

## EXPERIMENT 2.

### PREPARATION OF RADIOACTIVE SOURCES

*(Experiment 2.1 and 2.2 should be written up as one combined report due 1 week after completion of part 2.2)*

The detection and measurement of nuclear radiation requires the preparation of a suitable sample of the radioactive material. The type of sample prepared is dependent upon a number of factors, both theoretical and practical, which stem from:

1. The type of radiation to be measured.
2. The chemical nature of the sample.
3. The practicality of preparing the sample.
4. The availability of instruments for detection and measurement.

The experimenter has the task of selecting the experimental conditions such that a maximum number of the factors are satisfactorily fulfilled. Generally, there is no definite choice which can be considered the best and the personal preference of the experimenter influences the final decision.

Gaseous samples are generally measured in some form of a gas ionization chamber in which the gas sample becomes, or is mixed with, the counting gas. A number of highly specialized techniques have been developed for these internal gas counters and, in certain cases, they are the best counting arrangement for the specific experiment. In other cases, when the mass and geometry of the sample is not important, the gas is collected in a container which can then be placed next to a conventional detector system. Still other sources can be constructed from gaseous samples by absorption onto solid surfaces such as activated charcoal. Many more ingenious devices have been conceived to form suitable samples from radioactive gases but, unless these gaseous samples are the only feasible ways of counting the source, the experimenter is generally better off to select either a liquid or solid form for the sample. The physical difficulties associated with handling gases adds a dimension of complexity which one generally strives to avoid.

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Liquid samples are quite often a very convenient form in which to measure nuclear radiation. The simplest arrangement of these consists of a test tube placed in a *well-type* solid scintillator. Due to the mass associated with such devices, they are restricted to gamma analysis and, if beta or alpha decay is to be detected, the sample must be mixed with a liquid scintillator. Such mixtures are very useful for low energy beta or alpha particles since the sample is effectively *inside* the detector and the problem of penetrating a window to the detector is avoided. This technique will be explored later in one of the experiments in this course.

Finally, samples may be prepared in the solid form. This is primarily a choice of convenience on the part of the experimenter. Solid samples are well-defined in size and character and they can generally be made and handled with greater ease. The most common methods of forming solid samples are:

1. Evaporation of an aliquot from a solution.
2. Precipitation and filtration from a solution.
3. Evaporation of a slurry.
4. Containment of a dry powder.
5. Electro-deposition from a solution.

Other more specialized techniques, such as sublimation, sputtering, etc., are also used for particular needs, such as the preparation of very thin (massless) sources. The first two methods are the most widely used and will be the ones of interest in this experiment. The last method will be used in a later study during the semester.

Of the two common methods for forming solid samples, precipitation of an insoluble material from solution and subsequent filtration is the most satisfactory type for the majority of counting needs. Most measurements are relative and the solid sample permits a high degree of reproducibility. In addition, it has the general advantages associated with ease of handling and long-term stability. However, some corrections are required if one sample is to be compared with another. Among these corrections are the following:

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1. Chemical yield or the fraction of the initial radioactive material ending up in the measured sample.
2. The *self-scattering* and *self-absorption* of the radiation due to its interaction with the sample material itself.
3. Geometric factors which are related to the position of the sample with respect to the detector.

The chemical yield correction can be made by standard analytical methods. The self-scattering and self-absorption corrections are related to both the type and energy of the radiation. Gamma radiation is relatively impervious to minor changes in sample thickness, but beta, and especially alpha particles can be seriously affected. Geometric corrections can be eliminated by duplicating the counting conditions. Where this is not possible, often simple corrections can be made. A very satisfactory method, devised by Bayhurst and Prestwood for counting beta particles, corrects for both sample thickness and geometric factors at the same time (see reference).

In order to introduce the student to working with radioactivity and to illustrate the standard techniques of source preparation, a number of solid sources will be made. In addition the effect of sample mass and sample backing on the observed count rate will be studied. However, at least a qualitative understanding of these effects is needed to understand the experimental results. Students are, therefore, urged to review these topics before progressing with the actual laboratory work.

## 2.1 A STUDY OF BETA BACKSCATTERING

Beta rays undergo large angle deflections due to their small mass. As a result of the relatively high probability for such events, a significant number of beta rays which start out in the direction away from the detector may be deflected back to it after multiple scattering. This scattering may be caused by the sample itself, if it is thick enough, or by the backing material upon which it is mounted. The backscattering factor,  $B$ , is the ratio between the count rate of the sample as prepared on the backing material and that of the same amount of sample suspended freely in a vacuum; it has a value between 1 (no backscattering) and 2 (100% backscattering). Scattering will occur also from the walls of the holder but this may be much less important than backscattering if the holder is constructed with low  $Z$  material, such as aluminum or Lucite. Backscattering will be appreciable from the floor of the holder for a thin sample if the lowest shelves are used for counting. The higher the atomic number of the scattering material, the larger is  $B$ . Also,  $B$  increases with thickness up to a saturation thickness, beyond which it is a constant. Counting is done usually with essentially weightless backing ( $B = 1$ ) or with a thick enough backing to have saturation. It might be expected that the saturation thickness would be one-half of the range of the beta particles in the backing material as this thickness must be traveled twice before detection. Actually, it is close to 20% of the range which is understandable when it is realized that approximately 95% of the beta particles are stopped in the first half of the total range.

Beta particles suffer degradation of energy during scattering, with the result that the scattered electrons have lower energy; as a consequence there will be a more important backscattering correction for windowless or thin window counters, since these low energy scattered electrons are more likely to reach the counter and be detected. Experimentally,  $B$  may seem to be a function of the quoted beta energy; however, for monochromatic beam of  $\beta$ -particles  $B$  is independent of beta energy.

## 2.1.1 Experimental

### 2.1.1.1 PREPARATION OF THE WORK SPACE

*(See previous experiment)*

### 2.1.1.2 SOURCE PREPARATION.

“Backless” sources of three different isotopes are to be used,  $^{137}\text{Cs}$ ,  $^{36}\text{Cl}$  and  $^{32}\text{P}$ . For the long-lived  $^{137}\text{Cs}$  and  $^{36}\text{Cl}$  isotopes, sources prepared in previous years will be provided. A  $^{32}\text{P}$  source will be prepared from an active stock solution. Using the adjustable autopipette (new tips), deposit an aliquot of the active solution directly onto a piece of plastic wrap, supported by a perforated Al card (exact amount of the source will be specified by the instructor). These samples can be dried under a heat lamp but care should be taken not to overheat the plastic wrap. Cover the sample mounted on plastic wrap with another layer of plastic wrap, as before (See Fig. 1).

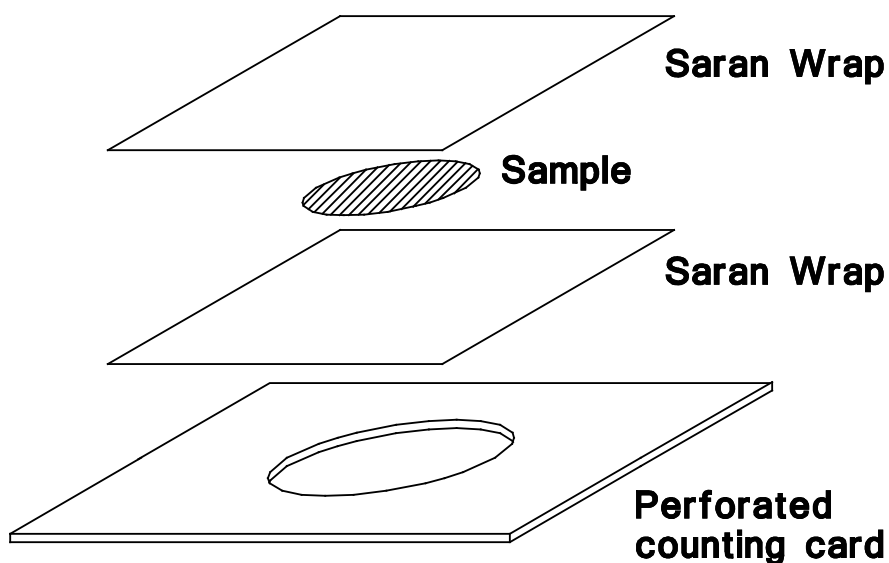


Fig. 1. “Backless” counting arrangement.

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#### 2.1.1.3 DETECTORS

*(This step may be omitted if the detectors have already been set-up)*

For *this experiment*, the detectors will be used outside their lead box to minimize backscattering due to the shielding. The detector and sample holder will be placed upside-down on two wooden blocks (do this with the detector bias voltage *off*). In this position, the sensitive area of the detector is facing upward (and therefore exposed). *Please* be careful not to poke anything into the detector! Turn the bias voltage on. Place the sample holder with the backless sample on shelf 1, and then rest the backing material directly onto the source. Cover the whole assembly with a cardboard box (when the bias is on, the detectors are light sensitive).

#### 2.1.1.4 DATA COLLECTION

Count the *backless* samples in the following arrangements (decide on a duration of counting such that the error on the count rate is no more than  $\approx 2 - 3\%$ ):

1. Count each sample with aluminum absorbers placed on top of the sample, increasing the thickness of the absorber after each count. Continue until no further change in count rate is observed with additional aluminum.
2. Count each sample with squares of various material available (plastic, aluminum, copper, lead, etc., at least  $250 \text{ mg cm}^{-2}$  thick) on top of the sample holder.
3. Before leaving, perform wipe tests of your area.

#### 2.1.2 Results and Discussion

Tabulate clearly all the data collected during the session; include an error estimate for all your measurements (when you turn in your report, submit your data as an EXCEL file using the template `expt2a_results.xls`, [www.sfu.ca/~brodovit/files/nusc346/templates/](http://www.sfu.ca/~brodovit/files/nusc346/templates/))

From data collected in Step 1, plot the net count rate *versus*  $d$ , the thickness in  $\text{mg cm}^{-2}$  of backing material for each beta source and estimate in each case the saturation thickness. From reference obtain the average  $\beta$  energy and/or  $E_{\beta\text{max}}$  for each of the sources used; tabulate your

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results as a function  $\beta$  energy of each source (average  $\beta$  energy or  $E_{\beta\text{max}}$ ). From the data of Step 2, plot the *net* count rate *versus*  $Z$ , the atomic number of the scattering material.

Discuss your results, taking into account the expected error on the counting rate. In particular, give an estimate of the minimum number of counts required to be able to observe the backscattering effect. You may want to verify the correlation (or lack of) between the saturation thickness you obtained from your data and the  $\beta$ -particle range found from a range-energy plot. The standard aluminum cards used for sample counting in this lab have a thickness of  $350 \text{ mg cm}^{-2}$ . Are they adequate for beta counting? Discuss how the phenomenon of beta backscattering could be used to identify an unknown element; would you recommend such a method?

The results of the wipe tests should appear in your report.

## 2.2 PREPARATION OF RADIOACTIVE SAMPLES

### BY PRECIPITATION AND FILTRATION: A STUDY OF SELF-ABSORPTION

As was mentioned before, quantitative measurement of radioactivity can most easily be made by preparing solid samples from precipitates. Uniform samples of constant dimensions can be formed by using a filter chimney arrangement and all other corrections necessary for an accurate determination of the disintegration rate of the sample can be obtained from the weight of the sample. To illustrate this technique and to study some of these effects, a set of AgCl samples of constant specific activity (constant activity per unit mass) but varying mass (same area but different thickness) will be prepared. This is done by precipitating all the chloride (as AgCl) from a series of solutions containing different amounts of  $\text{Cl}^-$  and a proportional amount of  $^{36}\text{Cl}$  tracer.  $^{36}\text{Cl}$  being a beta emitter, the series of AgCl precipitates will be counted on a silicon detector. If there was no self-absorption, the activity of a particular sample should be proportional to its mass. Within experimental error and for a given geometry, deviation from direct proportionality can be attributed to self-absorption by the sample.

The actual number of radioactive atoms which are normally dealt with is extremely small when compared to the number of atoms in a mole of substance. In fact, if only the active atoms are to be handled chemically, very special techniques are to be used. For example, 10 kBq ( $\approx 0.4 \mu\text{Ci}$ ) of radioactive  $^{36}\text{Cl}$ ,  $t_{1/2} = 3.01 \times 10^5$  years (this is the order of magnitude of activity handled, in general, in this laboratory), represents only  $2.3 \times 10^{-7}$  moles (or  $8.0 \times 10^{-6}$  g) of this particular isotope. Such a miniscule amount is not easy to handle. Generally, inactive isotopes of the element of interest are added as a *carrier*; this is referred to as the isotopic carrier. Sometimes inactive atoms of a different element, having close chemical properties, can act as a *non-isotopic carrier*. This permits one to handle normal quantities of material and to use regular chemical procedures. Another advantage of adding carriers is that chemical yields for the chemical procedure can be obtained from a comparison of the amount of carrier recovered to

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the initial amount used. In the present case, natural stable chloride in NaCl will act as a carrier for the radioactive label  $^{36}\text{Cl}$ .

**2.2.1 Experimental**

**Bench preparation.** *Prepare your working space as in the previous experiments, but in addition make sure that the filtering flask is resting on an absorbent pad and organize the work space in such a way as to have the filtration setup within easy reach without having to lean over other implements. It is a good idea to have the procedure written up on a piece of paper which can be posted next to the working bench. Double-gloving is recommended and do check yourself and your working area frequently with the GM survey meters available in the lab.*

**Counting cards.** Prepare six “backless” counting cards, one for the standard and five for each sample of various thicknesses. Weigh five small filter discs to the nearest 0.1 mg on the analytical balance.

**Active solution.** To prepare a  $^{36}\text{Cl}$ -labelled NaCl solution, pipette (with a volumetric pipette) 25.00 ml of a  $5 \times 10^{-2}$  M NaCl solution (record the exact concentration) into a 50 ml beaker and add a *spike* of the  $^{36}\text{Cl}$  source provided (exact amount will be specified by the instructor).

**Specific activity.** To obtain the specific activity (cps/mole Cl, for example) of your  $^{36}\text{Cl}$ -labelled NaCl solution just prepared, deposit with an autopipette 100  $\mu\text{l}$  of this solution onto a filter disk. Dry under the heat lamp, cover with plastic wrap, label the card and mount on a backless counting card. You may want to start measuring the activity of this standard sample while you are proceeding with the rest of the experiment.

**AgCl sample preparation.** Pipette into five test tubes respectively 0.2, 1, 2, 5, 10 ml of this active solution. Precipitate AgCl by approximately doubling the volume of solution in each test tube by addition of 0.1 M  $\text{AgNO}_3$ ; use adjustable pipettes or measuring cylinder to do this. Digest the mixture in a hot water bath for 2 min. Filter the precipitates using the filter

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chimney arrangement (Fig. 2) (*do not forget the sintered disc supporting the filter disc*). Break up lumps with a stirring rod. Control the speed of filtration by reducing the flow of the water aspirator in order to obtain an even deposit. Rinse with acetone and air-dry for a few minutes under suction. Carefully take the filter assembly apart and remove the filter paper, using a spatula and tweezers. If disposable plastic petri dishes are used, make sure that *all* the acetone has evaporated as this particular plastic is soluble in acetone. Dry the sample slowly under a heat lamp. Use a large Petri dish with its lid on to carry active samples around the lab. After drying, weight each sample on a watch glass placed on a plastic Petri dish to avoid contamination of the balance pan. Mount the filter disc in order to obtain a backless source (previous experiment Fig. 1). Label each prepared source with your name, date and isotope name. At the end, be sure to leave your sintered glass discs in the cleaning bath provided. All contaminated glassware must be first rinsed over the “Waste Liquid Container” to wash off the bulk of the contamination, and then it can be washed with detergent in the sink.

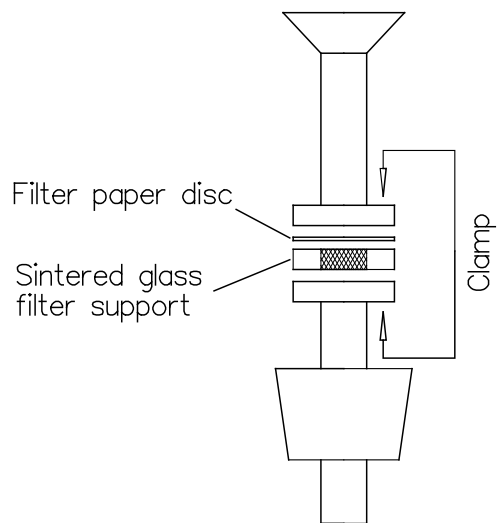


Fig. 2. Filter chimney arrangement.

**Activity measurements.** Count all your samples on the beta counter on the same shelf. Use the same experimental arrangement as in the previous experiment. The duration of counting should be adjusted depending on the activity of the sample and how long you can afford to

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stay in the lab! For low activity samples, several hours may be necessary to obtain the count rate with the required precision; in this case, overnight counting would be an option, or letting the sample to be counted for a preset length of time the following days with occasional visit to the lab just to record the results and change sample.

**Sample disposal.** When all your measurements have been performed, carefully dismantle<sup>1</sup> your samples and discard into the “Active Solid Waste” container; place the counting cards into the container assigned for this purpose.

Perform wipe tests of your area before leaving.

**2.2.2 Results and Discussion**

Tabulate clearly all your data as collected during the lab session; include error estimate for all your measurements. Along with your report, submit as an email attachment your data tabulated in the format provided in the EXCEL template **expt2b\_results.xls**

([www.sfu.ca/~brodovit/files/nusc346/templates/](http://www.sfu.ca/~brodovit/files/nusc346/templates/)).

Construct the appropriate charts and graphs to illustrate the effects of sample mass on the observed count rate; make sure to include an error calculation for all the reported results. Try to rationalize your results by taking into consideration actual sample imperfections (eg, uneven lumpy deposit) and the chemical yield for each sample. In your discussion, comment on the experimental procedures used to prepare your samples and also on your counting arrangement; include recommendations to follow when counting beta samples. Indicate the strengths and weaknesses of the method you propose.

In your discussion define and/or explain

1. Chemical yield

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<sup>1</sup> The samples are in a powder form and can easily disperse. This operation must be performed under the fume hood. Do check afterwards with the GM detector for “specks” of AgCl precipitate which may have scattered around.

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2. Isotopic carrier
3. Non-isotopic carrier
4. Carrier free
5. Self-scattering
6. Self-absorption
7. Counting efficiency
8. Backscattering
9. Massless source

The results of the wipe tests should appear in your report.

**2.2.3 References**

1. Overman and H.M. Clark, *Radioisotope Techniques* (McGraw-Hill Book Company, Inc., New York; 1960).
2. Friedlander, J.W. Kennedy and J.M. Miller, *Nuclear and Radiochemistry* (Second Edition: John Wiley and Sons, New York; 1964).
3. O'Kelly, *Detection and Measurement of Nuclear Radiation* (Nuclear Science Series, NAS-NS 3105; 1962).
4. Bayhurst and R.J. Prestwood, *Nucleonics* **17**, 92 (1959).