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RADIOISOTOPE TECHNIQUES FOR CHEMICAL,
PHYSICAL, AND INDUSTRIAL APPLICATIONS

By
Andrew J. Cassell

September 1961

Argonne National Laboratory
Argonne, Illinois

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RADIOISOTOPE TECHNIQUES FOR CHEMICAL
PHYSICAL, AND INDUSTRIAL APPLICATIONS

by

Andrew J. Cassell

A Laboratory Manual Designed for Use with
Programs of the International Institute of
Nuclear Science and Engineering

September 1961

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PREFACE

This laboratory manual is designed to teach persons affiliated with the International Institute of Nuclear Science and Engineering some of the basic fundamentals of the application of radioisotopes. It is also designed to introduce the principles of radioisotope technology.

The experiments in this manual are designed to be practical. With this in mind, I feel that through the participation of the laboratory worker in completing these experiments, he will be able to derive the broader applications of each experiment. The fields of endeavor that are touched upon in the manual are chemistry, physics, and industry. It has been the opinion of the author that radioisotope applications in the biological and health safety fields should be conducted within their own divisions. In this manner specialized applications can be conducted.

From the Table of Contents, it can be seen that the majority of the experiments are designed for the scientific personnel who have had little background in radiochemical or radioisotope technology. A few of the experiments are designed for the more advanced personnel. Because of the broad coverage of this special field, I feel that a review of the fundamentals of radiochemistry is warranted. This will assist the individual participant in reorganizing his or her knowledge for better application to this phase of the nuclear field.

There are several important objectives which the author is attempting to achieve in the manual. First, the manual will provide a broad basic knowledge in the field of radioisotope technology. Second, rather than approach specialized fields, the experimenter will be given a more complete coverage of the overall applications in the three aforementioned fields. Third, since the laboratory manual is designed to be used in the programs of the International Institute, it can be assumed that many of the participants will be scientists from other countries. It is my desire to give to these scientists sufficient information on radioisotope experiments so that they will have an opportunity to expand on the principles of the experiments to fit their own personal needs. Fourth, it is of the utmost importance that these experimenters become completely familiar with the apparatus which is generally found in a radiochemical laboratory. Many of these scientists will return to remote locations throughout the world. In some cases, they will have to augment their laboratory with equipment which is at hand.

A series of twelve lectures is usually given with the laboratory course. This is done in order to introduce the theoretical aspect of the experiment as well as to acquaint the experimenter with the scope of the particular experiment. I feel that anyone who uses this manual as a tool for learning should attend the lectures or obtain a written form of the lectures.

If the individual, through the use of this laboratory manual, becomes better acquainted with radioisotope technology, I feel that much will have been done to increase further the untold uses of the radioisotope.

A. J. Cassell

International Institute of
Nuclear Science and Engineering
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RADIOISOTOPE TECHNIQUES FOR CHEMICAL, PHYSICAL, AND INDUSTRIAL APPLICATIONS

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INTRODUCTION TO LABORATORY WORK

To better acquaint persons working in radioisotope laboratories, there are certain important points that must be considered. An instructor, technician, or student working in the laboratory should not fear the radioactive material that they may come in contact with. An atmosphere of unsafe conditions is generated if the experimenter does not have complete confidence in the work being done.

If all the people working in a radiochemical laboratory would follow a general set of rules, set down for the purpose of reducing these unsafe and hazardous conditions, a more purposeful meaning could be placed on their work. It should be very obvious that carelessness cannot be tolerated.

Some thought should be given to the normal accepted procedures that are generally used in radioisotope laboratories. Since the majority of the work consists of the preparation and then the analysis of radioactive material, it should be understood that the laboratory areas should be divided in such a manner that one phase of the operation will not interfere with the other. Specific areas should therefore be divided into specific uses. The preparation of samples, special processing, and other applications which deal with the handling of the radioactive material should be carried out in special work areas. In order to reduce the possibility of contamination, special absorbent paper should be used. This would include use in glove boxes as well as on work benches.

In designing a radiochemical or radioisotope laboratory, one must consider the location of the preparation area with respect to the counting room. The counting room is usually shielded from the work area in some manner that is suitable for the reduction of radioactivity. The storage of radioactive material should be such that it will not exceed the safety factor set down for the particular laboratory.

Normally, all work conducted in the glove boxes should be performed in some type of "catch all" tray which would prevent the spreading of contamination from the immediate area. By this method we not only confine the spillage to a given area, but the disposal of the spilled material is more easily handled.

Solid waste material to be disposed of is always put into DAW (Dry Active Waste) cans unless the activity is too high; then the material must be placed into special shielded pots. Liquid waste is usually placed into special plastic bottles that may be easily removed. Close check of the total activity of the waste containers should be made by the radiation safety man.

Whenever personnel are working with a radioactive source, the hands should be protected by rubber gloves. The lab is the only place that the rubber gloves should be worn. Laboratory coats are also a must and should be worn only in the work area. There is always the possibility that some contamination can be carried to the counting room by careless action.

The following is a list of thirteen regulations that should be adhered to while in the laboratory. This is the only way that safe conditions can exist and your fellow workers can survive.

Laboratory Regulations

1. No eating, drinking, storing of food, or use of cosmetics shall be allowed in the laboratory.
2. Active liquids shall never be pipetted by mouth, but should be handled with special mechanical suction apparatus.
3. When a person is ready to leave the laboratory, the hands should be washed first. If rubber gloves are on the hands, remove them and throw them in the DAW can. If there is any detectable radiation present after the washing, it should be reported to the radiation safety personnel.
4. Under no circumstance should suspected contamination be allowed to stand around without the material being surveyed.
5. One of the easiest ways of personnel becoming contaminated is to work with active material with skin abrasions or open wounds. No person should be allowed to come in contact with radioactive material unless wearing rubber gloves. Any accident that occurs, as far as personal injuries, should be reported to the instructor at once.
6. Active material should not be handled by anyone without the necessary monitoring devices. In the radioisotope laboratory both the pocket dosimeter and the film badge must be worn.
7. As previously stated, all solid waste should be placed in DAW containers, unless the activity is such that it is prohibitive.
8. All liquid waste should be placed in special liquid waste containers, identified as to the type of radiation and the approximate activity.

9. If spillage occurs, the following should take place: (1) carefully blot up as much of the active material as possible, (2) place the blotting material in a DAW can if the level of activity allows it to become disposable, and (3) carefully mark the contaminated area as to the type of activity and the dose rate.

10. During the clean-up period, any apparatus which shows activity above background must be washed in sinks that are designed to empty into retention tanks.

11. The storage of radioactive material in the laboratory should be done in such a manner that there will be no possibility of increasing the background in the counting room.

12. All injuries, spills, or other such emergencies should be reported to the person in charge of the laboratory at once. Delay of this regulation can result in disastrous results.

13. Any calculations or data taken in the laboratory must be completed before the worker leaves the area.

Note: Figures in this manual depict apparatus or suggested design for experimental procedure. Several of these figures are charts from which additional information may be taken.

Laboratory Design

In radioisotope and radiochemical laboratories, the arrangement of the rooms is very important. Generally speaking, the units should be placed in one operating area. This decreases the probability of contamination of other working areas.

The operations should also be divided among areas of various assigned levels of radioactivity. Depending upon the type of specific activity that is expected, the division of the radioisotope area should be such that the rooms or working areas should be classified according to the levels of activity that may be used in them. The low-level activity area should be adjacent to the intermediate area, and the high-level active area adjacent to the intermediate. This type of setup will tend to reduce the possibility of interference of the high-level activity with the background in the low-level counting area. It also will reduce the possibility of cross-contamination from one level of activity to the next.

Normally the counting room is placed in such a position that it is the greatest distance from the high-level area and quite often a part of the nonradioactive or low-level areas. One of the major offences in a radioisotope laboratory is the unnecessary contamination and effect on the background in the counting room. Special precautions must be taken at all times to prevent this.

Due to the possibility of air-borne particulates, the ventilation of the laboratory area should be such that the air flow should be directed from the counting room toward the high-level active area. In this way the probability of air contamination in the counting room is reduced. The drawing of the typical laboratory layout in Fig. 1 was designed by the author; it is set up to accommodate eight (8) people, excluding the laboratory instructor. The level of activity is limited to the millicurie range. It should be noted that special efforts have been made to reduce the possibility of contamination to a minimum.

Fig. 1

A Typical Laboratory Layout

Radiochemical Laboratory Equipment

In the following section, pages 8 through 11, some of the equipment which is normally found in a radiochemical laboratory is described. The purpose of this section is to acquaint the laboratory worker with some of the apparatus which is mentioned in the experiments of this manual but are not described in detail.

Since this represents a small portion of the equipment which is normally found in a radiochemical laboratory, a more complete list may be found in United States Atomic Energy publication number AECU-2226, titled, Design of Laboratory for Safe Use of Radioisotopes. The apparatus

listed in this publication are given for several different laboratory sizes, therefore the listing may be far from complete for specific needs. When designing a radiochemical laboratory, it is suggested that reference be made to other laboratory manuals which have been published. Several of these manuals are listed in the Reference Section of this laboratory manual.

Micropipettes of the type illustrated in Fig. A and Fig. B are available in a variety of sizes from 1 to 800 λ ($1 \lambda = 10^{-3}$ ml). The specified volume marked on each pipette refers to the volume of liquid contained rather than the volume delivered. To deliver the total quantity in each pipette, it is necessary to rinse the pipette with the solvent involved. Rinsing consists of repeatedly drawing fresh solvent into the pipette to the calibration point and extracting the solvent on the source planchet. The pipette control shown in Fig. B consists of a glass syringe which is connected to the pipette by means of a solid rubber stopper through which a hole has been bored. The hole should be slightly smaller in diameter than the tip of the pipette. The instructor will show each person the correct procedure for filling and transferring the liquid.

Fig. A

Fig. B

When transferring volumes of liquid larger than one-half milliliter, the apparatus shown in Figs. C and D is to be used. The pipettes are calibrated from one-half milliliter to one hundred milliliters, and are designed to deliver the required quantity without the necessity of flushing the pipette. The special type of bulb used as the suction mechanism is designed with a release button on the top of the bulb. Fig. D shows the correct position for transferring the solution. The thumb is used to control the release of the solution by the application of a slight pressure on the release button. The liquid will then approach the calibrated mark on the pipette. This method will completely eliminate the possibility of using the mouth as a method of suction. Practice the delivery of distilled water from one container to another until you have mastered the technique.

Fig. C

Fig. D

The primary purpose of the proportional counter is to provide a device which will give an output pulse produced by some initial ionizing event. The counter is designed to detect and count the number of electrons that reach a central wire in the counter mechanism (see Fig. E). The type used in most radiochemical laboratories is of the 2π type, that is, designed to detect only in a 180° arc. Since the electrons are being emitted in 360° arc, it is possible to construct a 4π counter which would be more efficient. After the samples are prepared on planchets, they are introduced into the counter. In the instrument shown in Fig. F, the large knob is turned counterclockwise and pulled toward the operator. The planchet is placed in the receiving well and the slide is then returned to its original position. For a more complete discussion on proportional counters see reference number 22.

Fig. E

Fig. F

For the measurement of beta and gamma radiation, one of the more suitable methods is based on the use of scintillation counters. The scintillation detector can absorb the beta particle or secondary electron produced by gammas and this will produce a light in the scintillator. The light is transmitted to the photocathode of the photomultiplier tube. By means of an electron-multiplier section of the photomultiplier tube, the photoelectrons are multiplied many times. The resulting voltage pulse is fed into the pre-amp system. It is then fed to a discriminator and then to a pulse shaper and finally to an electronic counter. By using a suitable shield, the counting of gamma activity alone may be accomplished. In Figs. G and H a side and a top view of the detector system, which is encased in a suitable shielded container, are shown. This setup is designed for liquid samples only. The electronic counter is similar to the one shown in Fig. E.

Fig. G

Fig. H

Experiment 1 - ISOTOPE PRODUCTION

DISCUSSION:

The basic theory connected with the production of isotopes deals with the most suitable irradiation source. In normal isotope production, the procedure is to activate a stable isotope to a condition at which the activity will be large enough to give a good counting rate but low enough to eliminate additional monitoring.

In order to facilitate the production of a specific isotope, calculations must be made to determine the relative time necessary for activation as well as the range of activity necessary for experimentation. In order for the range of activity of the isotope thus produced to be optimum, the proper time of activation should be calculated.

The time of activation for any stable nuclide will depend on the particular specie of nuclide used and, in turn, on the half-life of the radioisotope thus formed. For any particular desired activity, the factors which determine the time of activation are: the number of atoms in the sample undergoing activation, the thermal neutron flux of the reactor (or other source) used, and the absorption cross section of the nuclide being activated. (A high reactor flux of, say, 2×10^{12} neutrons/cm²/sec would cause saturation^{(1)*}).

In isotope production and activation analysis, the purity of any element must be considered. Many of the elements which exist in nature appear in several stable isotopic forms. By using a specific reference which gives the percentage of the stable form existing in nature as well as the thermal neutron cross section, such as contained in BNL-325,⁽²⁾ calculations can be made rather easily. The activity of the specific isotope to be produced can be determined for any time of activation up to a point where saturation activity reaches 100 per cent by means of the formula:

$$A = N \phi \sigma (1 - e^{-\lambda t}),$$

where

A = Activity (disintegrations per sec)

N = Number of nuclei present in sample

ϕ = Neutron flux (neutrons/cm²/sec)

σ = Absorption cross section (cm²)

*Numbers in parentheses refer to references listed at the back of the manual.

e = Base of natural logarithms, 2.71828

λ = Decay constant (sec^{-1})

t = Activation time (sec).

Since $\lambda = 0.693/T$, where T is the half-life, the equation is often written as

$$A' = N \phi \sigma (1 - e^{-0.693t/T}).$$

In some cases, the time of activation for a small-size sample is unduly short, and under these conditions it is impossible to provide a feasible time of activation with the high neutron flux available. In this case, it would be necessary to use a larger sample size and then dilute the isotope in order to provide a counting rate that is not excessive.

APPARATUS: (See Fig. 2)

Aluminum containers (two needed) for dry salt samples

Aluminum wire for securing container caps

Aluminum foil for wrapping salt within container

Analytical balance for weighing salt samples

The CP-5 Reactor for activating salt samples

Volumetric flasks (50 ml - two needed) for exact volume of solution

Pipets (10 and 25 λ) for obtaining aliquot portions

Stainless steel planchets

Scissors

Spatula

Rubber gloves

Bunsen burner

Proportional counter

Scintillation counter (well-type)

Infrared drying lamp

Beakers (30 ml)

Lead bricks

1. Infrared Drying Lamp
2. Scissors
3. Stainless Steel Planchets
4. Aluminum Containers for Irradiating Samples
5. 10- λ Pipette
6. 25- λ Pipette
7. Spatula (One Stainless and One Porcelain)
8. 250-ml Volumetric Flasks

Fig. 2

Apparatus for Experiment #1

MATERIALS:

Sodium nitrite (NaNO_2), dry saltManganous acetate ($\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$), dry salt

Distilled water

PROCEDURE:

Select suitable isotopes for activation - sodium nitrite and manganous acetate will be used in this experiment.

With the scissors, cut a small piece of aluminum foil (about 5 cm x 6 cm) and weigh it to the nearest 0.0001 gm on the analytical balance. Next, place approximately 0.2 gm of NaNO_2 on the foil, the exact weight to be determined to an accuracy of 0.0001 gm. Repeat with $\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$, using a sample of approximately 0.14 gm.

Carefully fold the salt in the foil to form a tight seal, taking precaution not to lose any of the salt. Carefully place the packet into an aluminum container and secure the cap. (See instructor for this procedure.)

[Containers are now ready to be placed (see Fig. 3) in the CP-5 Reactor for activation. However, in this case, previously prepared samples of NaNO_2 and $\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$ which have been activated will be ready for completion of the experiment.]

Fig. 3

Rabbit Mechanism for Isotope Production

On receiving the activated samples, monitor the aluminum can in order to determine whether excess dose rate is present. (Excess dose rate, in this case, would be 100mr/hr or higher.) Carefully remove the aluminum packet from its container and empty the salt into a 30-ml beaker. Wash the foil several times with distilled water, using the squeeze bottle and collecting the washings in the beaker, until the foil shows no more activity. Pour the solution from the beaker into a 50-ml volumetric flask (do not spill) and rinse the beaker with distilled water. Pour the rinsings into the volumetric flask and repeat until the beaker shows no more activity. Now carefully fill the remainder of the volumetric flask with distilled water and replace the stopper. Repeat the above procedure with the other sample. From your data determine the concentration of the solutions expressed in mg/ml.

Remove an aliquot portion, 10- λ volume, of the NaNO_2 solution with the pipet and place on a stainless steel planchet under the infrared lamp. Allow the sample to dry; then heat the planchet over a Bunsen burner until the planchet becomes red hot and allow it to air cool. Repeat with the $\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$ solution using a 25- λ sample.

Determine an initial activity of each isotope by means of the proportional flow counter. Determine the half-lives of Na^{24} and Mn^{56} by taking counts of each every 10 min and 5 min, respectively. Plot a graph on semi-log paper with counting rate (cpm) as ordinate vs. time (min) as abscissa. Because of the difference in half-lives of the two isotopes, use a separate sheet of paper for each, making the time scale of appropriate length in order to show a good slope.

DATA:

NaNO_2

$\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$

Weight of foil and salt _____ mg

Weight of foil and salt _____ mg

Weight of foil _____ mg

Weight of foil _____ mg

Net weight of salt _____ mg

Net weight of salt _____ mg

Concentration of solution _____ mg/ml

Concentration of solution _____ mg/ml

Initial activity (Time 0) _____ mc

Initial activity (Time 0) _____ mc

Experiment 2 - SAMPLE PREPARATION AND HALF-LIFE DETERMINATION

OBJECTS:

To demonstrate the techniques of preparing radioactive samples for α , β , and γ counting.

To illustrate the method of determining the half-life and decay constant by using radioisotopes.

DISCUSSION:

Since this experiment is divided into two parts, the discussion of theory and procedure will be dealt with accordingly.

Part 1

In the preparation of radioactive samples for counting purposes, the selection of equipment is of the utmost importance. Because of the problems arising from contamination, it is important to repeat at this time the necessity for keeping all equipment clean. The mounting of the sample, either on metal discs or plastic sheets, is the first approach to preparing the sample. In most cases the sample is placed on stainless steel plates called "planchets." They are also available in glass or plastic. It is upon the surface of the planchet that an exact volume of isotope solution is placed in order to count the sample or to determine its activity.⁽³⁾

After making up the stock solution of a selected radioisotope, a given quantity of the stock solution is transferred to a planchet by use of a "micropipette." This instrument is designed to hold an exact quantity of solution, not to deliver the quantity which is designated on the stem of the pipette. A demonstration by the laboratory instructor will be made to show the proper handling of the micropipette. Since the pipette will not deliver the full amount marked on the stem, it will be necessary to flush the pipette with a pure solution which will have little effect on the solution containing the radioisotope.⁽⁴⁾ This will insure the complete removal of the test solution from the micropipette.

Often it will be found that the sample will not wet the planchet. This can be corrected by using a wetting agent such as DuPont's Duponol.⁽⁵⁾ One drop of the wetting agent will be sufficient in wetting down the sample. The reduction of volatility of certain substances such as iodine may also be necessary. This can be accomplished by adding reagents which tend to reduce the rate of evaporation. Reference to certain publications can be made by the instructor for making up reagent solution for the purpose of reducing volatility.

The next step is drying the sample. The mother liquor is removed and only the dry form of the isotope is left. This can be accomplished with infrared heating lamps or a hair dryer. When complete evaporation is accomplished, the planchet is ready for counting. To insure a tight bond between the sample and the planchet, it is permissible to bake the planchet over a Bunsen burner, taking the necessary precautions not to volatilize the sample. The sample upon cooling is now ready for counting.

PART II. Half-life Determination

For any radioactive material the activity can be expressed as a function of time:

$$A_x(t) = A_{x0} e^{-\lambda t} ,$$

where $A_x(t)$ represents the activity of x at time t , A_{x0} represents the initial activity, and λ is the disintegration constant. For any one substance the quantity of interest is the ratio of the number of atoms at two values of the time:

$$\frac{N(t_1)}{N(t_2)} = \frac{A(t_1)}{A(t_2)} . \quad (2)$$

Hence, the use of N and A as equivalent quantities can be done without too much confusion in the case of a single component:

$$N_x(t) = N_{x0} e^{-\lambda t} . \quad (3)$$

Since radioactivity is a first-order reaction, the rate of decay is proportional to the number of radioactive atoms present. If we differentiate equation (3), there is obtained

$$-\frac{dN}{dt} = \lambda N , \quad (4)$$

where $N(t)$ has been abbreviated as N . The expression dN/dt represents the disintegration rate of the radioactive atoms. The minus sign indicates a decrease in the number of active atoms with a change in time. If we rewrite equation (3) in terms of common logs, we obtain

$$\log \frac{N(t)}{N} = -0.4343 \lambda t \quad (5)$$

or

$$\log A(t) = \log A_0 - 0.4343 \lambda t . \quad (6)$$

Therefore, if the log of the experimental activity is plotted against time t , a straight line should be produced with a slope equal to -0.4343λ . If we let $T_{1/2}$ equal to a value called half-life, the time needed for one-half of the radioactive atoms to disintegrate, then

$$N(t)/N_0 = 0.5 \quad , \quad (7)$$

and according to equation (5), $\log 0.5 = -0.4343\lambda T_{1/2}$, or $\log 2 = 0.4343\lambda T_{1/2}$. The log of 2 = 0.3010; thus

$$T_{1/2} = 0.693/\lambda \text{ (half-life formula).}$$

According to the above calculations, we then can experimentally determine the relative activity (c/m) by using a β detector or γ detector, depending on the specific type of activity emanating from the source.

PROCEDURE:

PART I. Sample Preparation

(NOTE: Always wear rubber gloves and protective goggles while working in the laboratory. DO NOT wear gloves outside of the laboratory area.)

1. From a pre-irradiated sample of manganese salt, select a packet which contains approximately 0.14 gm per sample.
2. To a clean, dry 50-ml volumetric flask, add about $\frac{1}{4}$ volume of H_2O .
3. Empty the sample of Mn into the flask, being careful not to spill the Mn salt or allow too much to adhere to the side of the flask neck.
4. Carefully rinse the foil or container into the volumetric flask, using a flush syringe of some type. Have the foil checked by Radiation Safety to determine if any residual activity is left. Fill the volumetric flask up to the 50-ml mark on the neck of the flask, thereby obtaining the stock solution.
5. Mix the solution thoroughly (no salt is observed in the flask).
6. With a micropipette calibrated in λ (lambda) (see Fig. 4), practice several times the manipulation of the pipette with water as the sample to be tested. Once you understand the best method for filling and extracting, the sample solution is then transferred from the 50-ml stock solution to a stainless steel planchet. Pipette a $10-\lambda$ and a $20-\lambda$ sample.

1. Aluminum Foil Packet
2. 50-ml Volumetric Flask
3. Tweezers
4. Micropipettes
5. Plastic Planchet Carrier with Stainless Steel Planchets
6. Plastic Case
7. Spatula
8. Scissors
9. Infrared Lamp for Drying Samples

Fig. 4

Apparatus for Experiment #2

7. With tweezers, transfer the planchet to the seat under the infrared lamp, approximately 6 to 8 in. from the light source. Heat the planchet until there is no further question in your mind that total evaporation has taken place. Remove the planchet and place, sample side upward, into the hot flame of a Bunsen burner. This will bake the sample on the surface of the disc, thereby reducing the possibility of sample loss.

8. Allow the discs to cool down enough that they can be placed in a plastic planchet holder. Be sure and mark on the plastic box, directly over the planchet, the size or volume of sample removed from the stock solution by means of the micropipette.

PART II. Half-life Determination

1. Now that the planchet is ready for counting, take the plastic case, containing the stainless planchets, to the counting room.

PROBLEM ON HALF-LIFE DETERMINATION

The counting rates listed below were obtained when the activity of a certain radioisotope sample was measured. Plot the decay curve on semilog paper and determine the following:

- $T_{1/2}$
- λ
- Counting Rate at $t = 0$
- Identify the nuclide according to the list of elements given, following the data table

Time(days)	Counting Rate (counts/min)
0	-
5	603
8	540
9	520
13	448
16	400
19	358
22	322
25	287
27	267
30	238

(a) Iodine-131, (b) Barium-131, (c) Phosphorus-32, (d) Rubidium-86, (e) Argon-37.

Experiment 3 - RADIOACTIVE DECAY MIXTURES OF INDEPENDENTLY DECAYING ACTIVITIES

OBJECTS:

To demonstrate the change in activity of two radioisotopes decaying under independent conditions.

To illustrate a method for determining the decay curves of two isotopes in a single mixture.

DISCUSSION:

If two radioisotopes, particularly one of a short half-life and one of a long half-life, are mixed homogeneously, the observed activity will be equal to the sum total of the two separate activities⁽⁶⁾:

$$A = A_1 + A_2 = C_1\lambda_1N_1 + C_2\lambda_2N_2 \quad . \quad (3-1)$$

The detection coefficients C for the two radionuclides normally are not numerically close and very often their magnitudes differ considerably. When plotting the activity of several radioisotopes in a mixture, the curve will tend to become concave toward the time zero. This occurs, because as time progresses, the short-lived isotope becomes less apparent, or less detectable, until eventually the detection system will pick up only the long-lived component. After plotting the values of activity versus time, a straight line will eventually be found and the half-life of the long-lived component may be determined from this. Now if the linear portion of the activity curve is extrapolated back to $t = 0$ and the extrapolated line is then subtracted from the original curve, the residual curve represents all the components, with the exception of the long-lived isotope. If the residual line does not give a straight line, it may be treated again in the same manner as the original procedure, so that a series of straight-line plots are obtained. In practice, it may be difficult to get a satisfactory set of straight lines unless the half-lives of the independent isotopes differ by a factor of at least five.

If the half-life of the radionuclide is either very long or very short, methods other than the one mentioned may be used. When the half-life is very long, the disintegration constant is very small and a measurable change in activity may not be detectable. The experimental activity is equal to

$$A = C\lambda N \quad , \quad (3-2)$$

and to determine the value of λ for long-lived emitters by means of equipment where the geometry and efficiency are known, it may be seen that

$$\lambda N = A/C$$

The proportionality factor C which is sometimes referred to as the detection coefficient, will depend upon the nature of the detection instrument, geometrical rearrangement of the samples and detectors, and the efficiency of the detecting instrument. Values of half-lives up to 10^{10} years have been determined. Half-lives as short as 10^{-9} sec have been determined by other methods.

PROCEDURE:

1. Calculate the expected activity of manganese and copper which have been irradiated for one hour and for thirty minutes, respectively, in a thermal flux of 2×10^{12} n/cm²/sec in the CP-5 reactor. The manganese will be used as $\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$ and copper as the pure element. The specific activity should be expressed as disintegrations/second/gram.

If the total material is present in a solution of 250-ml volume, what quantity by volume would be needed to give the activities listed in Procedure 2? Assume the efficiency of the 2π proportional counters as 40% geometry.

Two irradiated samples will be selected: one is $\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$, manganese acetate, and the other Cu metal, the pure material. In a reflux condenser, (See Figs. 5 and 6), dissolve the copper in 1:1 (by volume) concentrated nitric acid. This should take about 15 to 20 min.

1. Reflux Condenser
2. Basket Heater
3. Rheostat Control for Heater
4. 250-ml Round Bottom Flask
5. 250-ml Volumetric Flask
6. Stainless Steel Planchets
7. Plastic Planchet Carrier
8. Infrared Heat Lamp

Fig. 5

Apparatus for Experiment #3

1. Reflux Condenser
2. Basket Heater
3. Rheostat Control for Heater
4. 250-ml Round Bottom Flask
8. Infrared Heat Lamp

Fig. 6

Assembled Reflux Condenser Unit
for Experiment #3

The manganeous acetate is water soluble. Warm approximately 100 ml of distilled water. Add the acetate and dissolve with the aid of an electric stirrer. Pour into a 250-ml volumetric flask and use additional water to flush vessels; the volume of the water is not to exceed 250 ml. A similar procedure should be used with the copper solution after digestion.

2. Upon a clean stainless steel planchet, add sufficient Mn-56 solution so the activity is about 5,000 c/min. To the same planchet, add Cu-64 solution such that the activity now totals 10,000 c/min.
3. Dry the combined sample under an infrared heat lamp.
4. Measure the background for five minutes.
5. Take a 2-min. count of the planchet and repeat the procedure to insure reproductibility.
6. Check the counting system with the C^{14} or Th^{204} Standard.
7. Recheck the value for the planchet every 30 min for the remainder of the laboratory period. During the following day, take at least three (3) sample readings of the sample planchet, and repeat the procedure the third day.
8. The elapsed time for good results should not be less than three half-lives of the longest isotope.

Experiment 4 - PREPARATION OF "CARRIER FREE" SOLUTIONS

OBJECTS:

To investigate the properties of "carrier free" solutions.

To learn the methods of avoiding errors in preparing the solutions.

DISCUSSION:

The term "carrier free" applies to a solution in which all the atoms of a particular element are radioactive. As an example, a solution of $\text{MgNH}_4\text{P}^*\text{O}_4$, where the asterisk on P indicates the atoms of P are radioactive, there would be no stable atoms of phosphorus and a solution which possessed an activity of one microcurie would contain 3.5×10^{-6} μgm of phosphorus. With this small quantity of radioactivity, there is always the possibility of some of the solution or radioactivity being absorbed on the walls of a container.⁽⁷⁾

To overcome this problem, several methods are suggested. First, containers which are siliconized, that is, on which a protective silicone coating has been introduced, can be utilized.

Secondly, a procedure of presaturation may be employed. If a dilution of carrier-free solution is to be made, the container to be used is pre-flushed with approximately a 1% solution of the stable form of the carrier-free solution. It is recommended that the carrier solution be allowed to stand in the vessel for several hours.⁽⁵⁾

Procedure for Coating Glassware:

There are several silicone materials suitable for surface protection, especially Dow-Corning 200 Fluid and G. E.'s DRI-Film.

- Step I. Thoroughly clean and dry the container to be siliconized.
- Step II. Introduce a solution of the correct strength so that the vessel is completely filled. If the container is too large, swirl the solution until all the surface area is wet.
- Step III. Drain the vessel containing the silicone solution back into the stock bottle.
- Step IV. Air dry the bottle in an inverted position until the solvent evaporates completely. If desired, dry the bottle in an oven for about one hour at 100°C . This

should bake the silicone on the wall of the bottle and form a good bond of the silicone to the glass wall.

PROCEDURE:

1. Select four flasks of approximately 100-ml volume with mouth opening large enough to receive a side-window Geiger tube. Clean thoroughly with chromic acid solution, rinse, and dry.
2. Prepare one of the four flasks as described in the theory of "Procedure for Coating Glassware." Mark as #1.
3. Prepare the second one by presaturation, using Na_2HPO_4 (1% solution by volume.) Let the flask stand completely filled for about 10 min. Pour the Na_2HPO_4 solution back in the stock bottle and rinse the flask with distilled water. Mark as #3.
4. The remaining two flasks should be untreated and marked #2 and #4.
5. To each flask add distilled H_2O or carrier as shown in the chart below until the flask is 50% filled. To each flask add 75 ml of a carrier-free solution of the P^{32} isotope, and then fill each flask to the mark so that each has the same volume. Use the diluting solutions for each flask as shown in the chart below.
6. Mix the solutions thoroughly until you are sure of good homogeneity.
7. From each flask draw out one ml of solution and place on a planchet. (See Fig. 7) DO NOT DRY THIS SAMPLE. Measure the activity of the solution immediately.

1. 4 100-ml Wide-mouth Erlenmeyer Flasks
2. 2 5-ml Pipettes
3. Micropipettes (25 and 250 Lambda)
4. Stainless Steel Planchets

Fig. 7

Apparatus for Experiment #4

8. Hold the solutions in their flasks for a period of 24 hr. Repeat step #7 being very careful to take out exactly one ml of solution.

9. Drain all four flasks into a waste bottle. Allow the flasks to remain inverted for a period of 10 min. Using a side-window G. M. tube, determine the residual activity in the flasks.

Flask No.		Activity of 1 ml of Solution		Activity of Empty Flasks
		At Once	24-hr Lapse	
1	Siliconed	_____	_____	_____
2	None	_____	_____	_____
3	1% Na ₂ HPO ₄	_____	_____	_____
4	None	_____	_____	_____

Experiment 5 - SOLUBILITY MEASUREMENT

OBJECT:

To show how a radioactive tracer is used:

A. for measuring slight solubility of an otherwise "insoluble compound" (determination of a solubility product constant);

B. for determining the tendency of a relatively stable complex ion to form in a suitable solvent (dissociation or instability constant).

DISCUSSION:

A. Solubility Product Constant - If an insoluble compound (such as a precipitate of silver iodide) is shaken with water, a very small amount of it will go into solution forming silver ions and iodide ions⁽⁸⁾:

These ions are in equilibrium with the bulk of insoluble material, and their numbers remain constant as long as saturation equilibrium is not disturbed. If, however, additional AgI were introduced, some new ions would be formed. (The original ions would give place to the new ones and be re-formed as solid AgI.)

If the gram molar concentration of silver is multiplied by the gram molar concentration of iodide, the result is the solubility product constant:

$$K_{sp} = [\text{Ag}^+] \times [\text{I}^-] \quad , \quad (2)$$

where K_{sp} is the solubility product constant.

Let both Ag^+ and I^- ion concentrations be designated by X.

Then

$$K_{sp} = X^2 \text{ and } X = \sqrt{K_{sp}} \quad . \quad (3) \text{ and } (4)$$

If the known value of K_{sp} for silver iodide is 1.5×10^{-16} , the ion concentration of either Ag^+ or I^- becomes $\sqrt{1.5 \times 10^{-16}}$ or 1.2×10^{-8} molar.

In this experiment, a trace amount of radioactive silver iodide (AgI^{131}) will be prepared from a small quantity of NaI^{131} . This will be brought about by the chemical reaction involving a small amount of NaI^{131} solution

(contained in a larger quantity of KI in solution) and a solution of silver nitrate (AgNO_3). The solubility of AgI in distilled water may then be determined by the activity of the AgI^{131} portion of the precipitate which actually dissolves (that is, does not remain as a precipitate) and is in equilibrium with the undissolved AgI and AgI^{131} . From this information, the solubility product constant of AgI may be calculated.

B. Dissociation or Instability Constant - If insoluble AgI is treated with ammonia, a complex ion is formed which is known as the silver diammino ion:

Although quite stable, this complex ion does dissociate somewhat:

The extent of dissociation of the ion complex is given by the dissociation constant (also called instability constant) K_d . This enables us to calculate how much silver iodide will dissolve in a solution of ammonia (ammonium hydroxide) of known concentration. Thus,

$$K_d = \frac{[\text{Ag}^+] \times [\text{NH}_3]^2}{[\text{Ag}(\text{NH}_3)_2^+]} \quad (7)$$

for which the numerical value is 6.8×10^{-8} .

NOTE: Since the reaction (Eq. 5) obeys the law of Mass action, the two molecules of ammonia (2NH_3) becomes $[\text{NH}_3]^2$ in Eq. 7 from the law of Chemical Equilibrium given as $\frac{[\text{C}]^c \times [\text{D}]^d}{[\text{A}]^a \times [\text{B}]^b} = K_e$,

where K_e is the equilibrium constant.

If X is the concentration of silver iodide which can be dissolved by the ammonium hydroxide, then

$$X = \text{I}^- = \text{Ag}(\text{NH}_3)_2^+ \quad (8)$$

Substituting X for I^- in Eq. 2, we obtain

$$K_{sp} = [\text{Ag}^+] \times [\text{X}] \quad ,$$

from which

$$[\text{Ag}^+] = K_{sp}/[\text{X}] \quad (9)$$

Also, substituting X for $\text{Ag}(\text{NH}_3)_2$ in Eq. 7, we obtain

$$K_d = \frac{[\text{Ag}^+] \times [\text{NH}_3]^2}{X} ,$$

from which

$$[\text{Ag}^+] = K_d [X] / [\text{NH}_3]^2 . \quad (10)$$

Since the concentrations of Ag^+ ions are equal in both equations and the equilibria are the same, because they are in the same solution, we can set Eq. 9 equal to Eq. 10 and obtain

$$\frac{K_{sp}}{[X]} = \frac{K_d [X]}{[\text{NH}_3]^2} , \quad (11)$$

from which

$$X^2 = \frac{K_{sp} [\text{NH}_3]^2}{K_d} \quad \text{and} \quad X = \sqrt{\frac{K_{sp} [\text{NH}_3]^2}{K_d}} . \quad (12) \text{ and } (13)$$

Replacing K_{sp} and K_d with their respective numerical values of 1.5×10^{-16} and 6.8×10^{-8} , we obtain for X a value of $4.7 \times 10^{-5} [\text{NH}_3]$. Thus, the concentration of silver iodide will be proportional to the concentration of ammonia.

In the experiment, the solubility of AgI^{131} in a known concentration of NH_4OH will be determined by measurement of the radioactivity of the AgI^{131} in solution. From the data, the dissociation constant (or instability constant) of $\text{Ag}(\text{NH}_3)_2^+$ can be calculated.⁽⁹⁾

APPARATUS: (See Fig. 9)

- Centrifuge
- Centrifuge tubes
- Gamma-counting tubes
- Scintillation well-type counter
- Pipets (1-ml)
- Pipets (200- λ)
- Beakers (30-ml) for KI, NaI^{131} , and AgNO_3 solutions
- Beakers (400-ml) for waste and washings
- Rubber gloves

MATERIALS:

NaI¹³¹ solution
KI solution (0.05 M)
NH₄OH solutions (0.1 M and 0.5 M)
AgNO₃ solution
Distilled water

PROCEDURE:

Using a pipet, measure exactly 1 ml of 0.05 M KI solution and transfer it to a 3-ml centrifuge tube. To this add exactly 1 ml of NaI¹³¹ solution having an activity of 0.05 mc. Now add AgNO₃ solution dropwise until silver ion is present in slight excess. Mix and centrifuge (see Fig. 8). (The supernatant liquid, if counted, would have an activity of essentially zero, showing that all NaI¹³¹ has been converted to AgI¹³¹.)

Fig. 8

Centrifuge with Centrifuge Tubes

Carefully pour off the supernatant liquid into a waste beaker and wash the precipitate with distilled water two or three times using the polyethylene squeeze bottle. Place the washings into the waste beaker. Now add 2 ml of distilled water to the precipitate and equilibrate it by shaking for 15 min. Next, centrifuge the mixture and draw off exactly 1 ml of supernatant liquid, placing it into a clean gamma-counting tube. Measure the activity in the well scintillometer. Repeat the above procedure by adding a 1-ml portion of distilled water to the precipitate, equilibrating it,

and drawing off another 1 ml of supernatant liquid. Repeat, using a fresh quantity of 0.1 M NH_3 to assure agreement of counting results.

Finally, using a 0.5 M solution of ammonia, repeat the equilibration and measurement of activity on two more 1-ml portions and record the counting results (if time allows).

1. Centrifuge Tubes
2. 1-ml Pipettes
3. 500-Lambda Pipettes
4. Beakers (30 ml) for Radioactive Solutions
Beakers (400 ml) for Waste and Washings

Fig. 9

Apparatus for Experiment #5

DATA AND CALCULATIONS:

1. Weight of AgI used -

Exact molarity of 0.05 M KI solution

_____ M

Volume of 0.05 M KI solution used

_____ ml

Molecular weight of AgI

$$M = \frac{\text{millimoles}}{\text{ml}} = \frac{\text{mg/molecular wt.}}{\text{ml}} \text{ and}$$

$$\text{mg} = \text{ml} \times M \times \text{molecular wt.}$$

$$\text{Calculated weight of AgI (mg)} = \text{_____ mg} \quad (1)$$

2. Total Activity of NaI^{131} stock solution used -
 Volume of NaI^{131} stock solution used _____ ml
 Dilution Factor (total volume/volume used) _____
 Activity of 1 ml of diluted NaI^{131} solution _____ c/m
 total Activity of NaI^{131}
 stock solution used = Vol. x Dil. Factor x Activity per ml
 = _____ c/m (2)

3. Specific Activity of AgI = (1) / (2) = _____ c/m/mg (3)

4. Solubility of AgI in water -
 Activity in 1 ml of water _____ c/m
 Weight of AgI per ml of water
 = $\frac{\text{Activity per ml of water}}{(3)}$ = _____ mg (4)

Molarity of AgI = $\frac{(4)}{\text{Molecular Weight}}$ = _____ M

5. Solubility of AgI in 0.1 M NH_3 -
 Activity in 1 ml of 0.1 M NH_3 _____ c/m

Weight of AgI per ml = $\frac{\text{Activity in 1 ml 0.1 M } \text{NH}_3}{(3)}$
 = _____ mg (5)

Molarity of AgI = $\frac{(5)}{\text{Molecular Weight}}$ = _____ M

6. Solubility of AgI in 0.5 M NH_3 -
 Activity in 1 ml of 0.5 M NH_3 _____ c/m

Weight of AgI per ml = $\frac{\text{Activity in 1 ml 0.5 M } \text{NH}_3}{(3)}$
 = _____ mg (6)

Molarity of AgI = $\frac{(6)}{\text{Molecular Wt.}}$ = _____ M

7. Calculation of Solubility Product Constant, K_{sp}
 Calculated Value of $K_{sp} = X^2$ = _____
 The molar concentration of AgI in solution represented by X,
 is the molarity of AgI calculated in (4) above.

8. Calculation of Dissociation or Instability Constant for

Solving Eq. 12 (see DISCUSSION) for K_d , we have the following:

$$\text{Calculated Value of } K_d = \frac{K_{sp} [\text{NH}_3]^2}{[\text{X}]^2} = K_{sp} \left[\frac{\text{NH}_3}{\text{X}} \right]^2 = \underline{\hspace{2cm}}$$

Experiment 6 - TRACER TECHNIQUES BY EXCHANGE KINETICS

OBJECT:

To demonstrate the use of tracers in isotopic exchange kinetics.

DISCUSSION:

An isotopic exchange reaction is one in which the atoms of a specific element interchange between at least two chemical forms of the element. An example of this can be shown by using ions of different structure containing sulfur. In this case, we will use the sulfite ion $(\text{SO}_3)^-$ and thiosulfate ion $(\text{S}_2\text{O}_3)^-$ to show how the atoms of different ions may differ:

We can see that the two sulfur atoms in the thiosulfate ion are different. It is the purpose of this example to determine if the tagged sulfur, S^* , in SO_3^- will, under varied conditions, exchange with the S_1 atom or S_2 atom, or possibly with both S_1 and S_2 in the thiosulfate ion. If no exchange takes place, the following reaction will not occur:

In the experiment to be performed, the best method to demonstrate exchange kinetics is the atomic exchange between only 2 atoms of the same element and to utilize the "Quantitative Exchange Law" whose derivation is given in Nuclear and Radiochemistry by Friedlander and Kennedy, under the chapter on "Tracers in Chemical Application"⁽¹⁰⁾:

$$R = \frac{ab}{a + b} \cdot \frac{0.693}{T_{1/2}},$$

where

R = Reaction rate for the reaction $AX + BX^* \rightarrow AX^* + BX$

$a = (AX) + (AX^*)$

$b = (BX) + (BX^*)$

$T_{1/2}$ = the time for the concentration of AX^* to reach one half of the final concentration. In the specific case of a reversible reaction in which n-butyl iodide and sodium iodide are used, it is necessary to know the extent of the reaction.

The reaction time will start when the NaI^* solution is first added to the n-butyl iodide solution. Equal aliquots are removed after a given time to insure equal amounts of butyl iodide being sampled. The first sample or aliquot is removed as soon as the tagged NaI^* is added. This is done to determine if there is any indication that there is no exchange of the tagged atom. The exchange may not take place because of impurities in the mixture. If this occurs, the chemicals must be discarded and new, purer material used. The aliquots are then transferred to test tubes which will contain the stable NaI and benzene solutions. The n-butyl iodide is soluble in the benzene and will not be found in the water layer. From this procedure the relative distribution of the butyl iodide can be determined by separating the benzene layer out and counting the activity present.⁽¹¹⁾

PROCEDURE: (See Fig. 10 for Apparatus)

1. 2-ml Volumetric Flasks
2. 5-ml Volumetric Flasks
3. Micropipettes
4. Centigrade Thermometer
5. Separatory Funnels
6. Planchet Equipment

Fig. 10

Apparatus for Experiment #6

1. Select five clean, 2-ml volumetric flasks and add the following:

1 ml 0.08 M NaI
1 ml benzene

Place this in an ice bath and wait 15 min.

2. Select one 5-ml volumetric flask and add the following:

1 ml 0.08 M NaI (in 90% acetone)
1 ml 0.08 M n-butyl iodide (in 90% acetone)

Place in either the ice bath or the hot water bath, according to the one designated by the instructor. Allow this also to set for a period of at least 15 min.

3. After the elapsed time, pipette 100 λ of the NaI* solution into the 5-ml flask containing the NaI and n-C₄H₉I. Note the time at which the NaI* is added; shake the flask for 2 min.

4. Remove 100 λ of the reaction solution from the 5-ml flask at the end of 2 min., and transfer to one of the 2-ml flasks which were prepared according to step No. 1. Be sure to record the time the aliquot is removed from the 5-ml flask.

5. For those performing the experiment at room temperature (75°F), allow at least 25 min before the second aliquot is removed.

6. Remove another 100- λ aliquot from the 5-ml flask, and add to another clean 2-ml flask. Repeat this procedure at 25-min intervals until 5 samples are taken.

7. For those performing the experiment at 0°C (32°F), allow a 30-min. time interval. Repeat until 5 sample aliquots are drawn off.

8. While the samples are aging for 25 to 30 min., complete the following:

- A. Shake the 2-ml samples for 2 min. Allow the benzene to settle out or separate from the water solution.
- B. Remove the benzene layer from the test tube by means of a solution pipette or use a separatory funnel, (see Fig. 11) which ever is available at the time. Transfer to a 50-ml test tube and hold for counting.
- C. Transfer the water remaining to another test tube and rinse the 2-ml flask with a small amount of water, adding this to the original water transferred. Determine the activity of each of the test tubes.

Fig. 11

Separatory Funnels Assembled for Kinetic
Exchange Experiment (Closeup)

9. Enter the data for all samples which have been checked by
scintillation counting.

ISOTOPE EXCHANGE DATA

DATE _____

SCALER NO. _____

BATH TEMPERATURE _____ °C

TIME | ¹³¹ ADDED _____

PERFORMED BY _____

SAMPLE	TIME OF DAY	ELAPSED TIME, MIN	AQUEOUS PHASE, GROSS COUNT/MIN	BENZENE PHASE, GROSS COUNT/MIN	BACKGROUND, COUNTS/MIN	NET COUNT OF AQUEOUS PHASE	NET COUNT OF BENZENE PHASE
1A							
1B							
2A							
2B							
3A							
3B							
4A							
4B							
5A							
5B							

Experiment 7 - DIFFUSION STUDIES

OBJECTS:

To make a thorough study of atomic and molecular diffusion as applied basically to metallurgy.

To demonstrate the application of metal and nonmetal diffusion.

DISCUSSION:

It is a known fact that the submolecular and molecular particles are continuously in motion. The limitations upon this motion are due to the physical conditions of the particular material. The motion of atoms and molecules play a very important role in studies of solid-state physics and chemistry. Of the various types of diffusion that exist, the least experimented upon is self-diffusion, that is, the diffusion of atoms of like material diffusing through an interface. The first studies of self-diffusion were carried out with the aid of the Fick Diffusion Law⁽¹²⁾:

$$\frac{\delta c}{\delta t} = D \frac{\delta^2 c}{\delta x^2} ,$$

where c is the concentration of the diffusing tracer, t is the time, x is the coordinate along which diffusion rate can be measured, and D is the so-called diffusion coefficient. The value for D in the diffusion law formula is generally expressed as area per unit time. Previous studies have shown that at a temperature of approximately 200°C, the diffusion of Pb in Pb is about 10 times slower than that of Sn in Pb and about 100,000 times slower than that of Au in Pb. In practice the use of diffusion studies gives good indication of erosion and corrosion of surfaces. If one can produce intense radiation on the surface of the metals, high sensitivity can be obtained.⁽⁴⁾

PROCEDURE:

This experiment will consist of the determination of the rate of diffusion between interfaces of identical material.

Step 1. Select a 1-in.-cube block of electrolytic pure copper (99.94% pure) and determine its exact mass in grams.

Step 2. Select one of the $\frac{1}{8}$ -in. plates of the same copper and secure it to the large block. Determine the total weight of the two blocks. A good method of securing the two blocks together is to use masking tape around the edge in such a manner that the tape does not extend over either edge of the combined blocks. Number the $\frac{1}{8}$ in. plate zero.

Step 3. By using the activation formula found on page 12 of the laboratory manual, determine the activation time necessary to produce a specific activity of Cu^{64} equal to $75 \mu\text{c}$. Since the copper will be activated in the thermal neutron flux of CP-5, it will also be necessary to determine the activation time for Cu^{66} , which will also be produced, and will have a specific activity of $2.87 \mu\text{c}$. The reactions involved are $\text{Cu}^{63}(\text{n}, \gamma) \text{Cu}^{64}$ and $\text{Cu}^{65}(\text{n}, \gamma) \text{Cu}^{66}$, respectively. The copper blocks should be allowed to stand for at least two hours in order to allow any Cu^{66} ($T_{1/2} = 5.1 \text{ min}$) to decay to prevent any interference with the normal counting of the Cu^{64} ($T_{1/2} = 12 \text{ hr}$). The Cu blocks that you will use will be prepared ahead of time and should correspond to the precalculated specific activity.

Step 4. Select a series of the nonactive copper blocks, $\frac{1}{8}$ in. thick, which, when combined, will give an overall thickness of $\frac{1}{2}$ in. The plates should be numbered one through four. Mount the $\frac{1}{8}$ -in. plates on the needle supports given to you as part of the apparatus. The main activated block should then be mounted on the needle stand supplied for this purpose.

Step 5. The mounted blocks are then introduced into the muffle furnace in such a position that the "C" clamp (see Figs. 13 and 14) is opened to a point that the jaws will just be out of contact with the mounted copper. In this position, the sample is now ready to be heated (see Fig. 15). The temperature is set at 500°C and the system is allowed to be heated in this position for one hour. At this time, with the use of asbestos gloves, the "C" clamp is closed while still in the furnace and the nonactive plates are allowed to come in contact with each other. The time is noted and the samples are then heated for a 2-hr period. This will enable the radioactive atoms to move more rapidly through the complete system.

At the end of 2 hr, remove the four plates by opening the "C" clamp and allow the $\frac{1}{8}$ -in. plates to be separated by some mechanical tong system. Allow at least a 15-min cooling period before the plates are measured for activity. During this 15-min period, prepare four more nonactive plates in the originally described position. Each plate should be secured into position so that there will be no possibility of their falling. Increase the temperature of the furnace to 900°C and allow the system to heat for one hour before clamping the plates together.

Step 6. Measure the activity of the plate #0 which is attached to a large block of copper. This is to represent the activity of the original radioactive system. This is to be accomplished before the next set of plates are introduced to the furnace. During the one hour preheat time for the second set of plates, this time should be used to check the activity of the first set of plates that were tested.

Step 7. Using the setup indicated in Fig. 12, measure the total activity of each $\frac{1}{8}$ -in. plate numbered 1 through 4. Enter the values determined for each plate on the data sheet. Be sure to check the background often, at least every 15 min. Since the net count rate is important, make sure your values are accurate.

Fig. 12

Fig. 13

"C" Clamp Holding Copper Plates

Fig. 14

"C" Clamp Assembly with Copper Block
and Copper Plates

- 2. "C" Clamp Assembly - Upright Position
- 3. Muffle Furnace for Heating Copper Blocks

Fig. 15

Assembly for Heating Copper Blocks

DATA SHEET D3

1. Specific activity of Cu^{64} cube _____ c/min/mg

2. Activity of copper plates (after heating)

Plate No.	Gross Count	Background	Net Count/min
0	_____	_____	_____
1	_____	_____	_____
2	_____	_____	_____
3	_____	_____	_____
4	_____	_____	_____

3. Plot a curve of distance vs. activity (A_t)

Show All Calculations on This Page

Experiment 8 - DETERMINATION OF DEPOSIT THICKNESS

OBJECTS:

To demonstrate a simplified method of determining thickness of metal deposits on metal.

To discuss other methods which are available as a comparison.

DISCUSSION:

Depending on the overall thickness of a deposit, various methods have been suggested to determine the thickness of specific materials. In the case in which pure (>99%) materials are used, the method described in this experiment is readily acceptable. When the purity is less than 99%, other methods may be more suitable.

Thicknesses of deposits can, to a certain degree of accuracy, be determined by using a beta or gamma gage. The theory of this application is based upon the passage of radiation through the substance (transmission) rather than upon the reflection of radiation. If it is assumed that the change in intensity of radiation passing through a substance is of an exponential form, then an experimental determination of the change in intensity or activity permits the thickness of a specific material to be estimated by the following formula:

$$I = I_0 e^{-\mu x} \quad , \quad (8-1)$$

where I equals the resulting intensity, I_0 the initial intensity, μ the linear absorption coefficient, and x the thickness of the substance through which the radiation passes. By standardizing the base material, any change in intensity from that precalculated for the base material would indicate a change in thickness x of the substance being added as a deposit. This may appear as a rather indirect method of approach; nevertheless, the method is accurate enough.⁽⁴⁾

The absorption process is itself quite complex. It involves ionization, electron scattering, backscattering, and loss of energy by excited states of the electron. Of these, the most important in beta absorption is backscattering. Assuming a deposit thickness x on a thick base material, the backscattering radiation can be shown to be given mathematically by

$$\frac{I}{I_0} = \frac{B_1}{\mu_1 + \lambda_1} \left[1 - e^{-(\mu_1 + \lambda_1)x} \right] + e^{-\mu_1 x} \left(\frac{B_2}{\mu_2 + \lambda_2} \right) (e^{-\mu_2 x}) \quad , \quad (8-2)$$

where the symbols are defined in accordance with Fig. 16.

Fig. 16

Depending on the circumstances, the formulation of more complex back-scattering can be obtained, but will be of little consequence.⁽¹³⁾

ELECTROPLATING: (see Fig. 18)

The following solution will be used as the electrolytic bath for the plating of copper on iron (steel plates). The iron plates are submerged into the electrolyte and a current density of 3 to 10 amp/ft² will be maintained:

Chemicals	Quantity, oz/gal
CuCN	3
NaCN	4.5
NaNO ₃	2
NaCN (chloride-free NaCN)	1

PROCEDURE:

- Step 1 Select an iron plate, approximately 20 mils in thickness, as the base material. Using a Co⁶⁰ source determine the I value [the linear absorption coefficient for Fe for gamma rays from Co⁶⁰ (≈ 1.5 Mev) should be taken as 0.40 cm⁻¹]. See Step 4 for absorption determination.
- Step 2 Enter the experimentally determined value in data section. Using an electrolytic bath, coat the iron base with copper. In order to deposit the copper on one side, coat one side of the iron with a thin coating of silicone grease. Plate an even coat of copper on the iron by leaving it in the bath for at least 2 min. At the end of the plating time, carefully remove the protective silicone coating.

Step 3 Run a recount on the γ absorption by the same method as in Step 1 to determine the change in absorption due to the addition of the copper coating. By using formula (8-1), calculate the value for the thickness x which would be necessary to reduce the intensity by the experimental value. The value of μ for copper is $0.058 \text{ cm}^2/\text{gm}$. Introduce this value in the space allotted on the data sheet.

Step 4 See Fig. 17 for absorber setup. Diagram is drawn to scale in terms of distance, not size.

Fig. 17

Suggested Method for Determination of Thickness
by Absorption Method

1. Tray for Holding Electrolytic Solution
2. Copper Cathode
3. 20-mil-thick Steel Plate
4. Anode Contact

Fig. 18

Apparatus for Electroplating

PROBLEMS:

1. A certain material has an absorption coefficient μ of 0.10 cm^{-1} for a certain type of penetrating gamma radiation. By what percentage would a beam of this radiation be reduced by passing through one foot of this material?
2. An unshielded source emitting 1-Mev γ produces a dosage rate of 500 mr/hr at one foot. What individual thicknesses of Pb and Al would be needed to reduce the dosage rate to 49.5 mr/hr?

DATA SHEET

a) Count Rate for Fe Sheeting

a) _____ c/ μ gb) _____ c/ μ gc) _____ c/ μ gAvg. _____ c/ μ g

b) Count Rate for Cu Deposit

a) _____ c/ μ gb) _____ c/ μ gc) _____ c/ μ gAvg. _____ c/ μ g

c) Change of Intensity (b/a)

$$\frac{I}{I_0} = \underline{\hspace{2cm}}$$

d) Cu Deposit Thickness

$$\frac{I}{I_0} = e^{-\mu x}$$

_____ cm

Experiment 9 - BLENDING EFFICIENCY

OBJECT:

To study the use of tracer techniques in measuring the efficiency of blending equipment as specifically related to an industrial application.

DISCUSSION:

"There is an optimum time for every blending operation. Too little gives a non-uniform product; too much is uneconomical and slows production. Tracer tests on any operation will determine this optimum time and result in worthwhile economies in many large manufacturing industries."

--from Isotopes Solve Mixing Problems by D. E. Hull, B. A. Fries, J. G. Tweksbury, and G. H. Keirns of the California Research Corp., Richland, Calif. as published in *Nucleonics*, 14 (5), (May 1956).

Methods for studying the efficiency of various industrial mixers, blenders, mills, stirrers, agitators, etc., now involve the use of tracers as a means of improving this type of equipment. In turn, this has led to overall product improvement in numerous process industries (such as petroleum, paint, cement, and paper) in which tracer studies have been made on various applications.

As an example, in the paint industry the efficiency of the blending equipment could not be determined by a single process. The major problem was to distribute pigments equally throughout the paint slurry. The method which was previously used, and realized to be most tedious, was a process of applying the blended mixture to sample boards and allowing them to dry in normal fashion. The finished sample was tested for hardness, color stability, pigment distribution, and related properties by comparison with previously prepared standards.⁽¹⁴⁾

This process often involved weeks of continuous checking to arrive at an acceptable blended form. However, today much of this process can be controlled within more convenient limits by the use of radioactive tracers. On the whole, this application typifies one of several hundred specific instances where radioisotopes can improve the operation of industrial concerns utilizing blending equipment.

In this experiment, the application of tracer techniques to two specific kinds of blending equipment, the ball mill and the paddle mixer, will be investigated. In the ball mill, a dry mixture of fine sand and ammonium phosphate will be employed, whereas in the paddle-type apparatus a liquid slurry of Celite and ammonium phosphate will be used.

Ordinarily, consideration should be given to the chemical effect of the isotope upon the ingredients of the mixture and, ideally, one of the ingredients or phases should be made radioactive. In this case, the radioactive ingredient will be made part of one phase, and it is assumed that no chemical reaction takes place between the two phases. In the case of the liquid slurry, the liquid is labeled by dissolving radioactive material in it which remains in solution during the mixing process.

Investigation of the dry mixture will be carried out through use of the isotope P-32, which will be introduced in a trace amount in the form of dry ammonium dihydrogen phosphate, $(\text{NH}_4)\text{H}_2\text{P}^*\text{O}_4$, a soluble compound. Samples of the dry batch will be taken from the ball mill at periodic intervals and placed in a given volume of water. The soluble radioactive phase will dissolve; after centrifuging the mixture, an aliquot portion of the supernatant liquid may be removed and planchatted for counting. The activities of subsequent samples which are successively uniform will indicate completeness or thoroughness of mixing and thus homogeneity of the bath.

For the liquid slurry, the aqueous phase will be tagged by adding $(\text{NH}_4)\text{H}_2\text{P}^*\text{O}_4$ to the water in a trace amount. The solid phase, Celite, may be separated by centrifuging, and an aliquot portion of liquid may be removed, planchatted, and counted. Again, the activities of subsequent samples should become more uniform, indicating approaching homogeneity of the slurry.

APPARATUS: (See Fig. 19 and 20)

- Ball Mill
- Paddle Mixer (multiple type)
- Centrifuge
- Centrifuge Tubes (40 and 15 ml)
- Pipets
- Planchets, stainless steel
- Beakers (1000, 600, 50, 30, 5 ml)
- Rubber gloves

MATERIALS:

- White sand (50 μ dia, approx.)
- Stable ammonium dihydrogen phosphate, $(\text{NH}_4)\text{H}_2\text{PO}_4$
- Radioactive ammonium dihydrogen phosphate, $(\text{NH}_4)\text{H}_2\text{P}^*\text{O}_4$
- Celite
- Distilled water

1. Ball Mill for Blending
Porcelain Balls - Blending Media
2. Electric Mixer with Rheostat Control

Fig. 19. Apparatus for Experiment #9

2. Paddle Mixer (Multiple Type)
4. Pipettes (100 ml and 250 ml)
5. Stainless Steel Planchets
6. 600-ml Beakers

Fig. 20. Apparatus for Experiment #9

PROCEDURE:

A. Ball Mill

Into the ball mill, place 1 kg of fine sand and 490 gm of $(\text{NH}_4)\text{H}_2\text{PO}_4$. Now add 10 gm of $(\text{NH}_4)\text{H}_2\text{P}^*\text{O}_4$ and start the ball mill. Allow it to run for 2 min and, using the measuring cup provided, remove an exact dry volume which will represent between 4.1 and 4.5 gm of the mixture.

Place this into a 40-ml centrifuge tube and add enough water to produce a total volume of 30 ml. Equilibrate the mixture in order to dissolve the salt; then centrifuge for 5 min and withdraw a 50- λ sample of the supernatant liquid. Planchet, dry, and count the sample for activity. Dump the remaining mixture from the centrifuge tube into a waste beaker and rinse in order to get rid of residual activity.

Operate the ball mill for another 2 min and repeat the above procedure. Do this for additional 2-min periods until consistent activities are obtained.

B. Paddle Mixer

Select a clean 600-ml beaker. To this add 10 gm of Celite, a diatomaceous silica compound, and 5 gm of $(\text{NH}_4)\text{H}_2\text{P}^*\text{O}_4$. The ammonium dihydrogen phosphate will serve as a tag for the water phase.

Next, add a total volume of 400 ml of water to the total combined dry material. Mix the slurry with the paddle type mixer for exactly 2 min. Allow the mixture to stand at the end of this period in order to permit the major body of the Celite to settle out. This should take place in 5 to 10 min.

Now draw off 5 ml of the supernatant liquid and centrifuge in a 15-ml centrifuge tube. Extract a 100- λ sample from the centrifuge tube and prepare it for counting.

Return the remainder of the solution from the centrifuge tube to the 600-ml beaker and repeat the mixing for another 2-min period. Rinse the centrifuge tube into a waste beaker in order to get rid of residual activity.

This part of the experiment should consist of at least three 2-min runs. The increase in counting rate should be directly proportional to the increase in mixing time for a 100% efficient blending system.

CURVE:

Plot Activity as ordinate against Time as abscissa in order to simulate a strip chart which might be utilized in connection with a mixing operation.

Experiment 10 - BETA AND LIQUID LEVEL GAGING

OBJECTIVE:

To show the principal techniques employed by industry in using radioisotopes for determining thickness and for level gaging.

DISCUSSION:

In thickness gaging, the key elements of the gages used are a radioisotope source (usually sealed in a container) and some type of detection device. In most applications, the thickness gages employ the source on one side of the material being tested and a detector on the other side. This type of application is referred to as "transmission gaging"⁽¹⁾ (see Fig. 21)

ADVANTAGES:

- 1- RADIATION SOURCE SELECTED TO SUIT MATERIAL
- 2- NO CONTACT - NO TEARING - NO MARKING MATERIAL
- 3- RAPID AND RELIABLE

Fig 21

Radioactive Source for Gaging Thickness

Some thickness gages work on the principle of reflected radiation. The source and the detector are placed on the same side of the material being measured in such a manner that the radiation will pass through the material, strike a steel roller or plate, and be reflected back through the material into the detector. This type of measurement is referred to as "reflected gaging" or "backscattering" (see Fig 22).

In some applications, a series of gages are employed. The gages can be used to control the thickness of a base material as well as the coating thickness which is being applied to the base material. Radioisotope thickness gages are most useful for continuous measurements when it is desirable to avoid contact with the material being tested. This normally occurs when the material being tested is moving very fast in a horizontal or vertical position (15)

Fig. 22

**Radioactive Source for Reflection
(Backscattering) Thickness Gage**

The theoretical basis for beta gaging depends on the rate of absorption in the material being tested. The radioactive source and the detector are positioned at a given distance from each other in such a manner that the space between them is fixed. This space is referred to as the "air gap" (see Fig. 25). By determining the maximum voltage that can be produced through the air gap between source and detector, a value for one-hundred per cent transmission can be determined. When materials are placed in the air gap, there is a reduction in transmission of the radioactive particle and therefore a reduction in the voltage. The reduction of one-half of the total transmitted activity through a specific material would constitute a 50% reduction in voltage and is referred to as the half-value thickness (HVT). The half-value thickness is the thickness of a certain material which is required to reduce the intensity of radiation by fifty per cent:

$$I_d/I_0 = 0.5 \quad , \quad (10-1)$$

where I_d is the intensity observed through an absorber of thickness d , and I_0 is the measured intensity without absorber. The half-thickness is given by

$$d_{1/2} = 0.693/\mu \quad \text{or} \quad d_{1/2} = e^{-\mu x} \quad (10.2)$$

where μ is the linear absorption coefficient and x the surface density. More frequently, the half-thickness is expressed in grams per square centimeter, and then is equal to $0.693\rho/\mu$ and varies about as A/Z , where A is the mass number and Z the atomic number.

The purpose of this experiment is, first, to determine the absorption coefficient for aluminum and, second, to use formula (10-2) and determine the half-thickness of the same material.

PROCEDURE:

There are many different types of industrial beta gages now on the market. Through the courtesy of Radiation Counter Laboratories, Inc., Skokie, Illinois, USA, their Nuclear Thickness Gauge, Model No. 51000, was set up in our laboratory for experimental purposes. The gauge is equipped with a krypton-85 source. The "C" frame is designed with a 24-in. throat.

1. Before determinations can be made, the electronic control panel must be checked to determine if the system is operating correctly (see Fig. 23). This is accomplished by using the dial indicated by the term "External Zero." The ten positions listed below must be checked by setting the control in the positions stated and noting if the values read are approximately the same as the prestandardized values:

<u>Position</u>	<u>Standard Values</u>	<u>Position</u>	<u>Standard Values</u>
V4B	2.3	HT	2.3
V4A	3.0	HT	2.3
V2B	1.8	External Zero	0.0
V2A	2.0	RV1	0.0
V1	1.2	RV2	0.0

Once the control panel has been checked out, the gaging system is ready to be calibrated.

Fig. 23

Control Panel Opened for Calibration. External Zero Calibration Knob Near Top Left Side of Control Panel. (Courtesy of Radiation Counter Laboratory)

2. The krypton source is shielded within the upper arm of the gauge. To expose the source, turn the release knob to the open position. The dose rate at the front, and in direct contact with the gauge, is approximately 2 roentgens. Be sure that no worker stands at a distance less than two feet from the source. When both operation positions are on, that is, the control panel and the gauge, the weight band (WB) on the control panel should be set to zero (see Fig. 24). If the needle on the thickness control is not at zero then the operator must compensate for the decay of the radioisotope by using the decay setting (see Fig. 23). Once this is accomplished the system is ready to operate.

Fig. 24

Control Panel Closed. Zero Indicator Shown at Top of Panel and the Weight Band (WB) Shown at the Bottom of the Panel. (Courtesy of Radiation Counter Laboratory)

Fig. 25

Beta Thickness Gauge Shown in Above Photograph. The Air Gap Can Be Seen as Well as the Grilled Voltage Plate Located at the Bottom of the Air Gap. (Courtesy of Radiation Counter Laboratory)

3. Measure the thickness of the aluminum foil with a 1-in. micrometer and record the thickness in mils.

4. Calculate the surface density of the aluminum. Units for this value would be mg/cm^2 . If you were to start with a 3-mil-thick aluminum foil, it would show a reading of 45 on the weight band in order for the needle to return back to zero. To increase the reading to 50, which would represent a half-value thickness, then

$$3/45 \text{ equals } 1/15 \text{ mil/WB};$$

5 divisions of WB must be increased.

Therefore,

$$(1/15 \text{ mil/WB}) (5 \text{ WB divisions}) = 0.33 \text{ mil} ,$$

so that

$3 + 0.33 \text{ mil} = 3.33 \text{ mils thickness}$ would be needed to give a half-value thickness.

To determine the absorption coefficient for aluminum,

$$\frac{2700 \text{ mg}/\text{cm}^2/\text{cm}}{394 \text{ mil}/\text{cm}} = 6.87 \text{ mg}/\text{cm}^2/\text{mil} ;$$

then,

$$(6.87 \text{ mg}/\text{cm}^2/\text{mil}) (10/3 \text{ mil}) = 22.9 \text{ mg}/\text{cm}^2$$

which is the surface density. Using formula (10-2)

$$d_{1/2} = e^{-\mu x}$$

$$0.5 = e^{-22.9\mu}$$

$$e^{22.9\mu} = 2 .$$

Taking natural logarithms,

$$22.9 \mu = 0.693$$

$$\mu = 0.693/22.9$$

$$\mu_{\text{Al}} = 0.0303 \text{ cm}^2/\text{mg}$$

5. Once the thickness gauge has been calibrated, any specific setting may be made to determine if a continuous sheet passed through the air gap is of uniform thickness. Once the determination has been made for one sheet of aluminum foil, it is recommended that this procedure be repeated using two, and then three, thickness of the foil in order to verify the accuracy of the instrument. Enter all data on the sheet below.

INFORMATION NEEDED FOR MATHEMATICAL CALCULATIONS:

- a) Specific gravity of Al foil - 2.829 gm/cm^3
- b) Voltage output from Beta Gage - 50 V
- c) Foil thickness _____ mils
- d) Surface density of Al foil _____ mg/cm^2
- e) Value of μ for Al foil _____ cm^2/mg
- f) WB setting for one thickness _____
- g) WB setting for 2 thickness _____
 WB setting-experimental _____
- h) WB setting for 3 thickness _____
 WB setting-experimental _____

Experiment 11 - SPECIAL HANDLING OF RADIOISOTOPES IN HOT CAVE OPERATION

OBJECTS:

To acquaint one with hot cell operations.

To demonstrate a method of radioisotope encapsulation of Co^{60} , using master-slave equipment.

DISCUSSION:

Any country which plans on or has already existing a power reactor will at some time tend to enter the field of radioisotope production. Under this category one must include the correct method of handling high-activity material, especially gamma-emitting sources. Recommendations have been made, internationally, regarding the necessity of hot cave facilities when radioactive sources exceed 800 millicuries of activity. When working with kilocuries of gamma activity, specially designed hot caves will be needed. In any operation in which special manipulations are used, it is important to be completely familiar with the regulations set forth by Industrial Health Service for specific cave facilities.⁽¹¹⁾

In starting the operation for a special application of handling radioisotopes, the operator should be completely familiar with the procedure of operation as well as with the exact equipment needed. For this reason a mockup or a simulated condition should be set up beforehand to acquaint personnel with the exact procedures to be followed. A trial run should be carried out to check all possibilities against misjudgement. In some cases, particularly when new research application is to be made, it may be necessary to use a nonconforming procedure. In this case, during the course of the work re-evaluations at intervals of time should be made to again reduce the possibility of doing something which the procedure does not call for.⁽¹⁶⁾

In the process of encapsulation, an operator must consider five basic points:

1. the specific activity of the sample;
2. the sample size;
3. the type of capsule to be used;
4. equipment needed for this amount of activity; and
5. clean up of cave area following the encapsulation.

Under the Procedure for this experiment, all of these important points will be mentioned.

The individual should become thoroughly acquainted with the equipment needed for encapsulating a Co^{60} source. Therefore, it is of the utmost importance that all personnel working on this experiment pay complete attention to the Hot Cell Supervisor.

PROCEDURE:

1. If you do not have your personal monitoring system, film badge and dosimeter, obtain them from Industrial Health Division before entering cave area.
2. Special clothing may be necessary for the operation; this can be obtained from the person in charge of cave operations.
3. An area behind the cave is to be used for a mockup, simulated operation to check all procedures to be used for the cave operation. All the tools and necessary items to be used in the cave should be placed in the mockup area. A picture of the equipment necessary for the operation will be included and a complete coverage of the use of this equipment will be given.
4. A complete survey of the cave area is then made. The work area should be as clean as possible. The Co^{60} source should be of high purity and should not become contaminated with isotopes that may be present in the cave.
5. Returning to the front of the cave and using the manipulators, check the complete procedure again.
6. A lead-shielded container will be used to store the Co^{60} source until it is used in the experiment. The container is moved into the cave area by means of a mechanical lift. The lead container is centered in the cave work area so that there will be the least amount of effort in removing the Co^{60} source.
7. Carefully remove the lid and by mirror manipulation place the wafers on the holder specially designed for stacking. (See Fig. 27).
8. By observing the makeup of the capsule (see Fig. 26), one can see the basic structure and material design of the encapsulating tube. The wafers or rings of cobalt-60 are placed in a holder (see Fig. 27) so they can be inserted into the capsule by pushing them into position with a special tool. The first spacer is placed into the main cylinder, making sure the spacer is at the bottom of the tube. The wafers are then inserted into the cylinder and observation is made to determine the correct seating of the Co^{60} . Next insert the last spacer and then introduce the cap to make the assembly complete.

Fig. 26
Cross Section of Co^{60} Capsule

Fig. 27
Special Apparatus for Co^{60} Alignment

9. A special tool is then placed into position for crimping or sealing the tube (see Fig. 28). Place the crimper exactly 3 mm from the joint of the cap and main cylinder, toward the end of the cylinder; by rotating and applying pressure on the crimper, seal the cap. Place the capsule in the lead container supplied.

10. Carefully remove all working tools from the cave; have each piece monitored by the Radiation Safety man to insure that excess contamination does not exist.

11. Cleanup of the cave area is most important; be sure all tools are out and any loose material is properly stored.

12. BE SURE TO CHECK YOUR TIME OF OPERATION.

Fig. 28

Apparatus for Experiment #11

CAVE JOB OUTLINE FOR BETA-GAMMA FACILITY - D-301

1. Obtain dosimeter and film badge to be worn at all times in the cave area.
2. Obtain issue of clothing suitable to the work to be done.
3. Get acquainted with I.H.S. personnel and learn all general and special rules in force for the cave facility.
4. Obtain necessary tools and miscellaneous items of equipment and make a mockup in the service area behind the cave. Check and double check plan of operation.
5. Obtain survey of cave interior before entering to set up equipment.
6. Move equipment into cave.
7. Return to front of cave and, using manipulators (see Fig. 29), check the procedure again.

Fig. 29

Manipulators - as Viewed From
Exterior of Senior Cave

8. Prepare shipping container (pot or cask) for transfer of hot samples into cave. Casks with horizontal chambers can be unloaded through the cave's side ports if sample size permits. If samples are in cask and will be inserted through cave port, have I.H.S. personnel monitor the operation.

9. Commence work. Stop at intervals to re-evaluate procedure if work is not straight forward.

10. Check encapsulated source for leakage.

11. Transfer encapsulated source to shipping container and have it surveyed for shipment by I.H.S.

12. Perform remote cleanup in cave with manipulators and prepare hot waste for disposal. Remove hot waste from cave according to approved waste disposal procedures. Have I.H.S. survey cave after waste disposal.

13. Perform final cleanup and leave cave and equipment in proper condition for next job.

MATERIALS AND APPARATUS (Numbers refer to Fig. 28)

Co⁶⁰ Source (Wafer Shaped)

Hot Cave Cell

Manipulators

1. Stainless Steel Capsule
2. Tweezers
3. Magnifying Glass
4. Needle (Embedded in Wood Handle)
5. Micrometer
6. Steel Block for Aligning the Wafers
7. Crimping Tool
- Lead Pig
8. Mirror
9. Vise for Holding Capsule
10. Hammer

Experiment 12 - AUTORADIOGRAPHY

OBJECT:

To investigate the process of autoradiography and to become familiar with a basic method and procedure for obtaining an autoradiogram.

DISCUSSION:

Another important instance for which radioisotopes are applied to advantage is in the field of autoradiography. The word "auto" means "self" and the term "autoradiography" is used to describe a situation wherein a radioactive specimen is caused to take a picture of itself, thus revealing certain defects if present. This is in contrast to "gamma radiography" which implies the use of a strong, independent γ -emitting source as an "isotopic camera" to penetrate a thick, dense material, such as a steel cast or welded section, and thus disclose certain flaws which would otherwise remain hidden.⁽¹⁷⁾ Gamma radiography constitutes an important industrial use for high-level radioisotopes, such as cobalt-60, in the 15 to 20-curie range. Its use has eliminated much of the larger and costly X-ray equipment often used for this purpose. Many of the methods using X-ray and isotopic radiography are well known.

In general, for autoradiographic work, samples may be prepared in one of the following ways:

1. by having the specimen itself activated;
2. by coating the specimen with a radioactive material;
3. by adding a small amount of radioactive material to the specimen; for example, radioactive gold-198 could be added to an aluminum billet to show the cooling boundary.

(In botanical autoradiography, the radioactive material, in solution, is usually taken up by the root of an experimental plant.)

In this experiment we will employ the first method. In this case, a small piston ring which has been activated in the CP-5 reactor for a period of 5 days will be brought in contact with a photographic emulsion, and will thus be caused to record its own image. This ring, of the oil-slotted type, is made of steel and therefore contains mostly iron. During activation about $2\frac{1}{2}\%$ of the iron will be changed to Fe-59, which emits β^- and γ radiation. It might be mentioned here that it is not always advantageous to have strong γ radiation because certain types of flaws will not always be delineated well with radiation of high penetrability. In fact, even some β^- particles of high energy have too long a path in the photographic emulsion to produce high definition.

It should be pointed out that whereas knowledge of the process of autoradiography is well established - it dates back to the time of Becquerel - today it is still as much of an art as it is a science. Much work in this field can be done to create new methods of approach.(18)

MATERIALS AND APPARATUS:

- Kodax type KK films for autoradiography
- Light-tight box or Dark room
- Activated piston rings
- Several pairs of tongs
- Developing solutions and related facilities
- Industrial gamma-radiographic negatives for class examination.

PROCEDURE:

Extended time exposures of the activated piston rings will be made. Since the exposure time differs for each application, there is no set "exposure time" for the material. It is possible that the optimum time for exposure for this particular activity may exceed the lab time. The only method of determining the best exposure time would be by trial and error.

The experiment will be carried out in groups of two. The first man, stationed in the dark room, will remove one of the special sensitive film sheets from the package and place it on a flat surface. (Handle films by edges as emulsion is on both sides.) Outside the dark room, the other man, using tongs, will remove an activated piston ring from the lead "pig" and carry it into the dark room. The first man will be ready with tongs to take the ring from him and set it on the film. Both men will then leave the dark room.

According to the manufacturer, a definite exposure time for these emulsions has not been worked out. We shall, therefore, investigate this by having a series of 15-, 30-, 45-, and 60-min exposures designated for each of these sensitive type KK films. Other types of film which are recommended for Autoradiographic study are Kodak Autoradiographic Stripping Film Type NTB, used mainly for beta emitters; plate type A, used for beta and gamma emitters; Type No-Screen, used with beta and gamma emitters. The selection of suitable film is made by the cooperation of film manufacturer, generally with the Special Sensitized Division.

If time permits, the films will then be developed. Meanwhile, a review of some of the common flaws occurring in industrial welding, such as cracks, porosity, slag inclusions, and the like, will be held. An inspection of some negatives from a large midwestern fabricator of heavy pressure vessels (catalytic cracking towers, tanks, and heat exchangers) will supplement the discussion.

Experiment 13 - ENGINE WEAR STUDIES

OBJECT:

To study the methods and application of transient piston wear in single cylinder engines using Fe^{59} as the radioisotope.

DISCUSSION:

One of the major problems which confronts the automotive engineer, automobile manufacturer, and producer of small gasoline engines, all of which incorporate the use of a piston drive system, is that of transient wear and steady-state wear. The petroleum industry, as a supplier of lubricants, also takes a great interest in this particular problem.⁽¹⁹⁾

Normal procedure for any type of wear test consists of determining gravimetrically the loss of weight of the piston ring determining the metallography of piston cylinder, and determining the difference in lubrication from the normal. This is a continuous problem with the above-mentioned firms. Much of the work which is carried out in the study of engine wear by means of radioisotopes is conducted by the manufacturer, and any additional study in this field is generally more than welcomed.⁽²⁰⁾

The foremost work in this field of study deals with the determination of maximum wear under maximum load of the engine. At present, the major work is aimed at the elimination of excess wear at the maximum rpm rating for a specific engine. It has been found that for wear tests, which are conducted at various speeds, the greatest wear is exhibited in about a six (6)-min startup. After this normal startup time, the engine has reached optimum lubrication and there is much less wear over a longer period of time. As in Fig. 30, the time of run is plotted against relative total wear expressed in %.

Fig. 30

During a 2-hr run from cold startup, the wear on the piston ring exhibited a 75% total wear after a 6-min run, in which new piston rings and new piston cylinder walls were used.

Previous work on engine wear and piston ring wear showed that the greatest relative wear on any type of standard engine is exhibited in the first 10 min of the transient run. This part of the experimental research was carried out under constant load and fixed rpm. In order to determine the change in wear, the rpm can be altered or increased up to and beyond the maximum rated value for rpm. Examination of the motor, under constant load and varied speeds, can show a definite wear ratio if relative wear is plotted against time and includes change of speed as a third factor. In this particular application, it has been found that a change of speed shows a definite wear increase, but the wear has been reduced considerably by more efficient lubrication over a longer period of time (see Fig. 31).

Fig. 31

A note of importance, as can be seen in Fig. 31, is that the change of speed and the change of wear rate, tends to decrease as the time increases; that is, a change in speed does increase the relative wear in proportion to

running time and this proportion would be inverse according to the figure. This can be readily seen by reference to letters (a, b, c, and d). In the region rated greater than the maximum rpm of the engine, the heat produced cannot be dispersed into the air fast enough; therefore, the piston ring has a tendency to expand more than permitted and the wear on both ring and engine become the greatest.

PROCEDURE:

I. After preliminary determination of the specific activity (c/min/mg) ρ of the activated piston ring, the piston is removed from the motor to be tested (see Fig. 32). Unbolt the cylinder head and carefully remove, exposing the piston. Remove the oil ring, which is the bottom ring of the three. Use a special tool for removing. The activated ring should have a dose-rate of about $1\frac{1}{2}$ r at 2 in. (γ radiation). If at all possible, a tachometer should be attached to the drive shaft of the engine in order to show if a constant rate of speed is being maintained. Replace the nonactivated ring with one of the active rings - make sure the correct edge is pointing downward. Replace the piston carefully, making certain the two compression rings, as well as the oil ring, are seated tightly into position before pushing the piston into place. Once the piston is seated in the cylinder, reassemble the piston arm to the drive shaft and replace the cylinder head. The engine is now ready for wear testing.

Fig. 32

Single-cylinder Engines used in Experiment #13

II. At intervals of 1, 5, 15, 30, 45, and 60 min, draw off three samples of oil into 3 separate, clean, gamma-counting tubes, by the method prescribed below.

1. Release 15 ml of oil into a waste container. This is to preflush the draw-down tube, in case any particulate matter may be present in unusual quantity in the plastic tube. This also insures better homogeneity. The oil, which is drained for samples, will start at the same point from the crankcase.

2. Draw off exactly 2 ml of oil from the down-spout into a gamma-counting tube. The flow of oil should be directed in such a manner that there will be a minimum amount of oil adhering to the walls of the counting tube.

3. The three tubes should be marked, for example, as #1, #2, #3, or A, B, and C, for each time the oil samples are drawn out. Place them in position for counting and note net counting for each sample.

4. After a one-hour run, compile all data in such a form that the values experimentally determined can be plotted as activity against time.

5. From the values of specific activity $c/\text{min}/\text{ml}$ or $c/\text{min}/\text{mg}$ determine the amount of Fe that has been deposited per unit time.

6. At the conclusion of the wear test, the piston is dismantled by carefully removing from the cylinder. The motor is dismounted and an autoradiogram will be taken of the cylinder walls. Care must be taken not to contact the paper with an excess of oil on the cylinder walls; therefore, allow the oil to flow down before making the autoradiogram.

EQUIPMENT:

- 1 Activated piston ring
- 1 Single cylinder; 4-cycle engine
- 1 Deep-well scintillometer with scaler
- 20 gamma-counting tubes
- Tools for removing and reassembling piston ring
- Graph paper for plotting curves
- Rubber gloves
- Disposal container for sample oil.

MATERIALS:

Carbon tetrachloride

Ethyl alcohol

10-30 W Automotive oil (for lubrication)

Experiment 14 - DECONTAMINATION OF RADIOACTIVE SPILLAGE

OBJECT:

The purpose of this experiment is to demonstrate the techniques of clean up or decontamination of a radioisotope spillage.

DISCUSSION:

Any person who is associated with reactor work or isotope production or with any type of radioactive material is constantly cognizant of the fact that radioactive spillage may occur. Many times the degree of the spill may be so small that the clean up may be one of simple janitorial service. In such cases it would only be necessary to apply the process of clean up through the use of brooms, paper wipers, or by the application of mops and other such equipment.

In any type of radioactive spill, the use of the radiation safety personnel is needed, and it will be his decision to what extent the spill must be decontaminated. In certain cases the radioactivity involved in the spill may be sufficiently high to warrant the use of equipment which would not normally be employed in the simplified type of spill that has previously been mentioned. In this experiment we will simulate a spill for which the activity is in the range between 250 and 500 roentgen.⁽¹⁾ For this dose rate, special equipment must be called into use.

First, we must consider the number of men that will be used. For the amount of work to be done, a minimum of three men is required to complete the decontamination. Under no circumstance should any one individual be overexposed to the radiation. It is up to the discretion of the radiation safety man to determine the time that any one person is to remain in the contaminated area. Therefore, it is necessary for several other persons to be ready to replace the worker who has been removed from the area.

Where radioactive spillage occurs, we find that the condition of the surface area must be taken into consideration. Assume in this instance that the spill has occurred on a nonporous cement floor. If a porous floor has been involved, other procedures would have to be considered. In our procedure, however, we will assume that the radioactivity had not seeped into the surface of the cement. Once the degree of porosity of the surface has been established, the decontamination will be completed according to the following recommended methods.

Since the activity has been established as being high, the first step must be the removal of the greater portion of the radioactive substance. A worker will don a supplied air suit (see Fig. 33). The suit, either lead

lined or of heavy plastic construction, is designed to cover the entire body. On the head piece is found a connection to which a forced-air hose is attached. The worker will enter the contaminated area with soft paper tissue for wiping. After completing the wiping operation, the tissue will be monitored by the radiation safety man and then deposited in the dry active waste container.

Fig. 33

Supplied Air Suit - Constructed of Heavy-duty Plastic and Designed to Cover Entire Body.

After this, two workers equipped with the Scott air mask (see Fig. 34) will approach the area. These masks will protect the worker from excess inhalation of the vapors that are produced from any liquid that has a high vapor pressure. The mask covers only the face and part of the head. The intake valve for air is located on the under part of the mask. The two workers will complete the wiping job with the use of soft rags, designed not to leave particles of cloth behind. These rags are also checked by the radiation safety man to determine the amount of radioactivity that has been removed from the contaminated area. The area is again monitored for residual activity.

Fig. 34

Scott Air Mask - Designed to Cover Three-quarters of Head. Air Connection is Seen at End of Air Hose. The Air Connection is Attached at the Waist.

After the initial clean up has taken place, the next step is to use mechanical scrubbers (see Fig. 35).

Fig. 35

Electric Scrubber and Stainless Steel Vacuum Tank. Brush on Scrubber is Made of Heavy-duty Wire. Suction Hose for the Vacuum Tank is not Shown in this Figure.

The cleaning agent will be a strong detergent. After the area is thoroughly scrubbed, the excess detergent is removed by a special vacuum system (see Fig. 35). This will suck any liquid and will pass into a stainless steel tank to be processed as waste.

The area is to be monitored for the second time to determine the reduction of radioactivity. If the activity has not been reduced sufficiently, the next step will be the use of either inorganic acids or organic solvents which are known to good solvents for the particular isotope involved. Some of the more common solvents for clean up are nitric acid, acetone, and toluene or xylene.

Two important considerations are the nature of the contaminate and of the surface contaminated. The contaminate must be identified to the type of radioactivity as well as the degree of activity. If the contaminate is an alpha emitter, the major protection must be against inhalation of airborne particulates. If only alpha emitters are involved in a spill, the amount of needed equipment can usually be reduced considerably. Where beta and gamma emitters are found, all of the equipment listed under the materials and apparatus heading should be on hand for use.

The surface that is contaminated must also be considered. In any nuclear facility, the working area should be constructed of material that is nonporous, if at all possible. Any construction material which may be porous should be sealed with proper substances. This will help to reduce the extent of the decontamination procedure because of the prevention of seepage of liquid radioactive material into the porous surface. The contaminated area should be well defined by the radiation safety man. Avoidance of the marked-off area will help prevent the spread of the contamination to a greater area.

Each participant in the decontamination process should always have personnel monitoring devices. Film badges should be worn. Wrist badges, supplied by radiation safety, should be worn by those workers who come in close contact with the radioactivity. Pocket dosimeters should be included as part of the required equipment in this experiment.⁽²¹⁾

By carefully reading the discussion and procedure given in this experiment, the participants who will assist in the decontamination will have sufficient information for one type of decontamination procedures. Any special applications to this type of work will be discussed in lectures which are being given by the Reclamation Division of Argonne.

MATERIALS AND APPARATUS

1. Predetermined type of radioisotope used in the mock contamination
2. Monitoring devices for detection of dose rate
3. Supplied air suits
4. Scott air mask and face masks
5. Wiping tissue
6. Vacuum cleaner for removing liquid detergent
7. Automatic scrubbers
8. Cotton gloves
9. Dry active waste cans with cardboard inner liners
10. Rubber overshoes
11. Scott Air Pak (see Fig. 36) for supplying air to suits and face masks.
12. Special type of wrist dosimeter
13. Lead-lined apron (see Fig. 37).

Fig. 36

Scott Air-tank and Release Valve. The Air Tank is a Special Designed Portable Tank Which is Used to Supply Air to Protective Suits and Special Types of Face Masks.

Fig. 37

Lead-lined Apron - This Specially Constructed Apron is Impregnated with Fine Lead Shot. It is Used as a Shield Against High Gamma Radiation.

PROCEDURE:

Step 1. The radioactive spill is reported to the radiation safety personnel in the particular building. He will monitor the area with his detection equipment. The monitoring device will give an actual reading between 75 and 150 mr at 2 in., although we will assume that the range is approximately 250 to 500 r/hr at 2 in. After the initial monitoring has been completed, the first worker will don the supplied plastic air suit. Two persons will be needed to assist him in getting into the suit. Be sure that rubber overshoes are covering the plastic feet of the suit.

As soon as the air supply is connected and tested, the worker will enter the contaminated area with wiping tissue. By starting at the edge of the marked contaminated area, the worker will wipe the surface with the tissue in two even strokes. The tissue is then placed in the cardboard box which has been supplied by radiation safety. After completely wiping the area, the worker will give the soiled tissue to radiation safety, and it will be monitored for the amount of contamination removed.

Step 2. The next two participants, who should be wearing Scott masks, will approach the area with the scrubbing machine and detergent, as well as with the special vacuum cleaner for removing the detergent and excess liquid after the scrubbing has been completed. When this phase of decontamination has been completed, the area will again be monitored by radiation safety personnel.

Step 3. After the completion of the scrubbing, two more workers will enter the area to complete the clean up with the use of solvents. This phase of the decontamination is dependent upon the decision of the radiation safety man. If he feels that some of the activity has entered into the pores of the surface, he will require the removal of the activity by a suitable solvent. In many instances, it has been found that wiping rags which can be thrown into the active waste can be used. A container of these rags will be given to one of the two workers. The other worker will carry the can of solvent. The solvent is poured over the area lightly. Allow the solvent to dissolve the radioactive substance for one or two minutes. The solvent is then removed in the same manner mentioned in step 1, that is, by the use of paper tissues. The rags are also to be surveyed by radiation safety personnel.

Step 4. The final step of decontamination will be conducted by radiation safety. The complete area which had been previously marked off as contaminated is now monitored. If there is still some residual activity step number 3 will have to be repeated. A complete data sheet will be kept by the radiation safety man from the start of the experiment to the completion of the decontamination.

The data sheet will contain all changes and reduction in activity during clean up. This sheet is prepared so that a permanent record may be made and supplied to anyone who may be interested in the decontamination. Such information is often required by building supervisors.

The actual time to complete clean up of a radioactive spill will depend upon the efficiency of each individual in the group. If an individual is slow in completing his phase of the clean up, the overall work and quantity of equipment may be noticeably increased. It must be emphasized that teamwork is necessary for this type of work. You as an individual may never find the necessity of participating in the clean up, but you must familiarize yourselves with the step-by-step procedure as well as with the equipment required for this type of decontamination.

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