usually desirable, in any case, to have a minimum background count of one or two minutes.

## E. OSCILLOGRAPH MONITORING

When the chamber and circuits are working well, when the self-absorption in the sample is small, and when there are no hairs on the electrodes or sample, the counting procedure is perfectly straightforward and accurate. However, it is desirable to check on whether these conditions are fulfilled. The chambers

and circuits are pretty well-behaved, usually giving no trouble, but on occasion they do go bad. It is rarely possible to notice spurious counts by any observation such as counting rate. For these reasons, every circuit has associated with it a monitoring oscillograph, to check on the types of pulses coming out of the linear amplifier.

With no signal of any sort coming into the oscillograph, the beam is swept horizontally, and merely appears as a smooth straight line. When the amplifier is connected to the oscillograph but no pulse is entering; the smooth line is changed into a fuzzy line centering around the same position. This fuzziness is caused by the "noise" of the amplifier. When a pulse comes in, the beam moves vertically and traces out the graph of the pulse shape as it comes from the amplifier.

When the chamber and circuits are working correctly, the pulse has a very distinctive shape. To monitor correctly, one must learn to distinguish this type of pulse from the types of pulses arising when something goes wrong.

Two factors influence the apparent shape of the pulse: (1) the height of the pulse, which is regulated by the "vertical gain" control, and (2) the width of the pulse, which is regulated by the frequency with which the beam is swept across the screen.

The base line is taken as ten divisions below the center line, and in normal operation, the beam is set in this position. Each oscillograph tube has a red line running across the screen, usually around 12 divisions above the base line—this is the "tripping-height" line. When the oscillograph is correctly adjusted, any pulse which reaches or crosses the red line will register in the scaling circuit—this selection is performed by the "pulse-height selector". The amplifier has a circuit which prevents pulses from exceeding a certain size—the "limiter" circuit—this height being about four divisions above the tripping level. In order that the pulse be of the correct height on the screen, the "vertical gain" control must be fixed at the correct setting, which is shown by an indicator line on the Dumot oscillographs. The RCA oscillograph does not need this adjustment.

When the frequency with which the beam is swept across the screen is around 100/sec, (the usual frequency setting) the pulse appears as a vertical straight line with a bright spot at the peak. If the frequency is increased, the pulse appears as in Figure 2.

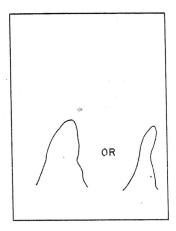


Figure 2.



Figure 3.

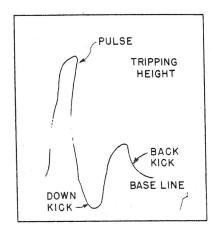


Figure 4.

- 10 - MDDC - 388

Disturbances of various sorts give characteristic patterns on the oscillograph screen. The normal fuzzy baseline showing the noise-level of the amplifier is usually from two to three divisions high; if it is any larger, it is indicated that the amplifier is working incorrectly and a member of the service group should be called to investigate. Even when the noise level is quite low, if large numbers of beta particles are emitted by the sample, the base-line of the oscillograph trace may be considerably widened, giving the same appearance as a high noise level. If the noise level comes back to a low value when the sample is removed, a high beta background is usually indicated. If the peaks of the beta "kicks" remain below the red line, the alphas may still be counted; otherwise a special magnet chamber must be used.

All of the chambers are somewhat "microphonic" although they are mounted by rubber suspensions to minimize this difficulty. Loud noises or sharp vibrations will cause the chamber to vibrate and deliver spurious pulses. On the oscillograph screen, a large number of regularly spaced pulses appear and last as long as the sounds continue. If these pulses go over the red tripping line, they will pass into the scaler circuit and be counted. Nothing can be done about this difficulty except to discard a count in which such an event has occurred. If the chamber seems to be unduly microphonic, i.e., if it reacts to minor sounds such as talking, call a member of the service group.

Hairs in the chamber give an unmistakably characteristic pulse. Under the influence of high voltage, the hairs stand up and cause point discharges. Because of the action of the limiter circuit, they appear as in Figure 3. Conversely, a pulse of this shape invariably means hairs in the chamber. The plate should be taken out and hairs removed from the lower electrode and sample.

It will sometimes be found that some of the pulses, while well above the noise level, fall short of the red line and do not register. These are known as "short kicks". They are due to alpha particles which have only a small portion of their range in the chamber. When a sample is mounted with a great deal of absorbing material, "short kicks" are more evident, although most of the counts lost due to self-absorption are never seen at all, since they are totally absorbed. "Short kicks" are also quite noticable in lusteroid samples due to the "sinking in" effect and the usually irregular surfaces. Samples mounted on platinum invariably show "short kicks" since the back-scattering on platinum is relatively high and the residual range of back-scattered alphas is usually quite small.

Sometimes peculiar pulses occur—short and wide with jagged peaks. These will occur even with no sample present and will be found during the background count. The "back kick" (See Figure 4) under certain conditions becomes so large that it may cross the tripping height line and cause a spurious count. Both of these difficulties are due to incorrect action of the circuits and should be called to the attention of a member of the service group.

# F. COUNTING OVER HIGH BETA BACKGROUND

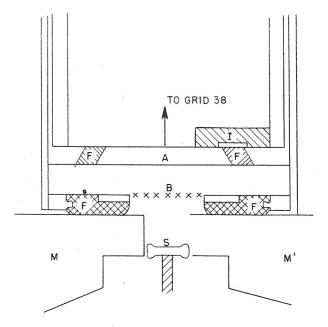
#### 1. Reason for the Difficulty

The height of the pulse produced by an alpha particle in a standard chamber is roughly proportional to the number of ion pairs formed. The number of ion pairs formed by a beta particle in such a chamber is about one-thousandth of that produced by an alpha particle. As a result, a beta pulse is considerably smaller, and is normally not recorded.

However, if the beta activity in the sample is too high, statistical variations in the beta emission rate will cause beta pulses to "pile up" on top of one another and record. For a single sample, the upper limit of tolerable beta activity for the standard chamber is roughly  $3 \times 10^6$  beta disintegrations per minute. This figure varies with the energy of the beta and is lower for smaller energies. If the sample contains slightly more than this tolerance beta level, it is sometimes possible to get an approximate count by reducing the gain setting one or two points (coarse gain control) from the recommended value. Depending upon the nature of the sample, this procedure may reduce alpha blank (i.e., a sample with the same alpha rate but with no

beta activity) at the recommended and at the reduced gain settings.

It is sometimes desirable to count alpha samples over much higher beta backgrounds. For this purpose, a special "magnet chamber" has been developed, a description of which follows.



- A. Collecting electrode
- B. High voltage screen
- C. High voltage electrode
- F. Paraffin support and insulator
- M, M'. Poles of permanent magnet
- S. Sample

Figure 5.

The radiation from the sample is allowed to travel a distance of 1 to 2 cm through a strong magnetic field (6,000 gauss). This causes the beta particles to be bent around sharply, while not appreciably deflecting the much heavier alphas. As a result, only the alphas reach and penetrate the openings in the HV screen to enter the ionization chamber where they produce pulses in the usual way.

With this type of chamber, it was found that a sample emitting 1.4 x 10<sup>9</sup> beta disintegrations/min could be tolerated, the sample being Sr<sup>89</sup>, (upper energy limit approximately 1.7 Mev). Since the magnetic field bender out lower energy betas more easily, softer beta emission can be tolerated in even higher amounts.

The disadvantages of this method are primarily:

- (a) The geometry factor is low (approximately 10%). This results in the difficulty that the position of the sample is critical, and that it must be spread evenly.
- (b) While traversing the magnetic field, the alphas extend some of their range. The sample must, therefore, not be too thick, or some of the alphas will never reach the chamber.
- (c) The distance between the magnet pole pieces is 1.5 cm. As the sample must be introduced between them, its size is limited.

#### 3. Preparation of Samples

To obtain accurate results, a sample should be spread uniformly over a circle of 9 mm diameter on special size platinum plates. The cutter for these plates will be furnished on request. The sample thickness should be kept below  $0.3~\text{mg/cm}^2$ .

## 4. Operation of Magnet Counter

After having prepared the sample in the appropriate way, open the door of the box surrounding the chamber and magnet, and also the door of the brass box surrounding the sample holder. Slide out the sample holder, and place the sample on it with uncontaminated forceps. Slide it back all the way between the two run-ways. Close both doors.

The counter is turned on and off by following exactly the same procedure as for the standard counter. To determine the disintegration rate of the sample, multiply the counts/min by the posted conversion factor (approximately 10).

## G. COINCIDENCE LOSSES.

The pulses in the ionization chamber last about 10<sup>-5</sup> minutes, and if they all came in at equal intervals, the chambers could handle many thousands of pulses per minute. Unfortunately, alphas are emitted at random intervals, sometimes following each other within a period of less than 10<sup>-5</sup> minutes. Two alphas arriving in the chamber in an interval 10<sup>-5</sup> minutes will count as one. Such losses—called coincidence losses—will increase with the counting rate. The normal counter will have losses of about 0.8 per cent per thousand counts per minute.

Table III
COINCIDENCE LOSSES FOR A TYPICAL COUNTER

Counting Rate				
(counts/min.)	500	1000	2500	5000
Per Cent Loss	0.4%	0.8%	2.0%	4.0%
True Count	502	1008	2550	5200

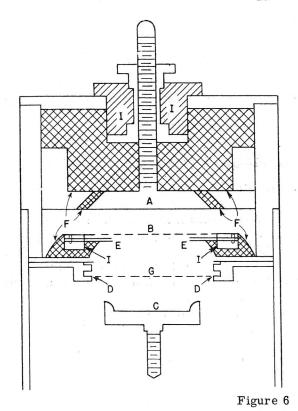
When only relative values are needed it is unnecessary to made a coincidence correction, even at high counting rates, provided the results to be compared are of about the same counting rate, and provided the same (or equivalent) counter is used. However, when rates of different magnitudes must be compared, the counting losses must be considered.

# H. LOW GEOMETRY COUNTER: AIR-SCREEN TYPE.

When samples with very high disintegration rates must be counted, it is not feasible to use the ordinary parallel plate chambers. A different type of chamber is in regular use for this purpose. As in the magnet chamber, the sample is placed below the high-voltage electrode, which is a screen.

The sample, which must be mounted on a platinum dish 16 mm or less in diameter, is placed in the copper dish lying in the housing. The dish is placed on the moveable sample holder which is then moved upwards, by means of a knurled wheel, to the counting position. The counting procedure is the same as for the standard chamber. After the counting is over, the sample holder is lowered, the sample removed, and the copper dish replaced on the holder.

Since the only alphas counted are those emerging within an angle of about 30 degrees vertical, the use of a flat sample plate is not necessary. In fact, it is more convenient to handle a platinum dish with raised edges. A punch for making such dishes is available.



- A. Collecting electrode
- B. High voltage electrode (screen)
- C. Sample holder
- D. Slots for supporting screens
- E. Collimating ring
- F. Paraffin insulator
- G. Screen
- I. Amphenol insulator

The "geometry factor" is about 10 per cent as compared to the 50 per cent of the ordinary chamber. To further reduce the counting rate it is possible to use screens with various transmission factors. The calibration on the chamber gives geometry factors ranging from 1/9.35 to 1/6420, where the total number of disintegrations/min (i.e., total number of alphas emitted/min, in the sample) is calculated by dividing the counting rate by the appropriate geometry factor. Thus, for a counting rate of 2000 counts/min, it is possible to count a sample emitting about 13 million alphas per minute.

$$\frac{2000}{1\sqrt{6420}}$$
 = 2000 x 6420 = 12,840,000 disintegrations/min)

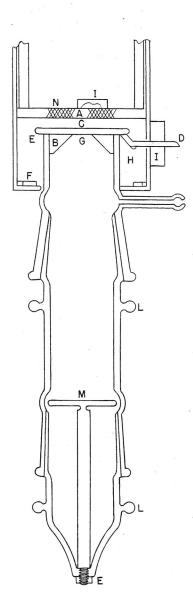
# 1. Low Geometry Counter: Vacuum Type ("Demultiplier")

For accurate counting of very active alpha samples there has been developed a special type of chamber with a geometry even lower than the air-screen low geometry chamber. This instrument is designed (Figure 7) so that the sensitive part of the chamber subtends a very small solid angle at the sample. An important feature of this type of counter is that the geometry may be accurately calculated from the dimensions. The particles travel through an evacuated glass cylinder, pass through a circular hole covered by a thin mica window (1.6 mg/cm²), and then enter the chamber at atmospheric pressure, where they are counted. The distance, D, can be varied in steps by inserting different glass extensions between the sample holder and the chamber. At the present time, distances of 5.00 cm,12.3 cm, 19.2 cm, and 26.5 cm are available; the diameter, d, is 1.016 cm. For point sources, the geometry factor is simply calculated from the solid angle subtended by the mica window at the source:

$$G = \frac{\pi d^{2/4}}{4 \pi D^{2}} = \frac{1}{16} \left(\frac{d}{D}\right)^{2}$$

When the samples are spread over an area approximately equal to that of the mica window (1 cm in diameter), the geometry factor is:

$$G = \left[\frac{1}{16} \left(\frac{d}{D}\right)^{2} + 1 - \frac{3}{8} \left(\frac{d}{D}\right)^{2} + \frac{25}{128} \left(\frac{d}{D}\right)^{4} + \dots\right]$$



- A. Collecting electrode
- B. High voltage electrode
- C. Mica window
- D. High voltage lead
- E. Cement seal
- F. Breech thread
- G. Defining aperture
- H. Glass insulator
- I. Amphenol insulator
- J. To pump
- K. Glass spacer
- L. Glass ears (to hold sections together with rubber band)
- M. Sample holder
- N. Paraffin

Figure 7.

In Table IV are shown the values of G for the various positions, both for point and extended sources (spread over an area approximately 1 cm, in diameter). Also shown are values of  $F - \frac{1}{G}$  for both cases, F being the number by which N (number of counts) is multiplied to determine the number of disintegrations. Because of occasional necessary changes in the instrument, the dimensions at any particular time may differ from the values corresponding to the table. The service group should be consulted on this point.

Table IV

	D	Point G	Source F	Extended G	Source F	$\frac{\Delta G}{\Delta D}$ (in cm <sup>-1</sup> )
Position 1	5.00 cm	25.8 x 10 <sup>-4</sup>	387.6	25.4 x 10 <sup>-4</sup>	393.7	10.3 -
Position 2	12.3 cm	$4.26 \times 10^{-4}$	2345	$4.25 \times 10^{-4}$	2350	• (
Position 3	19.2 cm	$1.748 \times 10^{-4}$	5720	$1.746 \times 10^{-4}$	5727	.168 ג
Position 4	26.5 cm	.919 x 10 <sup>-4</sup>	10880	.918 x 10 <sup>-4</sup>	10890	$.069 \times 10^{-4}$

Appropriate corrections should be made if D is varied because of the thickness of the plate on which the sample is mounted. By simple differentiation, we get:

$$\Delta G = -\frac{C^2}{8} \quad \frac{1}{D^3} \quad \Delta D$$

or 
$$\Delta G/\Delta D = -.1290/D^3$$

The negative sign implies that G decreases as D increases. Values for  $\Delta G/\Delta$  D are tabulated in Table IV. Because the sample plate thickness always decreases D, the correction increases G and hence decreases F.

This counter is not in regular laboratory use. A member of the service group should be consulted when it is desired to use the instrument.

#### SECTION II - MEASUREMENT OF BETA AND GAMMA RADIATION

#### A. GENERAL PRINCIPLES

A number of instruments are in use for the measurement of beta and gamma radiation. The most important are the Geiger-Müller counter, the Lauritzen quartz-fiber electroscope, and the ionization chamber used with a vacuum tube electrometer.

Beta particles are considerably more penetrating than alpha particles, and therefore, the use of windows in the detector is permissible in most cases. The beta rays of some radioelements can penetrate windows or walls over  $100~\text{mg/cm}^2$  in thickness, but since the penetrability increases with decreasing window thickness and in many cases the electron ranges are only a few  $\text{mg/cm}^2$ , it is desirable to use the thinnest possible walls or windows.

The sensitive part of nearly all detectors of beta or gamma rays is a volume filled with a gas, in which the production of ionization by fast electrons furnishes the means of detection. The ion density along the path of an electron in a gas depends on the electron energy, the nature of the gas and the pressure. As a typical example, a beta particle of a few tenths of a Mev will produce about 5 to 10 ion pairs per centimeter of its path in argon at 10 cm Hg pressure. Since these conditions are representative of those involved in

- 16 - MDDC - 388

Geiger-Müller counters, and since the dimensions are of the order of centimeters, it is evident that practically all beta rays which enter the sensitive part of the counter will produce one or more ion pairs within it. Only those passing through a corner, so that the path length is less than a few millimeters, will have an appreciable chance of getting out without producing an ion pair.

Unlike beta particles, gamma rays do not lose their energy gradually, but are generally stopped by one or a very few collisions with electrons. These electrons acquire a large part of the energy and it is by the ionization they produce that gamma rays are detected. Gamma rays are much more penetrating than beta rays, and they do not have definite ranges, but are absorbed in an exponential fashion. For example, gamma radiation of 1 Mev is half absorbed by 10 gm/cm² of Pb. Because of the resulting low ion density along their tracks, a large fraction of the gamma rays from a source may pass completely through a detector without leaving a single ion within its sensitive region. For efficient measurement of gamma radiation it is necessary to divert a large fraction of the energy of the quanta to that of electrons by using gas at fairly high pressures, by using thick walls of a fairly heavy element, or by placing such a layer in front of the window to act as a "radiator".

# B. GEIGER-MÜLLER COUNTERS

## 1. Description

The Geiger-Müller counter tube consists of a thin wire extending along the axis of a conducting cylinder. Between the two is a gas, generally at a pressure of several centimeters, and across the two is applied an electric potential of the order of a thousand volts with the wire positive. The gas pressure and the potential are adjusted so that in the absence of ions in the gas no discharge occurs, but if one or more ion pairs are formed in the sensitive volume, a relatively large flow of current takes place. The mechanics are roughly as follows. The negative ions, which are electrons, are attracted to the positively charged wire. Close to the wire the electrical field strength is strong enough to cause the electrons to acquire sufficient velocities between collisions to ionize the gas molecules, thereby producing additional electrons. Each of these electrons can similarly produce additional ionization, so that a large number of electrons reach the wire for the original electron formed in the gas. If the potential is not too great, the discharge stops at this point. Such a counter is called a proportional counter (also called a Geiger-Klemperer counter) because the size of the discharge is roughly proportional to the number of ion pairs originally produced in the gas. If, however, the potential is somewhat higher, the electrons on reaching the wire will have sufficient energy to eject photons, probably of ultra-violet wavelengths, and these photons can release additional electrons by photo-electric processes in the gas or wall of the counter. This causes the discharge to spread along the full length of the counter. All this takes place in a very short time, of the order of 10-7 seconds. The process then ceases because the positive ions, being very slow relative to electrons, accumulate around the wire in a sheath and by their space charge reduce the field strength to such a low value that electron multiplication can no longer occur. Every discharge thus builds up to a nearly constant size, regardless of the original number of ions produced. Such a counter is called a Geiger-Müller counter, and has the advantage of giving uniform pulses of such large size that very little amplification is necessary to detect and record them by electrical means.

The operating voltages of a particular tube are determined by counting a constant radioactive source in a fixed position at various voltages and making a plot of counting rate versus voltage. Such a plot of typical counter tube, called the "characteristic curve", is shown in Figure 8. The flat part of the curve is called the "plateau", and it is usually best to operate the counter on the flattest part of the plateau in order to minimize the effect of fluctuations of the voltage supply.

#### 2. Construction of Counter Tubes

The tubes in most general use for radio-chemistry on the project are of the end-window type. The standard design is shown in the accompanying illustration. The cathode is a flanged metal cylinder, usually of copper, brass, or steel. To the unflanged end is attached a glass dome containing a stopcock and through

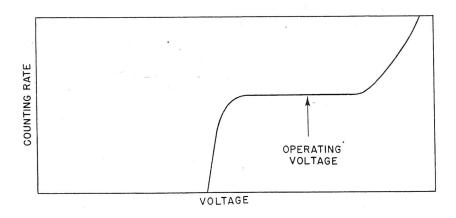


Figure 8.

which extends the anode, which is a thin tungsten wire. A thin sheet of mica extends across the other end, being waxed onto the flange. The wire extends down the axis of the cylinder almost to the window and contains on its free end a glass bead to prevent corona discharge. The tubes are evacuated and filled with a mixture of 90 per cent argon and 10 per cent ethyl alcohol to a total pressure of 10 centimeters. Samples are placed near the mica window which has a thickness generally between 3 and 6 mg/cm<sup>2</sup>. A mounting is provided with slots into which cards of a standard size  $(3\frac{1}{4})$  by  $2\frac{1}{2}$  containing samples, etc., can be inserted at several standard distances from the window. The tube and its mounting are placed in a lead housing having walls two inches thick, in order to reduce the background effect caused by cosmic rays and radioactive substances in the laboratory. The counter tube and its mounting are shown schemetically in Figure 9.

## 3. Circuits

The part of the electrical circuit directly associated with the counter tube depends on the nature of the tube. There is a fundamental distinction between self-quenching counters and those which require some auxiliary means of quenching the discharge.

Most tubes containing organic vapors are self-quenching; i.e., after a discharge the tube returns rapidly to its normal non-conduction state. It is only necessary to have a relatively low resistance (  $\sim 10^6$  ohms) between the high voltage supply and the wire so that the discharge will create a voltage pulse. It is necessary to amplify this pulse somewhat.

With many tubes, however, the discharge does not cease spontaneously, but continues indefinitely, probably because of the liberation of additional electrons from the wall under bombardment by positive ions. With such tubes, some external means must be used to lower the wire voltage below the value necessary for electron multiplication until all of the ions have been swept out of the counter.

This may be done by use of an electronic quenching circuit, such as the Neher-Harper arrangement, by which the high voltage on the wire is automatically lowered until the ion collection is complete, and then rapidly restored. This arrangement also amplifies the voltage of the pulse.

Self-quenching counters are faster than externally-quenched counters and are therefore to be preferred, but since it is somewhat more difficult to make reliable self-quenching tubes, the Neher-Harper circuit has been in general use in this laboratory.

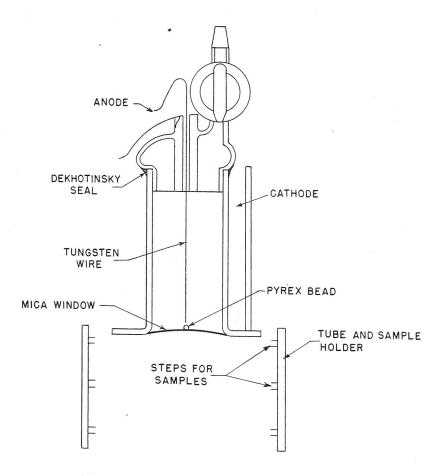


Figure 9.

The amplified pulses are equalized, scaled and recorded in somewhat the same manner as is done in the alpha-counters. However, stabilization of the high-voltage supply is more important in the case of Geiger-Müller counters than of alpha counters.

# 4. Operation

The procedure for taking a measurement with a Geiger-Müller counter is the following.

- 1. Turn on the "Master" switch and then after waiting one minute, the "Kilovolt" switch.
- 2. Note high voltage reading and compare with the operating voltage indicated on the counter housing. If necessary, readjust the voltage to the indicated value (ask a member of the maintenance group to explain how this is done.)
- 3. Place sample to be counted in the desired position below the counter tube. If there is a possibility that the sample is very active, it is well to have the "Count" switch in the ON position while bringing up the sample. If the recorder receives over 50 pulses per second or begins to jam, the sample should be removed immediately, as the counter tube may be damaged by such high counting rates.
- 4. With the "Count" switch OFF, turn the "Reset" switch ON and then OFF to clear the interpolation meter. Record the register reading and set the timer to zero.

12-209-p22-bu

- 5. Simultaneously turn on the count switch and the timer.
- 6. At the end of the desired time interval, simultaneously turn off the count switch and the timer. Calculate the counting rate as described under alpha-counting.

## 5. Background Corrections

The background of the counter must be subtracted from the recorded rate to give the true counting rate. This is determined by taking a count with no sample in the counter. The backgrounds of the counters in this laboratory are around 25 counts per minute. This is due mainly to cosmic radiation penetrating the lead shield, but may come in part from radioactive substance in or near the counter. The weaker the measured radioactivities, or the greater the desired accuracy, the more exactly the background rate must be known. For highest accuracy, backgrounds should be taken with dummy samples in place to duplicate the geometry as closely as possible. Normally, however, a count on the empty housing will suffice.

Care should be exercised in keeping the counter on its mounting from becoming contaminated with radioactive substances, as contamination will increase the background and thus decrease the accuracy of counts. If the background is appreciably higher than the above value, the source of activity should be located and removed. Covering the floor of the lead housing with paper may facilitate this.

#### 6. Coincidence Corrections

At high counting rates, some counts are lost because of the finite resolving power of the counter. If the interval between two particles arriving at the counter is less than the resolving time of the counter, they will be counted as only one. The resulting loss is called the coincidence loss. The fraction of counts lost increases as the counting rate increases, and for accurate work or at high rates, a correction must be applied. With the Geiger-Müller counters in use in this laboratory, the fractional correction is too a rough approximation proportional to the recorded counting rate. With Neher-Harper quenching the correction is about 1.5 per cent per 1000 c/m, and with self-quenching, the correction is about 0.5 per cent per 1000 c/m. The correction is made in exactly the same manner as with alpha counters. Examples are given in the table below:

	Table	V	Table VI			
1	Neher-Harper Queno (1.5%/1000 c/m	_	44			
Recorded	%	Corrected	Recorded	%	Corrected	
Rate	Correction	Rate	Rate	Correction	Rate	
500	0.75	504	500	0.25	501	
1,000	1.5	1,015	1,000	0.5	1,005	
2,000	3.0	2,060	2,000	1.0	2,020	
5,000	7.5	5,375	5,000	2.5	5,125	
10,000	15.0	11,500	10,000	5.0	10,500	
20,000	30.0	26,000	20,000	10.0	22,000	

For more accurate work, the correction at different counting rates should be determined by an impirical method, and a plot of correction vs. recorded rate made on log-log paper. From such a plot the correction can be read directly and added to the recorded rate. Such plots may be provided for some counters.

The background should be subtracted after applying the correction for coincidence loss.

## 7. Monitoring with Standards

When beta-ray standards of constant radioactive strength are counted many times with a given counter, there are observed day-to-day fluctuations which are greater than those expected from statistical considerations. The cause of this lack of constancy is not yet known. When very accurate work is to be done with Geiger-Müller counters, the results should be compared with a standard and corrected by a factor which will bring the standard count to its normal value. Ordinarily this precaution need not be taken, especially when only comparative values of samples counted the same day are required.

## C. QUARTZ-FIBER ELECTROSCOPE

The Lauritzen quartz-fiber electroscope is commonly used for measuring radioactivities because of its simplicity, stability, and relatively high sensitivity. The electroscope consists of a flexible quartz fiber fixed at one end and extending parallel to a stiff quartz rod. The whole system is gold-plated to make it conducting, and is highly insulated from the rest of the apparatus. Surrounding the wire is a cylindrical ionization chamber 3 inches long and  $2\frac{1}{4}$  inches in diameter made of aluminum and filled with a gas, usually air at atmospheric pressure. The bottom of the cylinder is usually cut out and covered with a thin aluminum foil to permit the penetration of soft radiations. A series of slots for holding samples, identical with those of the Geiger-Müller counter mounting, is located beneath the electroscope window.

The quartz system can be charged to 100-200 volts by a button which momentarily makes electrical contact with a source of DC potential. A vacuum tube rectifier operating on 110 V AC is used to supply this potential; batteries, or friction devices, though less convenient, can be used. The charge causes the fiber to bend away from the rod due to electrostatic repulsion, and its position is indicated by means of a shorter fiber fastened perpendicularly to the end of the long flexible fiber. The short fiber can be viewed by a microscope containing a graduated scale in its field for reading its position. The scale appears in Figure 10

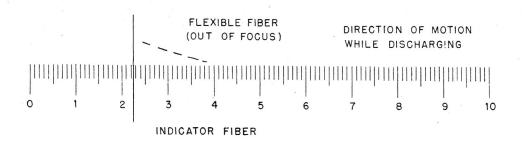


Figure 10.

On standing, the charge will leak off the fiber system at a very slow rate, partly because of the finite conductivity of the insulator, and partly because of the formation of ions in the gas within the cylinder by cosmic rays, etc. If, however, a radioactive source is brought near the chamber, the increased ionization will cause the charge to be diminished more rapidly, and the fiber will be observed to move across the scale at a rate dependent on the intensity of the radioactivity.

To take a measurement, the sample is put in place and the fiber charged by pressing in the red button and, if necessary, adjusting the voltage control of the charger until the fiber is at the zero end of the scale. The button is released, and with a stopwatch the time required for the indicator to pass two definite positions is measured. Because of non-linearity of the scale throughout its whole length, it is best to use only a portion of the scale. It is customary to note the time required for the indicator to drift from 10 to 50 i.e. over 40 divisions (divisions refer to the small intervals). However, if the rate of drift is so slow that this

12-209-p25-bu

would take more than a few minutes, a shorter interval may be used. Because of irregularities in the motion of the fiber immediately after charging, the initial setting must be somewhat to the left of the 10 division mark so that the fiber will drift a few divisions, or for several seconds, before crossing the starting mark.

Activities are reported in terms of divisions per second (div/sec.). The normal background is about 0.003 div/sec. The range of linear response extends to about 3 div/sec.

#### D. IONIZATION CHAMBER WITH FP54 ELECTROMETER

An ionization chamber used in connection with a vacuum tube electrometer, such as the circuit employing the FP54 tube, has a sensitivity intermediate between that of the quartz-fiber electroscope and the Geiger-Müller counter. Its great value is its large range of linear response and the high intensity of samples which can be measured with it.

The ionization chamber is shown in the following diagram:

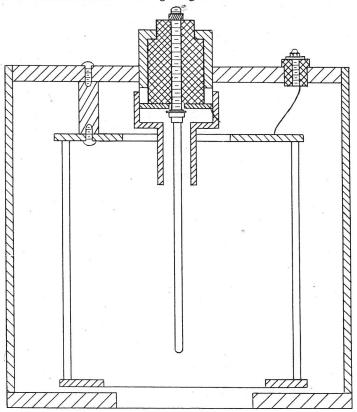


Figure 11.

The sensitive volume is between the rod and the surrounding cylinder. The cylinder has a cage-like construction to decrease its surface area and hence the natural radioactive contamination, particularly of alpha emitters. A potential of -450 V is applied to the cage and the highly insulated center rod is the collecting electrode. The ionization current to the rod flows to ground through a high resistance, and the resultant voltage across the resistor is measured by the FP54 circuit. For electrical shielding the ionization chamber is surrounded by a grounded casing. Across the bottom of the shield and of the high-voltage electrode are very thin aluminum foils ( $\sim 0.1 \text{ mg/cm}^2$ ) which act as a transparent windows to all but the very weakest electrons. The gas is air at atmospheric pressure. For high gamma ray sensitivity a chamber with thick walls and high-pressure gas, usually  $N_2$ , A, or Freon (CF<sub>2</sub> Cl<sub>2</sub>), is used.

For holding samples a series of slots is provided beneath the window, exactly as in the case of the Geiger-Műller counter and the quartz-fiber electroscope.

Because of the difficulty of operation of the FP54 circuit, the technique of its use is not described here. Measurements of samples will be made by members of the instruments group.

# E. MEASUREMENT OF BETA RADIATION

# 1. Sample Preparation

Because of the greater penetrating power of beta and gamma rays, the elaborate precautions necessary when preparing alpha samples for counting are not necessary when only beta and gamma counts are to be made. In particular, more solid matter can be tolerated, the limits being determined by the energy of the electrons. If the radiations consists of conversion electrons or soft beta rays of less than 0.1 Mev maximum energy, the solid material should be limited to one or two milligrams. For beta emitters of maximum energy of the order of 1 Mev, however, 10 to 20 mg of solids will not appreciably lower the count by selfabsorption, and more may be tolerated if corrections are made for this effect, as described below. Because the diameter of the counter window is one inch, and the counting yield for point sources drops off for positions more than a few millimeters from the axis of the counter cylinder, samples should not be more than 2 or 3 centimeters in diameter. If the sample diameter is greater than one centimeter, it is important that the distribution of material be uniform, or at least the same for each sample.

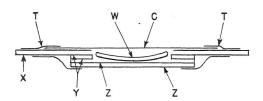
All samples should be mounted in the center of cards  $2\frac{1}{2}$  by  $3\frac{1}{4}$  inches, so that they can be inserted in the slots of the counter mounting and thus be properly centered. Unless very soft radiations are to be counted, it is generally desirable to cover the samples with a thin layer of cellophane or Scotch tape to prevent contamination of the counter and its mounting. This is very important.

The various techniques of preparing samples for beta activity measurement are described below:

One-inch watch glasses are convenient for holding samples. For assay of solutions, amounts up to 1 ml may be pipetted directly into the watch glass and evaporated to dryness in an oven at  $110^{\circ}\,\mathrm{C}$ , on an electric heater covered by an asbestos sheet, or under an infrared lamp. When using the electric heater, the temperature must not be high enough to crack the glass. The amount of solids must not be too great, as creeping over the edges may occur. The distribution of the residue over the surface of the watch glass varies for different types of solutions. In some cases even spreading results, in others a ring of solid is formed, whereas in still others the solid concentrates in the center. If a uniform procedure is followed, consistent results will usually be obtained. However, when very soft electrons are to be counted, or when the same sample is to be used also for alpha counting, it may be necessary to moisten the residue, spread it with a micro stirring rod, and re-evaporate.

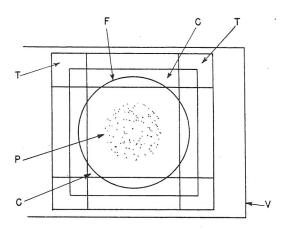
The above procedure is applicable when one ml or less contains sufficient activity for a count, and the amount taken contains so little solid residue that no self-absorption difficulties result. When the activity of larger volumes must be determined, or when the radioelement must be separated from inert solids or interfering radioelements, the desired element may be precipitated from the solution, adding sufficient carrier if necessary, and the precipitate separated and transferred to the watch glass. Separation is done best by centrifuging. The precipitate is dissolved or slurried in a few drops of water and transferred with a semi-micro pipet or a glass tube drawn to a point at one end and equipped with a rubber bulb on the other. Watch glasses may be supported for counting by means of an arrangement such as that illustrated in Figure 12.

Although the one-inch watch glass is the most convenient size, larger sizes may be used in some cases when larger volumes of solution must be evaporated. 10-ml ashing capsules, made of Coors porcelain, are also convenient. The latter are particularly useful when organic or volatile solid matter is present and can be removed by ignition without loss of the desired radioelement. Reproducibility of results may be some-



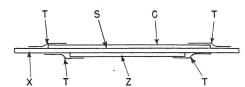
- W. Watch glass containing sample
- X. Card 2 1/2" x 3 1/4" with 1-1/16" hole
  Y. Card 1-3/4" x 1-3/4" with 1-1/16" hole
- Z. Card 1-3/4" x 1-3/4"
- Cellophane
- Scotch tape

Figure 12. Mounting of samples on watch glasses for counting.



- P. Precipitate
- F. Filter paper
- V. Card 2 1/2 x 3 1/2"
- C. Cellophane
- T. Scotch tape

Figure 13. Mounting of filtered precipitate for counting.



- X. Card 2 1/2 x 3 1/4" with 1-1/16" hole
- Z. Card 1-3/4" x 1-3/4"
- C. Cellophane
- T. Scotch tape

Figure 14. Mounting of powders for counting.

what more difficult to obtain under these conditions and should be carefully checked.

For smaller volumes of solution or precipitates of small bulk, platinum plates 1 to 3 cm in diameter may be used. Not more than 25 or 50 ml of solution should be placed on a disc at one time, or it may run off the edge when heated.

The lusteroid technique, described under alpha counting, can also be used for beta counting.

The radioelement may be precipitated with carrier and filtered with suction onto a flat filter paper on a small Hirsch or Buchner funnel. Care must be taken to deposit the precipitate uniformly and to prevent any from being sucked around the edges of the paper. The use of a second paper under the collecting sheet may reduce the tendency of fine precipitates to pass through the first paper. The paper and the precipitate are allowed to dry, spread flat on a standard size card by a cellophane covering, and sealed by means of Scotch tape around the edges of the cellophane, as shown in Figure 13.

In some cases small amounts of solid materials may be weighed directly into watch glasses, onto pieces of paper, or into a hole in a card which is backed by another card, as shown in Figure 14.

# 2. Counting Yield

The ratio between counting rate and disintegration rate is defined as the counting yield. For a given counter and geometrical arrangement the counting yield is not the same for all beta emitters, but varies with such factors as energies of radiations. If a given radioelement emits more than one radiation, or disintegrates by alternate modes, it is necessary when referring to counting yields to specify whether the counting yield for a particular radiation or for the radioelement as a whole is meant.

For a beta emitter the counting yield is the product of five separate factors:

$$Y = (G) \times (S) \times (I) \times (A) \times (V)$$

where Y = counting yield

G = geometry factor

S = scattering factor

I = ionization probability

A = absorption factor

V = self-absorption factor

When only comparative results with a single radioelement are needed, and all samples are prepared and counted in the same way, the value of the counting yield need not be known. However, for more accurate work, some or all of the above factors must be considered. The significance of the various factors is explained in the following:

Geometry Factor. The geometry factor is the fraction of the beta particles emitted by the sample which start out in the direction of the sensitive volume of the counter. For a given counter this depends on the size and position of the sample. The values of this factor for the three shelf positions of the standard Geiger-Müller counter mounting, for small samples centered with respect to the axis of the tube, have been determined as follows:

Table VII

Step	Distance From Counter Window	Geometry Factor
II II	0.2 cm 1.8 cm 3.4 cm	30% 10% 3%

The factor is somewhat lower for larger samples. The factor will also be slightly different if the sample is not even with the upper surface of a card fitting the slot.

Scattering Factor. The factor by which the counting rate is increased by scattering or reflection into the direction of the counter of particles originally directed elsewhere is called the scattering factor. Scattering may be caused by the sample backing, the counter mounting, the housing walls, etc. Accurate measurements

of scattering factors have not been made, but it is known that materials of high atomic number scatter electrons more effectively than light materials. Thus if a sample is spread on a platinum plate the scattering factor may be as much as 1.5. Scattering can be minimumized by using only light materials such as glass, paper, or cardboard for mounting the samples.

Ionization Probability. This is the average probability that an electron which enters the sensitive volume of the counter will cause ionization in the gas, instead of passing completely through it without making a single ionizing collision. As explained above, this probability is very close to unity for the type of Geiger-Müller counters in common use.

Absorption Factor. The absorption factor is the fraction of the beta particles which can penetrate the counter window and the air and other materials between the sample and the window. For a given arrangement the factor increases with increasing electron energy, and approaches unity for high-energy beta particles and small thickness of absorbing matter. For a given radioelement the factor is usually determined by taking an absorption curve as described below, and extrapolating the curve back to zero absorber thickness. The absorption factor is then the ratio of the counting rate under the experimental conditions to the extrapolated rate for no absorption.

Self-Absorption Factor. When thick samples are used for counting, some beta particles may be absorbed by the sample itself. The ratio of counting rate with a given sample to the rate which would be obtained if the activity were spread over the same area in a very thin layer is called the self-absorption factor. When the counting yield must be known, or when comparisons must be made between samples containing different amounts of carrier or other solid matter, the self-absorption factor must be determined as a function of sample weight. This may be done by preparing a series of samples each of which contains the same amount of radioactivity but different amounts of solid. The counting rate is plotted against the amount of solid and a smooth curve drawn through the points. Such a curve may appear as follows:

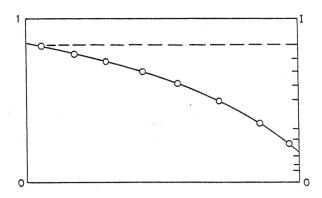


Figure 15.

For each weight, the ratio between the counting rate and the extrapolated rate for zero solid is the self-absorption factor. If a plot of this factor against sample weight is made (as indicated by the ordinate on the right-hand side of the above figure), the factor can be read off for each sample and the count corrected by dividing it by the factor.

An alternate method of determining self-absorption factors is to prepare a series of samples as above, using different amounts of solid having a constant specific activity. The amount of radioactivity will then be proportional to the amount of solid. To obtain the curve as shown above, one must plot counts/min/mg instead of counts/min.

# 3. Interference by Gamma Radiation

If a sample containing beta activity also contains gamma activity, the latter will also contribute some counts. For comparative measurements, the total (beta and gamma) count may be satisfactory. However, in some cases, it is necessary to determine the beta activity alone, and the correction for gamma activity must therefore be determined.

Because the counting yield of a thin-window detector for beta rays is about 100 times as great as for gamma rays, it usually happens that the major fraction of the counting rate is due to beta rays. The contribution of the gamma rays can be determined by taking an absorption curve with lead as described in II. C. 2. The gamma curve is extrapolated back to zero thickness, and the extrapolated value is subtracted from the total counting rate to give the beta counting rate. If the gamma: beta ratio is very low (about 0.001) the effect of bremsstrahlung may also need to be considered (see Section II. F. 2).

## 4. Correction for Decay

Quite frequently measurements of a radioelement are made over a period which is long enough with respect to the half-life to allow the activity to change appreciably by decay. In order that the measurements may be comparable each must be corrected to the value which would have been obtained if it had been measured at the same time. In other cases, a measurement must be corrected for the amount of decay since its formation, as when the absolute yield of a nuclear reaction is desired.

For the general laws of radioactive decay and growth, see Rasetti, "Elements of Nuclear Physics", p. 27-32. In particular, the relation between the activity of a preparation at two times  $t_0$  and  $t_1$  is given by

$$I_1 = e^- \lambda (t_1 - t_0)$$

where  $\lambda$  is the decay constant, and is related to the half-life T as follows

$$\lambda = \frac{\ln 22}{T} = \frac{0.693}{T}$$

A more convenient form of the above equation for calculations using logarithms is:

$$\frac{I_1}{I_0} = 2^- (t_1 - t_0)/T$$

If a sample is measured at time  $t_1$  the value of its activity at time  $t_0$  is given by:

$$\log I_0 = \log I_1 - 0.30103 \frac{t_1 - t_0}{T}$$

Example: A sample of radioactive phosphorus ( $P^{32}$ ; T = 14.1 days) is measured at 8:00 A.M. November 24, and found to give 980 counts/minute. What is its activity as of 2:00 P.M., November 6?

$$t_1 - t_0 = 24.3 - 6.6 = 17.7 \text{ days}$$

$$\log I_{O} = 980 - \frac{0.30103 \times 17.7}{14.1}$$

$$= 2.9912 - 0.3778$$

= 3.3690

I<sub>0</sub> = 2340 counts/minute

#### F. MEASUREMENT OF GAMMA RADIATION

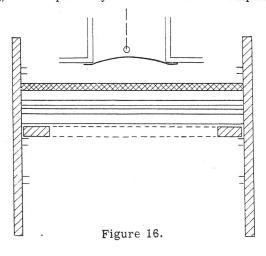
Since gamma rays are almost always accompanied by beta rays or other electrons, the latter must be prevented from entering the detector. This can be done by interposing an absorber between the source and the detector. A thickness of lead equivalent to  $2 \text{ gm/cm}^2$  is sufficient to stop all beta rays normally encountered, without appreciably reducing the intensity of the gamma rays.

## 1. Sample Preparation

Most of the methods which are used for preparing samples for alpha and beta measurement can also be used for the measurement of gamma radiation. An exception is that platinum plates should be avoided for accurate work where beta radiation is also present because of the bremsstrahlung effect, noted below. In addition, samples which are much bulkier can be used because the self-absorption effect is small. This is often made necessary because of the low counting yield for gamma rays. Thus several grams of a solid may be placed in an ashing capsule and counted. However, if the sample material consists predominantly of elements of high atomic number, bremsstrahlung will become important.

#### 2. Interference by Beta Radiation

There are several mechanisms by which beta radiation can give rise to gamma rays of X rays. The most important is "bremsstrahlung", the conversion of part of the kinetic energy of an electron into electromagnetic radiation in the force field of an atomic nucleus. The conversion may take place either in the field of the nucleus emitting the electron (innere bremsstrahlung) or of another nucleus (aussere bremsstrahlung). The external effect is several times as great as the internal effect and is roughly proportional to the square of the atomic number of the stopping material. The energy distribution of the quanta is continuous, extending up to the kinetic energy of the electrons, but with the softer components predominating. Thus some gamma rays are observed coming from all beta emitters, even when no nuclear gamma rays are emitted. In making quantitative measurements of gamma radiation the interference of bremsstrahlung must be considered. The effect may be minimized by stopping the electrons in a material of low atomic number, such as aluminum. A "sandwich" technique is commonly used. The sample, mounted in any of the usual manners except on platinum or other material of high atomic number, is placed in the second step of the counter. On top of the sample and immediately below it are placed aluminum absorbers sufficiently thick to stop all of the beta particles. About 1500 mg/cm² is commonly used. Above the sandwich is placed a lead absorber of about 1500 mg/cm² to partially absorb the softer components of the bremsstrahlung.



Although this arrangement tends to minimize the amount of bremsstrahlung it cannot reduce it completely, and corrections must be made if the true gamma count is low relative to bremsstrahlung count. As a rough approximation the bremsstrahlung count with the geometry described is  $2.5 \times 10^{-5}$  times the beta count taken with the sample on the top step. For example, suppose a sample gives 300 c/m on the second step with the sandwich arrangement, and an aliquot 1/1000 as large gives 2000 c/m on the top step with no absorber. This means that the beta count of the larger sample is  $2 \times 10^6$  c/m, so that the bremsstrahlung count should be  $(2 \times 10^6) \times 2.6 \times 10^{-5} = 50 \text{ c/m}$ . Therefore the true gamma count is 300 - 50 = 250 c/m. When the true gamma count is of the same magnitude or smaller than the bremsstrahlung count, this type of calculation is too crude for accurate results, since the conversion factor will be different for beta rays of different energies.

# 3. Counting Yield

The counting yield for gamma rays is much lower than for beta rays, because most gamma rays can pass completely through the counter without discharging it. For a given geometry, the counting yield depends on the energy of the gamma radiation. Counts are due mainly to photoelectrons or Compton recoil electrons liberated in the walls of the counter or the lead absorber in front of the window. The probability of a gamma ray releasing such an electron decreases as the gamma energy increases. However, the ranges of the electrons in the material in which they are produced increase more rapidly with increasing gamma energy, and therefore the net effect is an increase in counting yield with increasing gamma energy. Exact values are not known, but with the arrangement described (sample on second shelf) the counting yield is approximately 0.3 per cent times the energy of the gamma ray in Mev.

#### G. Characterization of Activities

Any radioelement can be characterized by its chemical identity, half-life, and type and energies of radiations. These properties must of course be determined for all new radioelements. An unknown radioactive substance can often be identified by measuring one or more of these characteristics. Determination of chemical identity is made by chemical separations. Half-lives are determined by taking decay curves. Information about the nature of the radiations can be obtained by taking absorption curves, although for exact measurements special instruments must be used. Mode of formation is often of value in identifying activities, as are also genetic relationships (radioactive parent or daughter) when they exist. Since decay curves and absorption curves can be taken with simple counting instruments, they will be described here.

## 1. Decay Curves

A plot of the radioactivity of a sample against time is called a decay curve, because almost always the activity decreases with time. To obtain such a curve, the activity is measured at a number of different times using the same instrument and geometrical arrangement each time. The activity is then plotted as a function of time using semi-log paper with the activity on the logarithmic scale. The reason for this is that the decay of a single radioactive substance, being exponential, gives a straight line on such a plot. Figure 17 shows a typical example.

If a straight line is obtained, it can be concluded that only a single active substance is present. The half-life is obtained by measuring the time required for the activity to decrease by a factor of two, this being the same for any part of the curve. For greater precision, a longer part of the curve may be used by noting the time required for a decay of a large power of two, and dividing this time by the exponent. For example, in Figure 17, the decay from 4096 c/m to 1 c/m requires 12 half-lives, since  $\frac{4096}{1000} = 2^{12}$ 

If more than one radioelement is present, the resulting decay curve will be the sum of the individual curves, and may appear as in Figure 18.

The solid line, which is the measured activity, is the sum of the three exponential curves indicated by the dashed lines. Such a curve may be analyzed by starting at either end, drawing a straight line tangent to the line, and subtracting this activity from the measured curve. From the residual curve the next activity may be picked out and subtracted, and so on. If the periods are close together, or the time of measurement is not sufficiently great, this method will not yield very good results.

If a decay curve is concave downwards, this is evidence for growth of the measured radioelement from a radioactive parent. If the initial growth is more rapid than the initial decay, the curve may rise before falling. If the rate of growth is constant, the activity rises to a constant value. Types of growth curves are shown in Figures 19, 20, and 21.

## 2. Absorption Curves

An absorption curve is a plot of the measured activity as a function of thickness of absorbing material between the sample and detector, and gives a measure of the penetrating power, and hence an index of the energy of the radiation. For beta rays, aluminum absorbers are commonly used (alternately paper, graphite, etc.) and for gamma rays, lead is used (alternatively tungsten, etc.). The plots are made on semi-log paper as for decay curves, since this gives very nearly a straight line forhomogeneous gamma rays and roughly a straight line for beta rays of a single natural spectrum.

Typical absorption curves are shown in Figures 22, 23, 24, and 25.

The important characteristic of a beta absorption curve is the end-point, or maximum range of the electrons. This is determined by inspection of the curve, and is the point at which the curve appears to have completely bent over to the next more penetrating component. Exact location of end-points is often difficult. From a curve or table of the range-energy relationship, the maximum energy of the beta rays is found; this is the important characteristic of a beta ray spectrum.

The important characteristic of a gamma absorption curve is the slope, usually given as the half-thickness, or thickness necessary to reduce the intensity by a factor of 2. This is determined in the same manner as the half-life from a decay curve. Complex gamma absorption curves are analyzed in exactly the same manner as complex decay curves. The energy of the gamma ray can be found from the half-thickness using standard tables or curves.

Precautions against contamination are especially important in taking absorption curves. Contaminated absorbers are an especially irritating source of trouble. For this reason, handle absorbers only by the tabs, which are covered with Scotch tape that can be exchanged frequently. Occasional background counts on the absorbers are desirable.

#### SECTION III - STATISTICS AND COUNTING

#### A. RELIABILITY OF COUNTS

The statistical accuracy (fractional error of a counting rate) depends only on the total number of counts taken. Thus, the counting time necessary to obtain a certain accuracy varies inversely as the counting rate, since total counts = counting rate x time of count. Because the laws of probability apply, one can never be entirely certain of one's results; the measured rate is never the <u>true</u> rate, which is the value that would be determined with an infinite number of counts. It is, however, possible to make estimates as to the likelihood that the true rate will lie within specified limits centering around the determined values.

For every set of limits there is a different probability that the result is correct to within these limits, and this probability is greater the broader the limits. For example, if a counting rate is determined to be 200, it is more likely that the true rate lies between 190 and 210 than between 199 and 201. The limits are usually expressed in terms of the fraction of the counting rate—in the example, the above limits are respectively 0.05 and .005 of the counting rate—and are spoken of as "fractional errors." The probability that the fractional error we actually make in using the experimental rate as the true rate is less than a specified fractional error is the probability that the true rate lies between the corresponding limits centering around the experimental value.

Mathematically expressed, if  $\rho$  is the fractional error and W the probability of making an actual error (expressed as a fraction) less than  $\rho$ , then

$$\rho = K/\sqrt{M}$$

where M is the total number of counts taken (M = counting rate x counting time), and K is a constant depending on W.

If we wish to consider the error such that we have nine chances out of ten (i.e., W = 0.90) of being within its limits, then K = 1.645, and  $\rho$  = 1.645/ $\sqrt{M}$  is called the "reliable fractional error".

If we wish to consider the limits such that we have only a fifty-fifty chance (W = 0.50) of committing an actual error less than  $\rho$ , we use K = .675, and  $\rho$  = .675/ $\sqrt{M}$  is known as the "probable fractional error".\*

A third error also in use is the "standard fractional error" in which K = 1.000 and the probability of having an error less than  $1/\sqrt{M}$  is .683. While the "errors" have been given special names only in these three cases, it should be realized that both  $\rho$  and the corresponding probabilities vary continuously. Table VIII summarizes the relationship of  $\rho$  and the corresponding probability for the various "fractional errors". The absolute value of the error is equal to the fractional error multiplied by M or K/ $\sqrt{M}$ . (M) =

Table VIII

Name of Error	K	Percentage Probability of making an actual fractional error less than $\rho = K/\sqrt{M}$ (where M = total counts)
Probable error	0.6745	50.0%
Standard error	1.000	68.3%
Reliable error	1.645	90.0%
	1.960	95.0%
	2.575	99.0%

Physicists in general use the probable error. Thus a result may be reported in the form

 $61.4 \pm 1.7$ 

<sup>\*</sup> Curves of  $\rho$  vs. M are shown in Figures 26 and 27 for both the probable and reliable cases. Figure 28 shows curves giving the probability of committing errors greater than 0.01, 0.025, and 0.05 of the counting rate for values of M from 10 to 100,000.

<sup>12-209-</sup>p34-bu

or

with it being understood that the probable error or the probable fractional error is meant. However, in this section, the reliable error is often used. In order to avoid confusion, it is desirable to use the convention of enclosing reliable error in brackets when they are used. Thus the above result would be written:

$$61.4 \quad \left[ \pm 4.2 \right]$$

or

$$61.4 \left[ \frac{+}{2} 6.8\% \right]$$

the brackets indicating that the reliable error or the reliable fractional error is meant. Of course all four of the above ways of writing the result mean exactly the same thing.

Example: If the counting rate is approximately 900 c/m and the count has been taken for nine minutes, then M = 8100 and the reliable fractional error,  $\rho$  r is:

$$\rho r = \frac{1.654}{90} = 0.0183 = 1.83\%$$

We have then nine chances out of ten of committing an error in our counting rate of less than 1.83 per cent which means that the probability is 0.90 that the true rate lies between 883.5 and 916.5 c/m. This fact is expressed by writing the actual count and the limits of error:

Similarly, we have five chances out of ten of having an error less than

$$\frac{.675}{90} = .0075 = 0.75\%$$

which means that the probability is 0.50 that the true rate lies between 893.2 and 906.8, and the result is written:

## B. COMBINATION OF RESULTS

#### 1. Some Formulae

It is often desirable to evaluate the error in a function of several counting rates rather than the error of one counting rate. Some useful formulae for a few simple functions are shown.

If <u>a</u> is the error (either reliable, standard or probable) in the count  $\underline{A}$  and  $\underline{b}$  the error in the count  $\underline{B}$  then the error in the:

#### (a) Sum of two counts

$$(A + a) (B + b) + ... = A + B + ... + a^2 + b^2 + ...$$

(b) Difference of two counts

$$(A - a) - (B + b) = A - B + \sqrt{a^2 + b^2}$$

(c) Square of a count

$$(A \pm a)^2 - A^2 \pm \sqrt{2 aA}$$

(d) Product of two counts

$$(A + a) \cdot (B + b) = AB + \sqrt{a^2 B^2 b^2 A^2}$$

(e) Quotient of two counts

$$\frac{A\stackrel{+}{\stackrel{-}{\phantom{}}}a}{B\stackrel{+}{\stackrel{-}{\phantom{}}}b} \ = \ \frac{A\stackrel{+}{\stackrel{-}{\phantom{}}}A}{B} \sqrt{\frac{a^2}{A^2} \ + \frac{{}^2}{B}}$$

Counting by Difference. As an example, we consider the case where a sample is counted before and after a chemical manipulation, and it is desired to find the number of counts lost or gained in this manipulation. This amount may be small, so that it is desirable to determine the accuracy of the difference of the two counting rates. If the sample is counted before and after, for the same time, t, the absolute error in the difference of the counting rates is K  $\sqrt{M/t}$ , where M is the total number of counts taken (lumping together the counts taken before and after the chemical manipulation), and K is the constant discussed above. (This equation follows from formula b above.).

Example: Suppose a sample is counted for 10 minutes, giving 340 c/m. After the chemical manipulation, a 10-minute counting period gives 300 c/m. The total number of counts taken was 6400. Our error is then:

$$K \sqrt{6400/10} = K \times 80/10$$

= 8K

Hence, we have nine chances out of ten that our difference 340 - 300 = 40 c/m be accurate to within  $\frac{1}{2}$  8 x  $1.645 = \frac{1}{2}$  13 c/m. Or we have a fifty-fifty chance that our difference of 40 c/m be accurate to within  $\frac{1}{2}$  8 x  $.675 = \frac{1}{2}$  5.4 c/m. We can then write: We have lost in the chemical manipulation:

$$40 \quad \begin{bmatrix} \pm & 13 \text{ c/m} \end{bmatrix} \text{ (reliable error)}$$
or
$$40 \quad \pm & 5.4 \text{ c/m} \text{ (probable error)}.$$

3. Ratio of two Counts. Example: Suppose two samples (800 c/m and 1000 c/m respectively) are counted for ten minutes apiece, a =  $\frac{KVA}{A}$  and b = KVB, so the error in A/B is A/B  $VK^2A/A^2 + K^2B/B^2 = KA/BV1/A + 1/B = KA/BVA + B/AB$ . Since A = 8000 counts, B = 10,000, the error is .012 K. The ratio is then:

## C. BACKGROUND COUNTING

It is unnecessary to count backgrounds for as long a time as the sample is counted. The most efficient way of counting backgrounds is to distribute the counting time as follows:

$$t_b/t_s = \sqrt{m_b/m_s}$$

Where  $t_b$  and  $t_s$  are the background and sample counting times, respectively:  $m_b$  and  $m_s$  are the background and sample + counting rates, respectively.

Example: Suppose that the sample and background count together approximately 1,000 c/m, and the background alone is roughly 2.5 c/m; then:

$$\sqrt{m_b/m_S} = \sqrt{2.5/1000} = 1/20$$

Hence the background need be counted for only 1/20 of the time that the sample is counted.

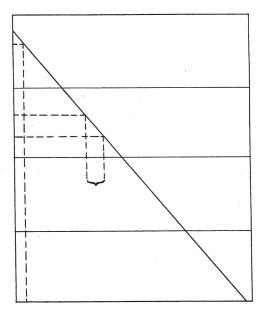


Figure 17. Simple decay curve, showing method of determining half-life.

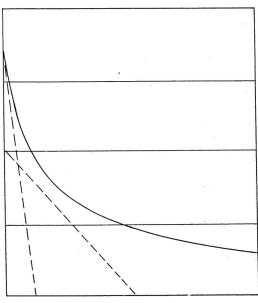


Figure 18. Complex decay curve, consisting of three components.

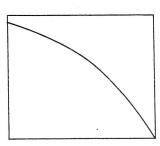


Figure 19.

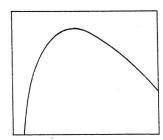


Figure 20.

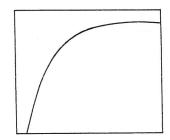


Figure 21.

TYPES OF GROWTH CURVES

12-209-p37-bu

#### REFERENCES

# Radioactivity - General

- 1. F. Rasetti, "Elements of Nuclear Physics", Prentice-Hall, Inc., New York (1936)
- 2. E. Rutherford, J. Chadwick, C. D. Ellis, "Radiations from Radioactive Substances", Cambridge University Press, Cambridge (1930)
- 3. G. Hevesy, F. A. Paneth, "A Manual of Radioactivity", Oxford University Press, London (1938)
- 4. E. Pollard, W. L. Davidson, "Applied Nuclear Physics", John Wiley and Sons, Inc., New York (1942)
- 5. J. M. Cork, "Radioactivity and Nuclear Physics", Edwards Brothers, Inc., Ann Arbor, Michigan (1946)
- 6. G. T. Seaborg, "Artificial Radioactivity", Chem. Rev. 27, --- (1940); Privately reprinted by Metallurgical Laboratory for use by project personnel.
- 7. G. T. Seaborg, S. G. English, V. C. Wilson, C. D. Coryell, "Lecture Series: An Introduction to Nuclear Chemistry", Report CL-440 (1942)
- 8. E. M. McMillan, E. Segre, E. Teller, F. Block, J. H. Williams, C. L. Critchfield, V. F. Weisskopf, R. F. Christy, "Lecture Series on Nuclear Physics", Los Alamos Report, LA-24 (1043) (Declassified)

# Alpha Counters

- 9. S. A. Korff, "Electron and Nuclear Counters", D. Van Nostrand Co., New York (1946); Chapters 2,3.
- 10. J. A. Simpson, Jr., "The Proportional Alpha Counter", Report CP-1527 (1944)
- 11. J. A. Simpson, Jr., "Manual for the Proportional Alpha Counter", Report CP-1817 (1944)
- 12. J. A. Simpson, Jr., "A Precision Alpha-Proportional Counter" (Abstract), Phys. Rev. 70, 117 (1946)
- 13. S. R. Chadwick, J. W. Kee, "Alpha Counters as used in Radio-assay of Plutonium", Report LA-488 (1946)

# Beta and Quantum Counters

- 14. Reference (8), Chapters 4,5
- 15. M. Deutsch, "Quantum Counters", Report CP-3085 (1945)
- 16. B. F. Scott, "Xenon-Filled Geiger-Müller Counters", Report CP-3501. (1946)
- 17. D. H. Copp and D. M. Greenberg, "A Mica Window G. M. Tube for Measuring Soft Radiation", RSI 14, 205 (1943)

#### Errors and Corrections

- 18. A. A. Jarett, "Statistical Methods Used in the Measurement of Radioactivity. Some Useful Graphs", Report Mon-P-126 (1946)
- 19. P. E. Yankwich, "Correction for Absorption of Beta Particles in Thick Samples. A General Method for Use in Tracer Work", Report CC-3567 (1946)
- 20. For growth and decay corrections, reference (2), Chapter I
- 21. R. P. Metcalf, E. Hennessee, "A Simple Method of Evaluating Coincidence Losses in Geiger-Müller Counting Circuits", Report CP-2582 (1945)
- 22. T. P. Kohman, "A General Method for Determining Coincidence Corrections of Counting Instruments", Report (H)CP-3275 (1945)
- 23. E. L. Brady, "Method for Determining Coincidence Errors in Geiger-Müller Counters", Report CC-3478 (1946)

# Sample Preparation

24. D. L. Hufford, B. F. Scott, "Techniques for the Preparation of Thin Films of Radioactive Material", Report CN-3328 (1945)

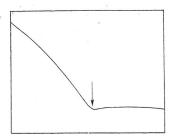


Figure 22. Simple beta spectrum. The flattening out is due to gamma rays or bremsstrahlung.

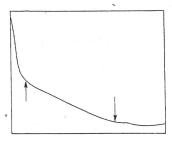


Figure 23. Complex beta spectrum with two components.

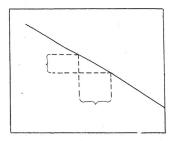


Figure 24. Single homogenous gamma ray.

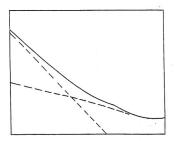
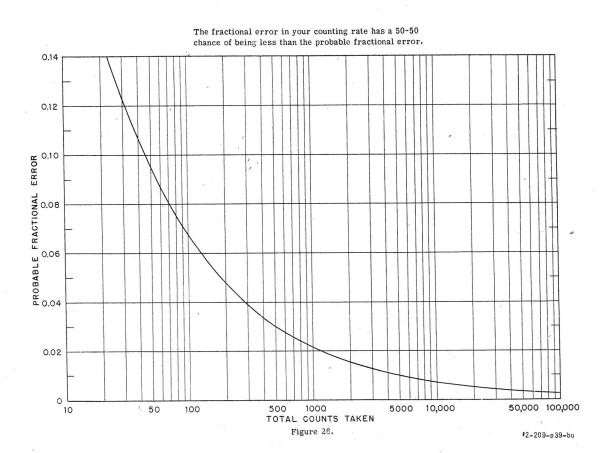
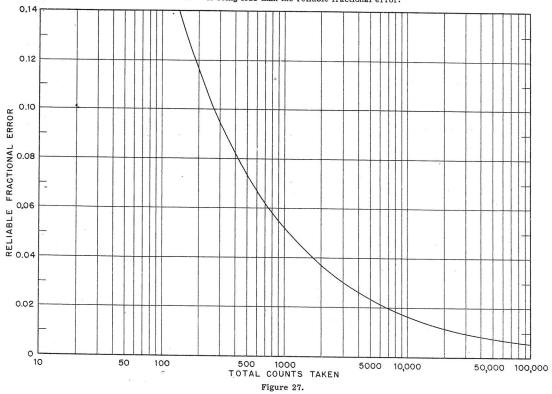


Figure 25. Complex gamma radiation with two components.



The fractional error in your counting rate has 9 chances out of 10 of being less than the reliable fractional error.



Probability of your counting rate being accurate to within a fractional error of .01, .025, .05.

