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NUCLEAR SCIENCE SERIES
Radiochemical Techniques

Rapid Radiochemical Separations

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Rapid Radiochemical Separations

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Subcommittee on Radiochemistry
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FOREWORD

The Subcommittee on Radiochemistry is one of a number of subcommittees working under the Committee on Nuclear Science within the National Academy of Sciences - National Research Council. Its members represent government, industrial, and university laboratories in the areas of nuclear chemistry and analytical chemistry.

The Subcommittee has concerned itself with those areas of nuclear science which involve the chemist, such as the collection and distribution of radiochemical procedures, the establishment of specifications for radiochemically pure reagents, availability of cyclotron time for service irradiations, the place of radiochemistry in the undergraduate college program, etc.

This series of monographs has grown out of the need for up-to-date compilations of radiochemical information, procedures, and techniques. The Subcommittee has endeavored to present a series which will be of maximum use to the working scientist and which contains the latest available information. Each monograph collects in one volume the pertinent information required for radiochemical work with an individual element or with a specialized technique.

An expert in the particular radiochemical technique has written the monograph. The Atomic Energy Commission has sponsored the printing of the series.

The Subcommittee is confident these publications will be useful not only to the radiochemist but also to the research worker in other fields such as physics, biochemistry or medicine who wishes to use radiochemical techniques to solve a specific problem.

W. Wayne Meinke, Chairman
Subcommittee on Radiochemistry

INTRODUCTION

This volume which deals with rapid radiochemical separations is the fourth in a series of monographs on radiochemical techniques which will parallel the series on the radiochemistry of the elements. The same general style is used in both series of monographs, including general reviews of the technique, discussions of the principles involved, a detailed survey of applications to different systems, and a collection of selected procedures which use this technique as reported in the literature.

This second series of techniques monographs will cover a number of radiochemical techniques which have not been reviewed elsewhere. Plans include revision of these monographs periodically as new information and procedures warrant. The reader is therefore encouraged to call to the attention of the authors any published or unpublished material on rapid radiochemical separations which might be included in a revised version of the monograph.

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Rapid Radiochemical Separations

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The radiochemical separation process is very important in radiochemical studies because it makes possible the removal of many interfering radiations as well as large amounts of target material.

Some isotopes are produced by a reaction which is attended by a large number of side reactions, one or all of which may cause interference in the measurement of the desired activity. In other cases, the irradiated material is so impure that separation of contaminating activities is required. In addition the techniques of radiochemical separation have for many years been an important tool in nuclear chemistry for establishing genetic relationships in nuclear decay schemes.

In order to determine the half-life, the decay scheme, and other nuclear characteristics of a radioactive nuclide, it is important to use a sample of very high radiochemical purity. In addition in the measurement of nuclear reaction cross sections, fission yields and in activation analysis, the amounts of the radioactive nuclide produced must be determined. Thus it is also necessary to determine the yield of

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the radiochemical separation.

Since individual radionuclides decay exponentially with time, the time interval from the end of irradiation to the beginning of counting is very important, especially for the measurement of short-lived radionuclides. If the time interval is equal to the half-life of the nuclide to be separated (and the nuclide has no parent nuclide), the nuclide decays during that interval to one-half the quantity which existed at the end of irradiation; if the time is two half-lives, it decays to one quarter the quantity, etc. Thus the time of separation must be considered in light of the half-life of the desired radioelement.

If however the short-lived radionuclide has a long-lived parent nuclide from which the former activity is produced genetically, the parent activity can be purified by standard radiochemical techniques. Then, after the establishment of transient radioactive equilibrium, the daughter activity is separated from the parent by rapid radiochemical techniques in which the time to separate the nuclide from the parent must be considered.

For short-lived radionuclides the time required for separation is a very important factor and should be made as short as possible. In this monograph the term "rapid radiochemical separation" is limited to techniques required to separate radionuclides which have half-lives of less than ~ 20 minutes. An effort has been made to collect from the literature as many practical examples of such techniques as possible.

In order to separate and study such short-lived radionuclides, researchers have used many excellent devices to speed up the usual analytical techniques, and have also devised special new techniques of particular interest to radiochemistry. A review of these various techniques should be very instructive and useful in the handling and study of short-lived radionuclides. Furthermore, the extension of new and more rapid radiochemical separation techniques should bring about many discoveries in the nuclear-scientific field and permit the elucidation of many new phenomena in that area.

This monograph is arranged in three main sections. A short section (I) describes general procedures used to prepare samples for rapid radiochemical separation. Then the separations reported in the literature have been categorized in two different ways. In section II the procedures have been arranged by type of process, such as recoil, distillation, solvent extraction, etc. In each subsection there are given typical examples of different kinds of techniques reported -- but the sections are not necessarily exhaustive.

In section III on the other hand the procedures have been arranged by element and in this case an attempt has been made to be as complete as possible. It is realized that pertinent references have been missed and thus the authors would appreciate learning of such omissions for inclusion at a later date in a revised version of this monograph.

I. GENERAL PROCEDURE PRIOR TO RAPID RADIOCHEMICAL SEPARATION

A. Target Material

The chemical and physical form of the target or irradiated sample must be selected so as to facilitate a rapid chemical separation. A material in the salt form, which is easily soluble in an appropriate solvent, is very suitable for the experiment. A rock sample which must be fused for dissolution should be powdered as finely as possible before irradiation. In metallic samples, use of a fine powder or a very thin film is advisable.

In application of the recoil separation during irradiation of solid substances, the target material should be very thin (such as $< 30\ \mu g/cm^2$) to permit collection of the recoiled atoms with a good yield.

In all of the experiments using a fast radiochemical separation technique, the form of the target has great influence on the separation speed as shown in the following examples.

In a study of the long-lived protactinium isotopes produced from thorium bombarded by high energy deuterons or helium ions (175), pieces of thorium metal of 25 mil thickness were used to increase the total yield of the protactinium. On the other hand, when the time for chemical separations had to be shortened in order to study the short-lived protactinium isotopes, thinner pieces of thorium, 5 mils or less in thickness, were used to ensure rapid dissolution. In some cases, thorium nitrate powders wrapped in aluminum were used as the target in order to reduce further the time for dissolution of the target.

In a study of 38-sec iodine-137 as a delayed neutron emitter in fission products, a novel target arrangement was used (235). This consisted of a hydrochloric acid solution (150 ml, containing 1 ml conc. HCl) of uranyl nitrate to which several milligrams of potassium iodide and bromide carriers, 10 ml of 5% sodium nitrite solution, and several ml of carbon tetrachloride had been added before irradiation. The extraction procedure was begun immediately after a two-minute irradiation with a flux of 10^9 n cm^{-2} sec^{-1} thermal neutrons, and the delayed neutrons from the extract in the carbon tetrachloride measured with a neutron counter. By this technique the measurement could be started 28 seconds after the end of the irradiation.

The quantity of the target also influences the separation speed and so, to speed up the separation, the quantity should be as small as possible.

B. Rapid Transference of Sample after Irradiation

In the study of short-lived radioisotopes, the time between the end of irradiation and the beginning of separation must be as short as possible.

For such purposes, facilities such as a "jiffy probe system" in the cyclotron or a "pneumatic tube system" in the atomic reactor have been used.

The former (175) is a long hollow tube with a thin concave aluminum end-window which can be inserted into the tank of the cyclotron. The inside of this tube is kept at atmospheric pressure; hence, the window must be strong enough to withstand the vacuum of the tank. A "rabbit" holding a target was blown by compressed air to a position at the end of the tube as close as possible to the window. At the end of the bombardment, the target is blown out through the end of the probe tube with compressed air.

Provision was made in the rabbit to hold either a metal target on edge or a small cylindrical container of a salt of the element to be bombarded. After bombardments of approximately a minute were made in this jiffy probe, the doors of the cyclotron were opened immediately, the target blown out of the probe into a lead carrier which was manually carried to the laboratory for processing. Later arrangements were made to transport the rabbit to the laboratory with a pneumatic tube.

Pneumatic tube systems are also useful for rapid transference of samples irradiated in a reactor (174). In one such facility the sample to be irradiated is placed in lusteroid or polyethylene capsules and is inserted in a polyethylene rabbit which runs through the pneumatic tube to the side of the reactor. After irradiation, the rabbit is returned to a hood in which chemical operations can be performed, is quickly opened and the sample removed. The pneumatic tube system is operated by vacuum rather than by compressed air to minimize the possibility of contaminating the receiving station with radioactive dust from the tubes.

II. TYPES OF RAPID SEPARATION PROCESSES

Although the detailed methods which have been developed for separating short-lived radioisotopes are extremely varied and depend on the particular problem at hand, all are based on a relatively few general types of separation processes such as: recoil, distillation, solvent extraction, precipitation, ion exchange, etc.

In many cases, these techniques can be used in combination with one another. Most of these techniques are also applicable to fast carrier-free separations of the various radionuclides (93).

A. Hot Atom (Including Recoil)

A nuclide which is produced by a nuclear reaction or as a daughter product after emission of radiation has during the process been given sufficient energy to destroy its original chemical bond or to transform it into a chemical state different from the original one. Sometimes in a multi-phase system such "hot atoms" go into a second phase from the original phase; for instance, from a solid target to the surrounding outer gas phase. Sometimes such "hot atoms" have a valency state or charge different from the original state in the target or mother solution.

Such recoil phenomena occur instantaneously in the process of production of the nuclide. Therefore, the separation step using the recoil phenomena can be considered as a "zero-time" separation.

Under suitable conditions, the recoiled reaction products can be collected on a collector plate during the irradiation. In this case, since other side-reaction products are also recoiled and some aggregate recoils occur, the desired activity may be contaminated. Therefore, we can find many instances where recoil reaction products were collected as the primary step in a fast separation and thereafter some radiochemical separations made.

1. Szilard-Chalmers. In 1934 it was reported by Szilard and Chalmers that radioactive iodine produced by thermal neutron irradiation of ethyl iodide could be extracted into an aqueous phase. Then by being reduced to iodide and precipitated as silver iodide, the concentrated radioactive iodide could be obtained (253).

Such Szilard-Chalmers reactions which depend on the recoil effect of the prompt gamma ray from the (n,γ) reaction have many possibilities for rapid radiochemical separations. A good example is in the classical study for the separation of 25-minute I^{128} in which the isotope is precipitated as silver iodide after neutron bombardment of aqueous iodate solution (3). 2.5-minute P^{30} and 10-minute Cu^{62}, produced by a (γ,n) reaction, can also be separated by the recoil effect (209,246). These isotopes are isolated without any addition of chemical reagent by irradiating fine suspensions of elementary phosphorus and copper with gamma rays from a betatron.

2. Fission gas recoil. Another interesting example of recoil separation in the (n,fission) reaction is the separation technique for krypton fission products from a uranium foil target (202). The target, an enriched uranium film (0.3 mg/cm^2) deposited on a 1.6-cm diameter platinum plate, was mounted as shown in Fig. 1 at one end of a vacuum-tight recoil chamber with a 3.12 mg/cm^2 aluminum foil between the uranium and the recoil chamber. The aluminum foil is used as an absorber for xenon fission fragments. A rubber serum bottle cap was used to cover and seal the exit line at the end of the recoil chamber opposite the target.

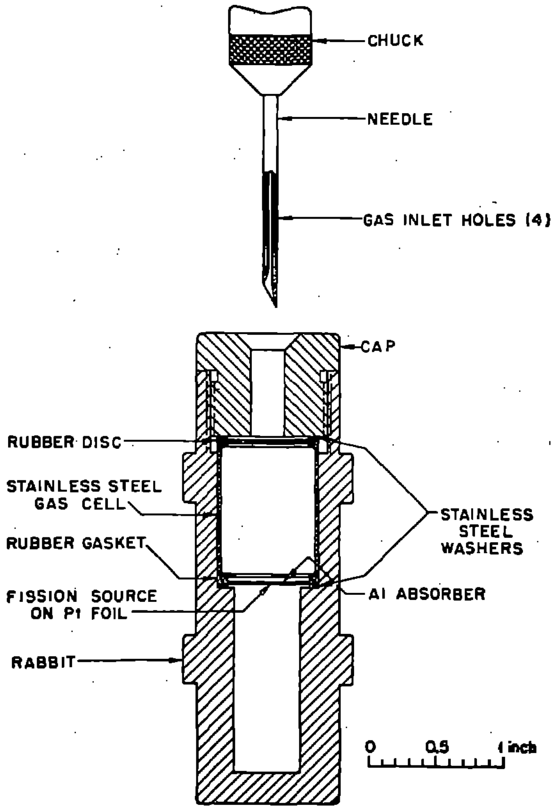

Figure 1. Apparatus for separation of fission fragments during irradiation and rapid removal of krypton gas (202).

This entire assembly was used as a rabbit and irradiated in a reactor for 3 minutes. After the irradiation, the rubber cap sealing the recoil chamber was punctured with a double-walled hypodermic needle, and air containing the recoiled krypton fission products was drawn out through the coaxial outer annulus of the needle. The effluent gases were passed through a trap packed with glass wool, cooled in a dry ice-acetone bath and finally passed into an evacuated cell. 15.4-minute Rb^{89}, the daughter of 3-minute Kr^{89}, was then collected on a negatively charged strip of Al foil for 3 minutes. The foil was removed and the Rb^{89} counted. By this technique, only a few percent of the activity of Cs^{138} (from xenon recoil contamination) was found on the foil.

3. Cyclotron bombardments. A typical example of an apparatus for collecting recoil atoms in cyclotron bombardments is shown in Fig. 2. This assembly was used for the separation of einsteinium (115) and of berkelium and californium (41).

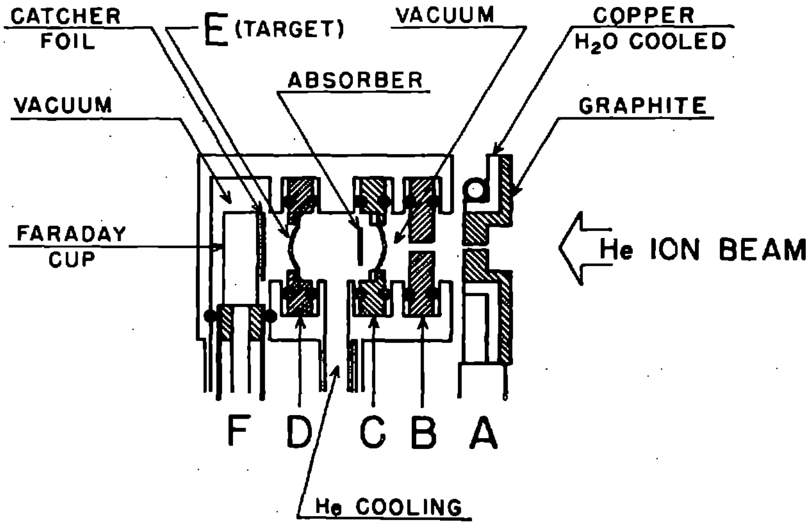

Figure 2. Cyclotron target assembly (schematic) (115).

The helium ion beam was first collimated by means of the water-cooled copper and graphite collimater A. The second collimator B reduced the beam to the required size. The stainless steel holder C supported a 0.04-cm thick duraluminum foil which sealed the rest of the probe assembly from the cyclotron vacuum. Aluminum absorber strips could be mounted behind this foil to decrease the beam energy. Holder D supported the target. Foils C and D were cooled by rapid circulation of helium between them.

The gold "catcher" foil E was mounted on the end of rod F with Scotch tape. Rod F terminated in water-cooled aluminum blocks which served as a Faraday cup for beam measurement. The space between the target and the catcher foil was evacuated.

At the end of bombardment, the Faraday cup rod was removed and the catcher foil torn off. The foil was dissolved in a few drops of 8 M hydrochloric acid containing a little nitric acid. The solution was forced rapidly through a column of Dowex-1 anion exchange resin to remove the gold and then separation of the actinide elements was made by the cation exchange method using Dowex-50 with a solution of ammonium α-hydroxy isobutyrate as eluant.

There are many examples of rapid separation using similar recoil collection methods. 20.4-minute C^{11} produced by the $B^{11}(p,n)$ or $N^{14}(p,\alpha)$ reaction was collected on paper and thereafter separated as carbon dioxide by burning the paper (12).

23-minute Rn^{212}, a spallation product of Th^{232} bombarded by 350 Mev proton, can be separated by recoil (99). The method used to isolate this activity was very simple but permitted separation of the emanation from tremendous activities of other α-emitting elements from bismuth to protactinium. The cyclotron target consisted of thin thorium metal strips sandwiched between thin aluminum foils to act as catchers for the transmuted atoms which were able to recoil out of the surface of the thorium. Rn^{212} was then separated from the aluminum foil by heating at very low temperatures in a vacuum system. A slow stream of argon carried the Rn^{212} through two cold traps at -50° C and into a final trap at -90° C where the rare gases froze out.

In this kind of separation, the target must be very thin to get good yields of the recoiled atoms. Thus the preparation technique for thin targets is very important. For such purposes electrodeposition or vacuum sublimation methods can often be used to advantage.

In other examples of this technique the 35.7-hour Cf^{246} produced by the $Cm^{244}(\alpha,2n)$ reaction was found to be projected forward under proper conditions into a rather narrow cone and it was possible to obtain an ejection yield very close to 100% (115). 25-minute E^{248} has been identified among the recoil products in bombardment of Cf^{249} with 18-24 Mev deuterons (40).

In the primary stage of separations of 30-minute Fm^{250} (4) and $\sim$ 30-minute Mv^{256} (98) a similar technique was applied.

4. Daughters of alpha emitters. The recoil method can also be useful for the separation of daughter products produced by α decay of a parent. This technique has been applied to studies of short-lived daughters in the radioactive decay series of uranium, thorium, and actinium (175).

When an electric field is imposed across a plate containing the sample and another plate, the daughter atoms recoiled out by α disintegration can be collected on the second plate if it is negatively charged. The collector plate, then, is free from the first member of the series present on the original plate, although there may be present on it varying amounts of subsequent members of the series.

Similarly, if a second order recoil transfer is made, in which a further recoil fraction is collected from the first recoil collection plate, neither the first nor the second members of the series is present. This procedure can be extended to third order recoils, etc., if sufficient activity is available initially.

The yield of this type of recoil collection was about 10%; higher yield (i.e. up to nearly 50%) could be obtained in vacuum without the necessity of an electric field. An adaptation of this technique made possible half-life determination on isotopes with values ranging from 30 seconds to about 20 mseconds (175).

Basically, the apparatus consisted of a 13-inch diameter rotating metal disc (phonograph turn table) which was wired electrically to act as a collector for the recoiling atoms, a sample holder which positioned the sample face down over the outer rim of the disc, and an α counting chamber which could also be positioned above the outer rim of the disc. The disc was rotated under the parent samples at a known speed.

Recoiling daughter activities were collected along the rim of the disc and the α-particles from these activities were counted through the slit window of the α particle counting chamber.

By changing the angle (really distance or "travel" time) between the sample and the chamber, counts at different intensities were registered. Plots of activity versus angle gave the usual type of semi-logarithmic curve from which the half-life could be determined (down to about 20 mseconds).

By a similar procedure 4.2-minute Tl^{206} (RaE") can be separated in the α decay of Bi^{210}(RaE) in which the disintegration ratio is only ~ 5 x 10^{-5}% (20). The Tl^{206} was collected on a brass plate (diameter: 2.0 cm) which was held at a distance of 6 mm from a platinum plate carrying ~ 21 millicuries of Bi^{210}. The collecting voltage was 450 volts and the exposure time 12 minutes. In order to reduce aggregate recoil effects to a tolerable proportion, a strong Bi^{210} source of minimum thickness should be used.

1.7-minute $Bi^{<198}$, 7-minute Bi^{198}, and ~ 25-minute Bi^{199}, produced by α disintegration of $At^{<202}$, At^{202}, and At^{203} respectively (which were present on a silver foil), were separated on a collecting plate maintained in a vacuum chamber at a few hundred volts potential negative to the astatine plate (14).

38-second Ra^{222}, the α disintegration product of Th^{226}, was also measured simply by the recoil technique (245). The Th^{226} was isolated with a minimum amount of carrier from a solution containing 100,000 c.p.m. of U^{230} (which had been produced by the irradiation of thorium with 100 Mev α particles in a cyclotron) and mounted on a platinum disc. By inserting this disc in a standard α-counting chamber for a few minutes, it was possible, because of the recoil phenomenon, to introduce several thousand counts per minute of Ra^{222} into the chamber. The Th^{226} disc was then removed and the recoil activity showed the decay of Ra^{222}.

The decay and energy of the 5.5-second Ac^{222} recoil daughter from a thin plate of 1.8-minute Pa^{226}, were measured with an α-particle pulse analyzer equipped with a fast sample-changing mechanism (176). The recoil samples were collected in a vacuum in one section of the sample changer. An interval of about 8 seconds occurred between the end of recoil collection and the beginning of pulse analysis.

5. Daughters of β emitters. Some brief consideration has also been given to application of the recoil phenomena produced by β^- disintegration. The Pb^{212}(ThB) - Bi^{212}(ThC) system was used for the experiments (243). In this case, it was pointed out that the temperature difference between the catcher plate and the emitter plate, the gas pressure, and the atomic number of the emitter plate material have a greater influence on the yield of the recoiled atoms, than the question as to whether the collection is made with or without a potential difference between the plates.

24-second Nb^{90m}, the positron disintegration product of Mo^{90}, has been isolated and measured by the recoil effect (171). An intense carrier-free sample of Mo^{90} was placed on the counting shelf of a scintillation counter for a one-minute period of growth. The aluminum front cover on the sodium iodide crystal was not grounded but carried a potential of 500 volts negative with respect to the sample. The sample was then quickly withdrawn and the recoil daughter atoms of Nb^{90m} remaining on the front cover were counted.

6. Isomeric transitions. Under proper conditions, the recoil effect in an isomeric transition is also applicable to the separation of short-lived daughters. 17.6-minute Br^{80} has been isolated as the bromide ion from tetrabutyl bromide containing Br^{80m} in an aqueous alcohol solution (224). 24.2-second Ag^{110}, produced by the isomeric transition of Ag^{110m}, has been separated on a copper electrode in an ether solution

of silver tetraphenyl porphin by means of a potential field of 600 volts (178).

24.8-minute Te^{131}, the isomeric transition daughter of Te^{131m}, exists as tellurous acid even though the parent Te^{131m} is in the form of telluric acid. Thus the daughter can easily be reduced to elementary tellurium by sulfur dioxide in 3 N hydrochloric acid solution (223), while the reduction of the telluric acid form of the parent is very slow. By a similar technique, 2-minute Te^{133} can be separated from a telluric acid solution of Te^{133m} (206). In this case, the reducing agent was hydrogen sulphide.

18.2-minute Se^{81}, an isomeric transition daughter of Se^{81m}, has also been separated by a similar technique (153).

B. Distillation

In this category are included all separation techniques in which the desired nuclides are separated as gaseous substances. If the nuclide to be separated is a gaseous element to begin with or can be transformed easily at room temperature or at a somewhat elevated temperature into a readily-volatile compound, it can be separated very easily and rapidly from the sample and then can be collected either by cooling or by absorption in a suitable solution. The ease or speed of such a separation method seems to be dependent upon the boiling point of the substance to be separated, as well as on characteristics of the matrix material.

1. Separation during production. In some cases, the gaseous radionuclide product can be isolated during its production, i.e., during the bombardment of the target, and thus a "zero time" separation can be realized. The study of 20-second C^{10} produced by the $B^{10}(p,n)$ reaction (228,229) can be cited as a typical example of such a separation technique. When elementary boron and boron compounds such as boric acid,

borax and calcium borofluoride are bombarded in a thin copper cell in a cyclotron with 16-18 Mev protons, the resulting C^{10} and C^{11} activities can be swept out as carbon dioxide by blowing a gas through the cell. Strong activities could be blown 40 feet in a fraction of a second into a thin-walled container placed adjacent to a recording Geiger counter.

By this technique, in addition to the 20.4-minute C^{11}, a new 20-second positron activity due to C^{10} was found. The cyclotron probe and the gas handling system used are shown in Fig. 3 and Fig. 4 respectively. For a target, a small amount of finely powdered material was held in place at the tip of the cell by a mat of glass wool. After or during irradiation, the radioactive gases formed and released by the bombardment were swept to an inspection chamber by a flushing gas through the concentric tubes in the probe as shown in Fig. 3.

Tubes of absorbing materials or traps of various kinds were inserted in the flow line to purify the gas. For the measurement of radioactive carbon (as carbon dioxide), a layer of Ascarite (sodium hydroxide absorbed in asbestos) was held across the center of the measurement chamber by wire screens. The gas inlet and outlet were so arranged that the gas could leave only by passing through the absorbing material. By this technique, nitrogen and neon activities were easily separated from the carbon activities by pumping off. Measurements for the carbon activities could be started within a fraction of a second after the end of bombardment.

This assembly was also used to study 72-second O^{14} produced by a $N^{14}(p,n)$ reaction. In this case potassium thiocyanate was used as the target material. The oxygen activities of the reaction product appeared in the form of water. The water was absorbed and collected on a layer of calcium chloride, while carbon, nitrogen, and other activities were pumped off.

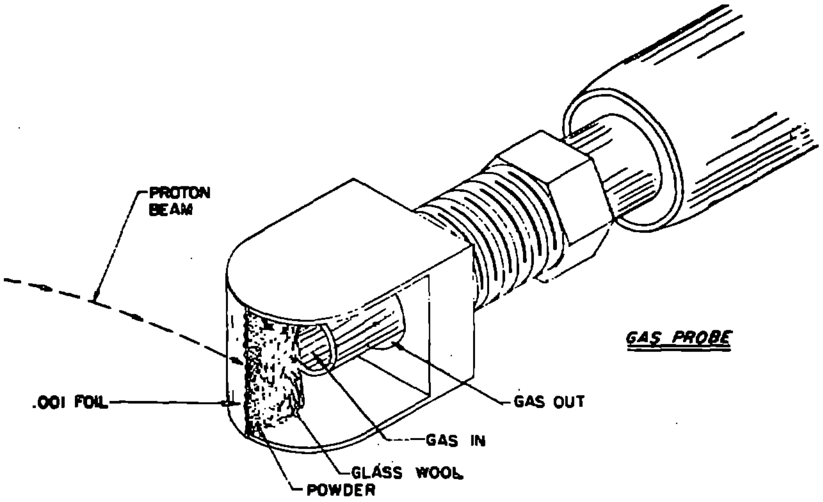

Figure 3. Cyclotron probe for collection of radioactive gases evolved in the bombardment of powdered targets. These gases are swept to an inspection cell by a flushing gas (229).

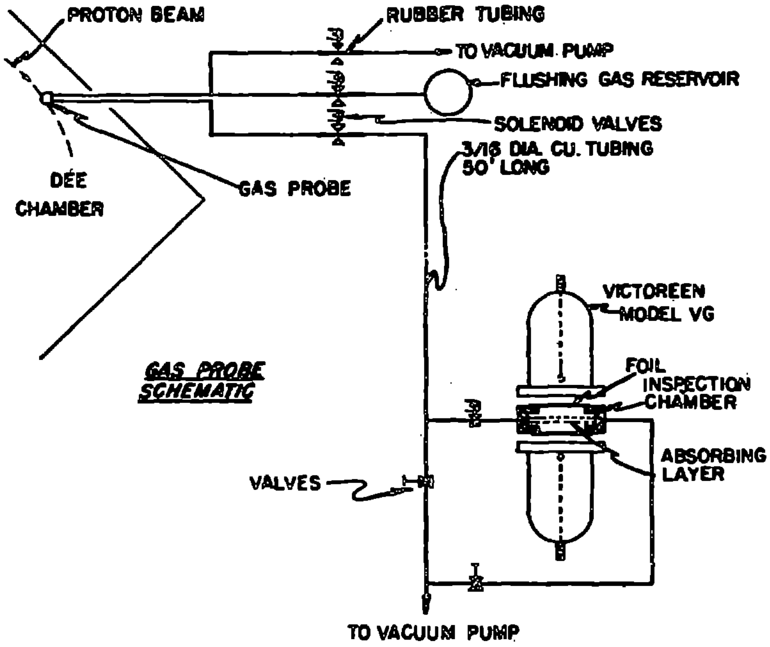

Figure 4. Schematic drawing of the gas probe system, showing the probe, control valves, and counting arrangement (229).

While such separation methods for radioactive gaseous products are very inefficient in terms of the total radioactivity available, sufficient activity for the measurement could be obtained. Furthermore the separation yield could be increased where necessary by using more finely divided target material.

2. Rare gases and daughters. Another interesting separation and detection method can be found in the study of very short-lived Na^{24m} (67). This nuclide is formed as a β^- disintegration product of Ne^{24} which is produced by a Ne^{22}(t,p) reaction). The 3.38-minute Ne^{24} activity, which was removed by a gas flow system immediately after the end of irradiation, was purified from the other radioactivities such as sodium and fluorine by passing the gaseous radioactive products through activated charcoal traps cooled with dry ice. The Ne^{24} was then collected in a chamber which had been previously evacuated. A schematic diagram of the assembly is shown in Fig. 5a.

The Na^{24m} daughter activities were then collected on a negative electrode and measured after removal of the neon gas

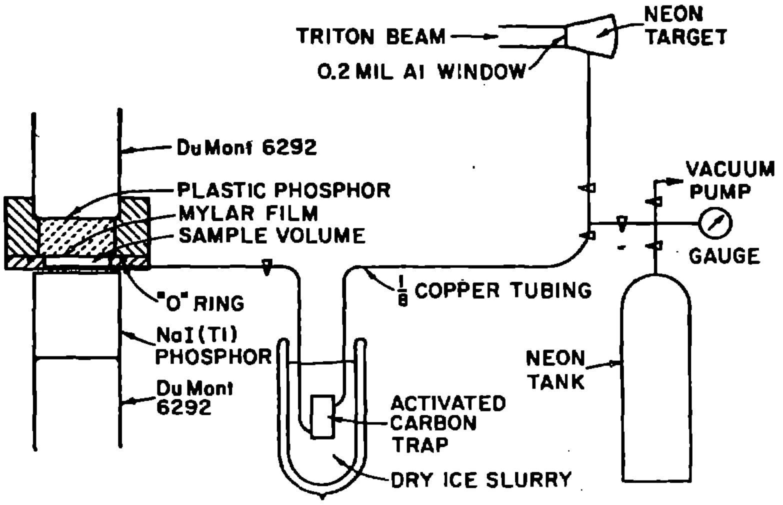

Figure 5a. Apparatus for the production and detection of Ne^{24}. The detector shown at the left was used for beta energy and beta-gamma coincidence measurements (67).

from the counting cell. The collecting cell is shown in Fig. 5b. After the counting cell was filled with active neon to a pressure of 5 cm Hg, a collection voltage of 22.5 volts was applied between the electrode and the case. The observed increase in counting rate of the γ detector indicated that Na^{24m} was being collected. After sufficient counts had been recorded to permit an accurate evaluation of the average counting ratios, a valve was opened rapidly and the gas allowed to expand into an evacuated 5-gallon bottle. By such a technique the investigators could estimate that the half-life of Na^{24m} falls in the range of 5 to 50 mseconds with a most probable value of $\sim$ 20 mseconds.

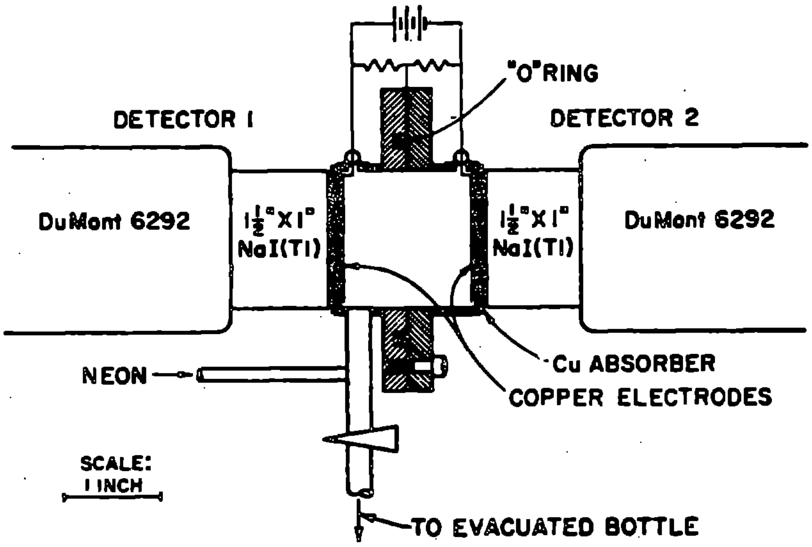

Figure 5b. Counting cell and scintillators which were used for the electrostatic collection and identification of the Na^{24} isomer (67).

3. Nitrogen chemistry. A very interesting example of rapid gas separation technique in radiochemical work can be found in the study of the chemical identification of 7.3-second N^{16}, produced by the $O^{16}(n,p)$ reaction (200). The target in this study was powdered potassium nitrite pressed

into pellets weighing about 300 mg which were sufficient to produce about 75 cc of nitrogen gas. These pellets were blown into and out of a reactor in a pneumatic tube device and were automatically delivered to the separation apparatus by means of a metal rod in the conveyor. The pellet was pushed through a thin paper seal and out through a trap door at the outside end of the pneumatic tube when the conveyor hit the trap. The apparatus is sketched in Fig. 6.

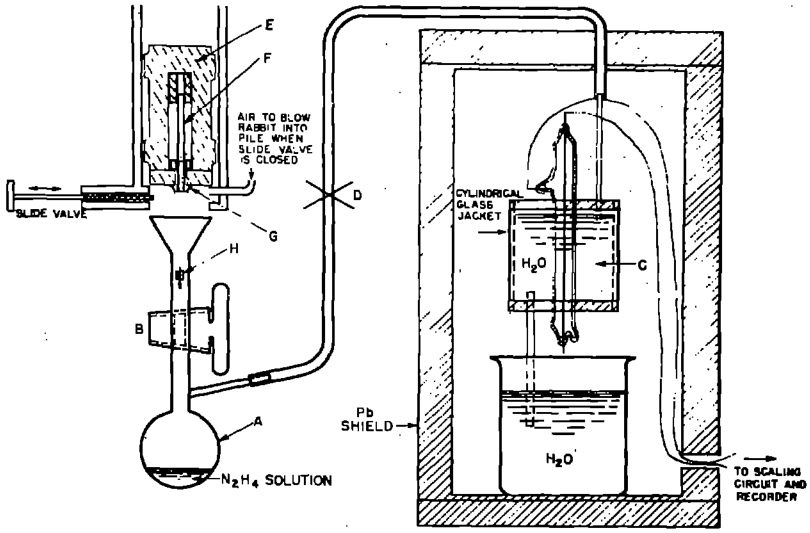

Figure 6. Assembly of apparatus for detection of N^{16} activity (200).

(A)	Reaction flask
(B)	Stopcock
(C)	Counting chamber
(D)	Pinch clamp to end flow of gas
(E)	Rabbit of resin-impregnated paper
(F)	Pill-ejector rod
(G)	Paper seal for pill
(H)	Nitrite pill

A typical run procedes as follows: The nitrite pill, H, is placed in the rabbit, E, and is sealed in with a thin piece of paper, G, held on by Scotch tape. The rabbit is placed in the pneumatic tube with the pill ejector, F, end down. Water is sucked into the counting chamber, C, and acid hydrazine sulfate solution is heated in the reaction flask, A. The system is closed at every point except at the stopcock, B.

Compressed carbon dioxide gas pushes the rabbit into the pile; timing is begun at the moment of impact of the rabbit against the stop at the center of the reactor.

A 20-second irradiation is made. The trap door at the end of the tube is then opened and the rabbit is blown out. The pill falls down into the funnel, through the stopcock, B, and into the solution. The nitrogen gas is generated very rapidly and displaces the water in the counting chamber, C. After 7 seconds the reaction is nearly complete, and the counting chamber is cut off by means of the pinch clamp, D, to end the flow of active gas to the chamber.

4. Selective gas adsorption. The gas chromatographic technique can be cited as another example of rapid radioactive gas separation. One procedure for the separation and detection of argon, krypton and xenon activities in the coolant or exhaust gases from nuclear facilities utilizes such a method (150).

In order to study some short-lived fission product gases, an interesting simple separation technique has been developed (264). The schematic diagrams of the sample preparation are shown in Fig. 7. For the rapid separation of krypton activities from fission products, a sample containing uranyl nitrate and Norite (activated charcoal) was prepared in a piece of 1/4" (i.d.) polyethylene tubing sealed on both ends by warming and crimping shut. Tissue paper pads were used as spacers to contain the source material and the charcoal filter. The Norite adsorbs xenon, but allows krypton to diffuse through into the gas chamber.

The sample was irradiated for 5-20 seconds, removed from the rabbit and a gas sample of about 2 cc taken from the gas chamber with a syringe and hypodermic needle. The gas sample was transferred to a second piece of tubing sealed under partial vacuum. This tubing was then placed in a polyethylene

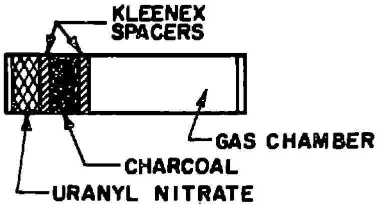

Figure 7a. Sample preparation for determination of krypton (264).

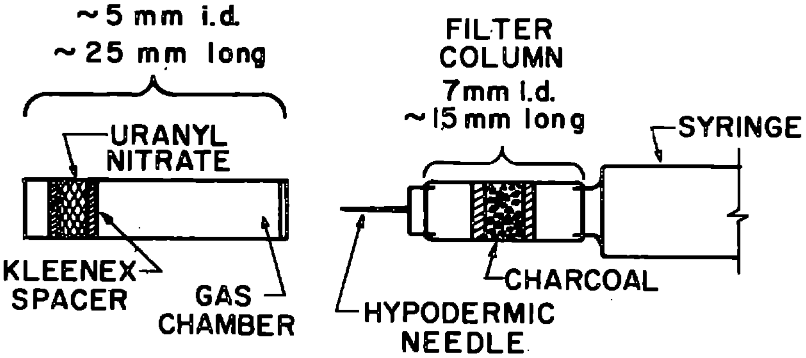

Figure 7b. Sample preparation for determination of xenon (264).

"bunny" which was sealed with a tight fitting polyethylene plug and sent to the detector in a second pneumatic system.

For xenon samples the Norite was placed in a piece of tubing connecting the needle to the syringe as shown in Fig. 7b. After irradiation of the sample and drawing off the gas sample, the tubing (containing the xenon and Norite) was placed in the bunny and sent to the counter. In this case the krypton passed through the filter into the syringe and was discarded.

For some samples, the gas was allowed to decay for two half-lives, the residual gas removed by purging with a hot air stream, and the remaining short-lived daughter activities observed. The time required for handling, separation and transport to the counter for regular gas samples was 20-30 seconds.

5. Emanation methods. As shown in the above example radioactivities of the rare gas elements are very easily

volatilized and separated from various kinds of material. A typical example of this kind of separation is the classical emanation technique originated by Hahn (263). The same kind of technique has many applications in the study of short-lived rare gas activities in fission products (19,48,64).

6. Glow discharge for separation and mounting. Hyde *et al*. have described the glow discharge method for the rapid collection and mounting of emanation activities produced by 340 Mev proton bombardment of thorium and C^{+6} bombardment of lead and bismuth (170,179). The method consists of transferring the emanation radioactivity together with 100 to 1000 microns pressure of some carrier gas such as nitrogen or ordinary air into a glass tube equipped with two electrodes. A d.c. potential of a few hundred volts is placed across the electrodes to cause formation of a glow-discharge. In this discharge the rare gas atoms are ionized and the resulting ions are collected on the negative electrode and affixed to the plate after being deposited. This method is very suitable for preparation of very thin plates of the emanation activities for α ray measurement.

This technique was also applied to collecting xenon activity on thin aluminum foil and should have considerable usefulness in the preparation of samples of other gaseous radioactive elements.

7. Volatilization and distillation. Many other kinds of short-lived radionuclides which can be transformed into volatile compounds can be separated by volatilization, distillation, or gas sweeping methods.

0.53-second Ge^{73m} can be distilled as the tetrachloride from a hydrochloric acid solution of As^{73} (33). 9-minute As^{79} has been separated as arsine from a fission product mixture and collected in an aqueous solution of silver nitrate (54).

Short-lived bromine isotopes in fission products, 15.5-

second Br^{88} and 1.6-second Br^{90} can be separated as bromine gas, absorbed in carbon tetrachloride and counted 2 seconds after the end of irradiation (212,213). Fig. 8 schematically represents the chief elements of the gas-flow system used. To a stream of air flowing at a rate of about 1 liter/min, there was added a small flow (about 2 ml/min) of saturated bromine vapor and the mixture bubbled through a solution containing 2 g of U^{235} in 1 $\underline{M}$ HNO_3 and 0.5 $\underline{M}$ $KBrO_3$. The total volume of 15 ml of solution was contained in a quartz cell and was irradiated with thermal neutrons.

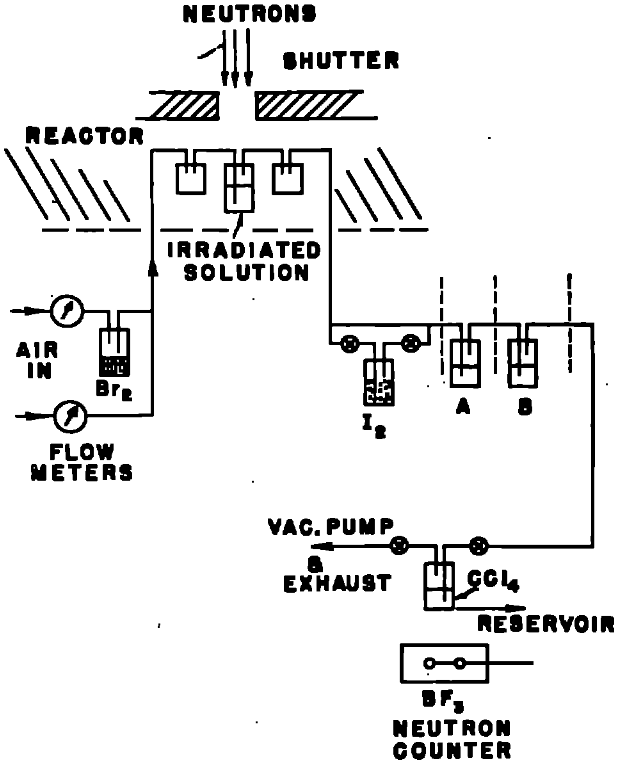

Figure 8. Gas-flow system for rapid chemical separation of bromine to permit study of short-lived activities (212).

Two small vessels flanked the irradiation cell to catch any active solution which might be carried out of it by accident. The BrO_3^- ion served a dual purpose. By virtue of its reaction with Br^- to form Br_2, bromides could not accumulate in the cell. The second purpose of the BrO_3^- was to oxidize iodine formed in its lower valence states to the non-volatile IO_3^- form, and thereby to prevent its escape into the air stream.

Iodine vapor was added as a hold-back carrier to the air stream after it left the reactor by passing about 10% of the gas over iodine crystals. This side stream then rejoined the main stream which was subsequently scrubbed of its iodine by an acid solution of $KBrO_3$ in trap A. Trap B was used only for auxiliary tests. In some tests trap A was removed also.

After the air stream left these traps, it passed through about 10 ml of CCl_4 where the bromine remained, and from there through some traps to a vacuum pump, and then to an exhaust vent of the room. The CCl_4 could be maintained at a nearly constant level by connection to a large reservoir.

Short-lived ruthenium isotopes, such as 52-second Ru^{93} produced by α particle bombardment of molybdenum (10) and the 4-minute Ru^{108} fission product (103), can be distilled as the tetroxide and measured.

Short-lived antimony isotopes, such as 19-minute Sb^{126}, 10.3-minute Sb^{128}, and 1.9-minute Sb^{132} produced in $Te(d,\alpha)$ and $Te(n,p)$ reactions or in the fission process, can be separated rapidly as stibine (1,87,88). More recently, 10-second separations of the antimony activities in fission products have been made (164). For this method Sb^{+3} carrier with the fission products and 30% sulfuric acid was dropped onto metallic zinc held in a distillation flask at 100° C. The stibine produced passes through a calcium chloride drying tube and then through a small diameter heated quartz tube where the stibine is converted into antimony metal.

The 10-minute Os^{190m} can be isolated as volatile osmium tetroxide from the Ir^{190} parent (46).

To measure the short-lived α emitters of astatine, such as 43-second $At^{<202}$, 1.7-minute $At^{<203}$, 7-minute At^{203}, ~ 25-minute At^{204}, and 25-minute At^{205} produced by α particle bombardment of bismuth in a cyclotron, a rapid distillation method was used (14) with a resultant separation time of less than 90 seconds. The 0.9-minute At^{219}, daughter of Fr^{223}, has been separated by a similar method (126).

8. Vacuum distillation. This method is applicable to the rapid separation of some relatively volatile metallic elements. By operating in a vacuum it is possible to reduce oxide formation while allowing good control of the vapor stream. Furthermore, since some carrier-free separations are also possible, the method can provide a rapid method for preparing very thin counting samples with high specific activity.

A general and extensive discussion on the application of this method to rapid radiochemical works has been reported (62). In this report, the radiochemical separation of cadmium and mercury, and the carrier-free separation of In^{113m} (the daughter of Sn^{113} produced by neutron irradiation of tin) were investigated experimentally. The aqueous radioactive solution containing various contaminants was used as the starting substance for the separation.

In a preliminary step of the separation, the cadmium radioactivity (with 1 mg Cd carrier) was reduced onto a copper foil or into a mercury drop by electrolysis, and the mercury radioactivity (with 16 μg Hg carrier) was reduced onto a copper foil by auto-reduction. In the second step, these radioactivities were separated by vacuum distillation. In the separation of the In^{113m}, granular tin metal was irradiated in a nuclear reactor and then vacuum distillation immediately applied.

The yields of these nuclides in the separation were about 70 to 90% and good decontamination factors (10^3 to 10^4) were obtained for a number of contaminants. These distillation techniques required about 10 to 15 minutes for the distillation separation step and, on the other hand, required about 40-60 minutes for the preliminary separation step by electrolysis. To speed up this latter step, higher current densities in the electrolysis step or other selective chemical reduction methods should be applicable.

The same kind of technique has been applied to the separation of 23-minute Hg^{189} from tantalum foil bombarded with a carbon or nitrogen ion beam in the cyclotron (38).

C. Solvent Extraction

The selective extraction of a radioactive nuclide as a nonpolar compound or complex from an aqueous solution by an immiscible organic solvent is frequently the most satisfactory method for making a fast radiochemical separation in either macro or trace concentrations. Separations by solvent extraction are simple, convenient, clean, and rapid. They usually require apparatus as uncomplicated as a separatory funnel and may readily be adapted to remote handling procedures.

A variety of extraction separation techniques has been applied to problems of isolation and radiochemical purification of nuclides. Most of the extractions employed in radiochemistry are based on previously developed conventional analytical extractions, and general information of the technique can be found in the literature (89,185).

With this technique it is easy to repeat the operations of extraction, back-extraction and washing with proper solvents to improve the separations without great expenditure of time. The back extraction technique is also applicable as a rapid "milking" method in which a daughter product is separated from the mother activity in the organic solvent layer by shaking with an appropriate aqueous washing solution.

There are in the literature many practical examples of the application of solvent extraction to the separation of short-lived radionuclides. A number of these are given below.

Extraction of the elemental form of bromine is used to separate short-lived bromine activities from fission products. Potassium chlorate in 8 N nitric acid solution (235), potassium permanganate in nitric acid solution (106,248), and a bromide-bromate-hypochlorite mixture in acidic solution (247) have been used as the oxidizing agent. The bromine is extracted into carbon tetrachloride and can be back-extracted into the aqueous solution by reducing agents, such as potassium nitrate (235), hydrazine hydrochloride (106,248), or sodium hydrosulphite (106).

Short-lived iodine fission products have also been separated by a similar method. In this case, an oxidant such as nitrite ion in acidic aqueous solution has been used (30,235), the iodine extracted into carbon tetrachloride, and then back-extracted into an aqueous solution containing sodium hydrosulphite (30).

Short-lived astatine isotopes produced in bismuth bombardment by α particles were extracted into diisopropyl ether after reduction to the elemental form by ferrous sulfate in hydrochloric acid solution (14).

Short-lived iron isotopes, such as 8.9-minute Fe^{53} produced by the $Cr^{50}(\alpha,n)$ reaction (160), and the iron reaction products in arsenic bombarded by 190 Mev deuterons (120), can be extracted into ether as the chloride from 6 N hydrochloric acid solution. The 2.6-minute Ga^{64} produced by the $Zn^{64}(p,n)$ reaction is also separated as the chloride in ether from 6 N hydrochloric acid solution (50).

Short-lived antimony fission products have been extracted as the antimonic chloride into isopropyl ether (205). The antimony activities produced in tellurium bombarded in a cyclotron have also been extracted into a mixed solvent of isopropyl ether and benzene (1:10) from 9 N hydrochloric acid solution after oxidation by bromine (146).

Arsenic activities in fission products can be rapidly extracted into benzene as the iodide from hydroiodic acid solution and then easily back extracted into water (145). This method is a good, rapid separation from the germanium precursor and makes possible an exact determination of the fission yield of As^{78}.

The 10-minute Co^{62} produced by the Ni^{62}(n,p) reaction has been extracted as the thiocyanate into an organic layer of amyl alcohol-ethyl ether (1:1) from a weak hydrochloric acid solution saturated with ammonium thiocyanate and then back-extracted with 6 N ammonia solution (156,208). The 5.2-minute W^{179} produced by a Ta^{181}(p,3n) reaction has also been extracted as the thiocyanate of W(IV) into ethyl acetate (268).

Short-lived uranium isotopes, such as 1.3-minute U^{227} and 9.3-minute U^{228}, the reaction products of α particle bombardment of thorium (173,175,176), and 26.5-minute U^{235m}, the daughter of Pu^{239} (83), have been separated as the nitrate in ether. Considerable salting was required in these extractions.

Tributylphosphate (TBP) extraction methods using dibutyl ether solution have been applied to the separation of 8-minute Po^{200} produced by the Bi(p,10n) reaction (136), and to the separation of 0.9-minute At^{219}, the daughter of Fr^{223} (126).

The 3.5-minute Cr^{55}, from the Mn^{55}(n,p) reaction, was extracted into ether in the peracid form after oxidation by hydrogen peroxide at a pH of 1.7 (83).

Short-lived technetium isotopes, produced as a daughter product of molybdenum radioisotopes or as fission products of U^{235} were extracted as the tetraphenylarsonium salt in chloroform together with perrhenate carrier (91,262).

Chelate extraction systems have also had very extensive application in rapid radiochemical separations.

Cupferron extraction has been applied to the separation of 5.8-minute Ti^{51} in neutron activation analysis (140), of 3.8-minute V^{52} in neutron activation analysis (25,90,132), and of 5.2-minute Cu^{66} in neutron activation analysis (132).

Examples of thenoyltrifluoroacetone (TTA) (in benzene) extraction can be found in the separation of 3.8-minute V^{52} in neutron activation analysis (90), of zirconium activities in niobium bombarded by protons (128), and of 1.8-minute Pa^{226} from the reaction products of thorium bombarded by a cyclotron (172,173,175). 16-second Y^{89m} (109) and 25-minute Am^{246} (74), the daughter products of Zr^{89} and Pu^{246} respectively, were separated by back extraction into acidic aqueous solution from the parent in TTA-benzene solution.

The 24-minute Pa^{235} was extracted by diisopropylketone from reaction products of uranium bombarded by protons (177). Similarly the 3-minute Ce^{145} in a fission product mixture was separated into methylisobutylketone from 10 M nitric acid solution (167).

Diphenyl thiocarbazone (dithizone) extraction methods have been applied to the separation of short-lived tin fission products (207).

Di-β-napthylthiocarbazone extraction has been used in the separation of short-lived zinc activities produced in α bombardment of nickel (159).

Extraction methods using 8-hydroxyquinoline (oxine) have been applied to the separation of 10.5-minute Co^{60m} in the neutron activation analysis of cobalt in biological tissue samples (133), and for the separation of 24-minute U^{239} in neutron activation analysis (56).

As one can see most of the extraction methods mentioned above are based on previously developed conventional analytical extractions. The last 10 years have seen a great increase in interest in this type of separation with efforts made to catalog extractions by system and by elements (89,185) as well as the development and extensive study of new types of systems such as the alkyl amines (182) and the phosphine oxides (267). Many other extraction methods used in chemical analysis could undoubtedly be readily applied to rapid radiochemical separations.

D. Precipitation

Precipitation techniques used in chemical analyses also have many applications in rapid radiochemical separations. Although this method tends to have some disadvantages, such as time consumed in filtration or washing and the coprecipitation of undesired impurities, it can still be very valuable, expecially when other rapid separation methods are not applicable.

The precipitation technique is often applied in the primary step and/or the final step of a procedure in which another technique of rapid separation, such as recoil, solvent extraction, or distillation, is used.

In general, the use of a specific or selective reagent as precipitant for the element is advisable as is the use of a masking reagent for interfering elements. In this way a single or double precipitation procedure often becomes sufficient.

The coprecipitation technique is also very applicable to this type of separation. Here centrifugation or suction systems are often used to speed up the filtration and washing time. The correct choice of filter is important to minimize filtration time.

An interesting example of a fast radiochemical separation technique using the coprecipitation method is the separation and measurement of 5-second Tc^{102}, the daughter of the Mo^{102} fission product (85). In this study, the Mo^{102} parent activity was purified by lead molybdate precipitation with subsequent dissolution in a mixture of hydrochloric acid and tartaric acid in the presence of perrhenate carrier.

The solution was then transferred to a Büchner funnel which had been fitted with a membrane filter and mounted on a filter flask. The entire assembly was placed directly beneath a GM counter as shown in Fig. 9. A solution of tetraphenylarsonium chloride solution was then added and suction applied. When filtration was complete, the Tc^{102} which had coprecipitated on the tetraphenylarsonium perrhenate could be counted. The time from the precipitation to the counting was only six seconds.

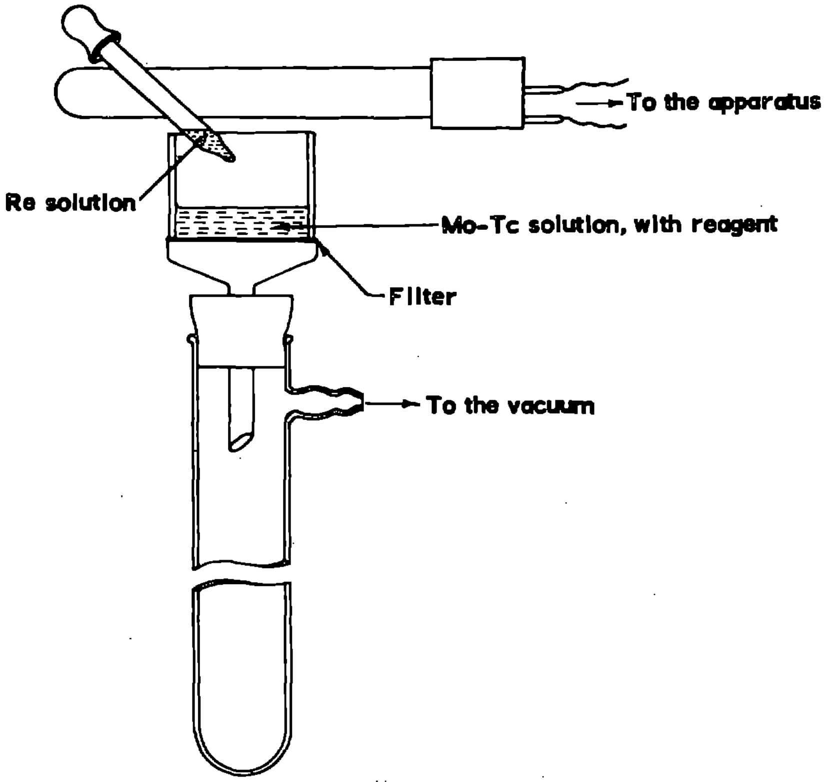

Figure 9. Separation and detection assembly for Tc^{102} (85).

The different types of precipitants used for various elements can be found listed by element in a later section.

E. Ion Exchange

Although this method can give very good decontamination factors and also a high yield, the speed of the separation as it is ordinarily used is rather slow. This technique, however, occupies a prominent position in the radiochemical separation of the rare earth elements (238) and the actinide elements (119).

A selective elution technique from the ion-exchange column is often successful for a very rapid isolation of a short-lived daughter nuclide from relatively long-lived parent activities which are strongly held to the ion-exchange column.

Examples of such a rapid "milking" technique can be found in the separation of 24-second Nb^{90m} from Mo^{90} using Dowex-1 anion exchange resin and 6 N hydrochloric acid as eluant (171), in the separation of 39-sec Ag^{109m} from Cd^{109} using anion exchange resin and 10 M hydrochloric acid as eluant (219), and in the separation of 4.9-second Ir^{191m} from Os^{191}, using anion exchange resin and 6 M hydrochloric acid as eluant (32). In the last two cases, the eluant was forced by compressed air or by the action of a hypodermic syringe through the column to enhance the elution speed.

A similar technique was used in the separation and measurement of 0.8-second Pb^{207m}, the daughter of 28-year Bi^{207} (31,32). For such a rapid separation the volume and the particle size of the resin must be as small as possible. In this study, the Bi^{207} activity was fixed on about 1 cm^3 of Dowex-1 anion exchange resin in 0.5 N hydrochloric acid medium. The Pb^{207m} daughter diffuses out of the small resin spheres into the interstitial solution and a few drops of this solution are transferred rapidly to a counter (31). The use of a hypodermic syringe enhanced the elution speed in this case.

In a later paper (32) these same investigators describe an interesting apparatus for the measurement of the half-life of Pb^{207m}. A schematic diagram of their apparatus for the

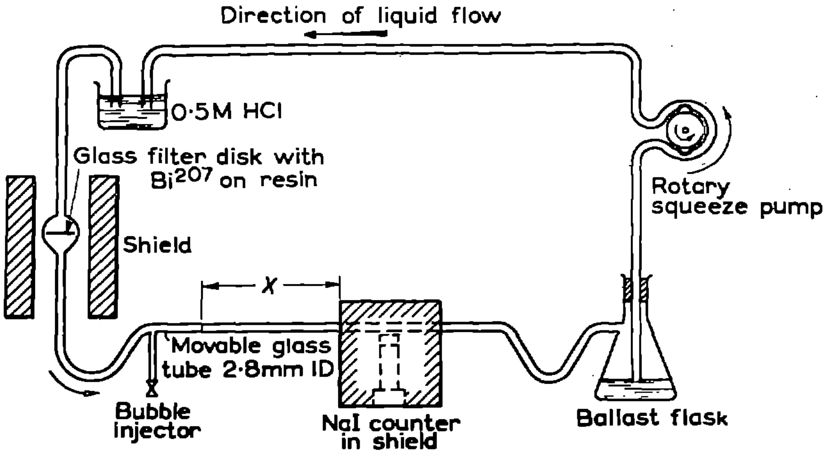

Figure 10. Schematic diagram of an apparatus for measurement of Pb^{207m} half-life (32).

continuous recirculation of effluent is shown in Fig. 10. The half-life of Pb^{207m} was obtained by measuring the effluent counting rate as a function of distance down stream from the resin for a constant flow of known velocity. As the position x of the detector below the uniform-bore tube is varied, the counting rate is expected to decrease according to $e^{-(\lambda x/Vo)}$, where Vo is the flow velocity and λ is the decay constant.

The horizontal tube can be moved through the counter shield. To avoid complications associated with non-uniform flow and also to provide a direct means of measuring the flow velocity, air is allowed to leak into the system to form uniform bubbles spaced at regular intervals. The time taken for the bubbles to travel the distance between two markers 100 cm apart on the tube is measured with a stop-watch during each of the counting intervals. The experimental results are shown in Fig. 11, in which all of the data have been corrected for background. A half-life of 0.84 seconds for Pb^{207m}, with a probable error of 2%, was obtained from these results.

The combination of a short column with a fine powder of ion exchange resin and a high flow rate device has also been

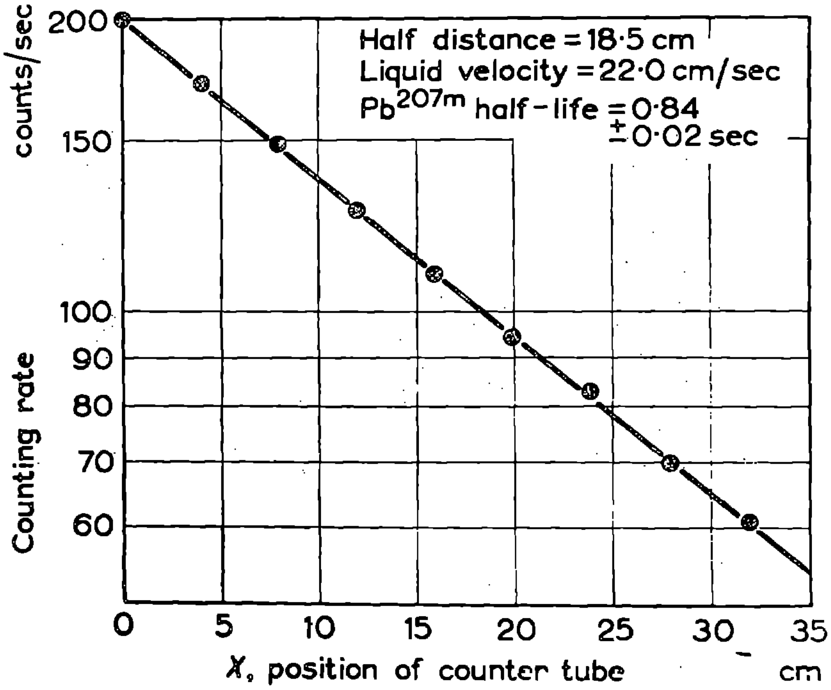

Figure 11. Attenuation of Pb^{207m} activity downstream from source in continuous flow experiment (32).

discussed in connection with the rapid separation of members of the heavy rare earth series (239). By using very fine particles of Dowex-50 cation exchange resin in a column 3-10 mm high and 2 mm i.d., and 0.25 M glycolate at the proper pH as the eluant, the separation of thulium and terbium, of lutetium thulium and erbium, and yttrium and thulium was possible within 20 minutes (not including dissolution and column loading time).

The anion exchange column separation of carrier-free Pb^{210}(RaD), Bi^{210}(RaE), and Po^{210}(RaF) (215) can be used as a good example of the rapid ion exchange separation method. Although these nuclides are not short-lived, separation by the ion exchange method can be made in a relatively short time. A column (7 mm high and 2 mm i.d.) of 50-100 mesh Dowex-A1 anion exchange resin was used. The resin was converted to the chloride form with concentrated hydrochloric acid and was washed with 1-2 M hydrochloric acid.

Pb-Bi-Po tracers in 2 ml of 1 M hydrochloric acid were added to the column. The lead activity was eluted by passing through 3 ml of 1 M hydrochloric acid in 1 ml portions. Then by eluting with 4 ml of concentrated hydrochloric acid in 2 ml portions, the bismuth activity was recovered. Finally, the polonium was eluted with concentrated nitric acid. The entire separation can be effected in 10-15 minutes and the products are obtained in carrier-free form.

An interesting variation of the ion exchange method for fast radiochemical separations is the absorption of trace amounts of cations on anion exchange resins by the formation of insoluble salts. It had been shown that cations can be precipitated around the beads of an anion exchange resin in a column when passed in aqueous solution through the resin which is in a form providing a precipitating anion (107). By employing a column in the hydroxide form, all the radioactivity in an aged fission product mixture with the exception of that due to radio strontium and cesium (assuming barium absent) can be absorbed (55,131).

Aliquots of fission product solution with added known amounts of cesium and strontium were passed through a column of Amberlite IRA-400 (OH) and eluted with several column volumes of water. The strontium and cesium in the eluate were separated from each other by the oxalate and perchlorate precipitation method. By this means, the determinations of these two elements were accomplished rapidly and with good precision. By using an anion exchange column in the carbonate form, only cesium in fission products can be eluted (186,271). This type of separation should have application to rapid procedures.

The use of a zirconium-tungstate ion-exchange column for fast separation of cesium and rubidium activities in fission products is also very interesting (52). In this study, the uranium metal irradiated by neutrons is dissolved in a solution of 6 N nitric acid and 6 N hydrochloric acid containing carriers of iron, yttrium, barium, strontium, cesium, and rubidium. After scavenging by precipitation with silver chloride, ammonia, and ammonium carbonate and centrifuging, the remaining solution is evaporated to dryness. This is then dissolved in 2 ml of 0.01 M ammonium chloride solution, pipetted onto a zirconium tungstate column (3 cm long, 8 mm i.d.), and pressure applied (5 lbs/sq. inch) to force the active solution through the column. The activity is eluted with 1 M NH_4Cl at a rate not greater than 0.2 ml/minute.

The experimental results are shown in Fig. 12. By this means the measurement of 32-minute Cs^{138} and 18-minute Rb^{88} in fission products can be made.

F. Isotopic Exchange

The exchange of silver ion between a precipitate of silver chloride and a solution of silver nitrate reaches isotopic equilibrium very rapidly (152). Because of the low solubility of silver chloride, a favorable ratio exists at equilibrium between the silver atoms in the precipitate and in the solution. For this reason, if a silver chloride precipitate is added to a solution containing only trace amounts of radioactive silver, a high percentage of this radioactive silver will have exchanged with the silver in the precipitate by the time equilibrium is attained.

The use of such techniques for radiochemical work has been discussed in detail (203,250,251). In these studies a platinum electrode of large surface area (52 mesh, 30 mm high, 10 mm i.d.) coated with silver chloride is placed in contact with 10 ml of solution containing a very low concentration of radioactive silver. The solution is stirred magnetically. The electrode is then removed from the solution, washed with nitric acid, rinsed with acetone, and the radioactivity counted.

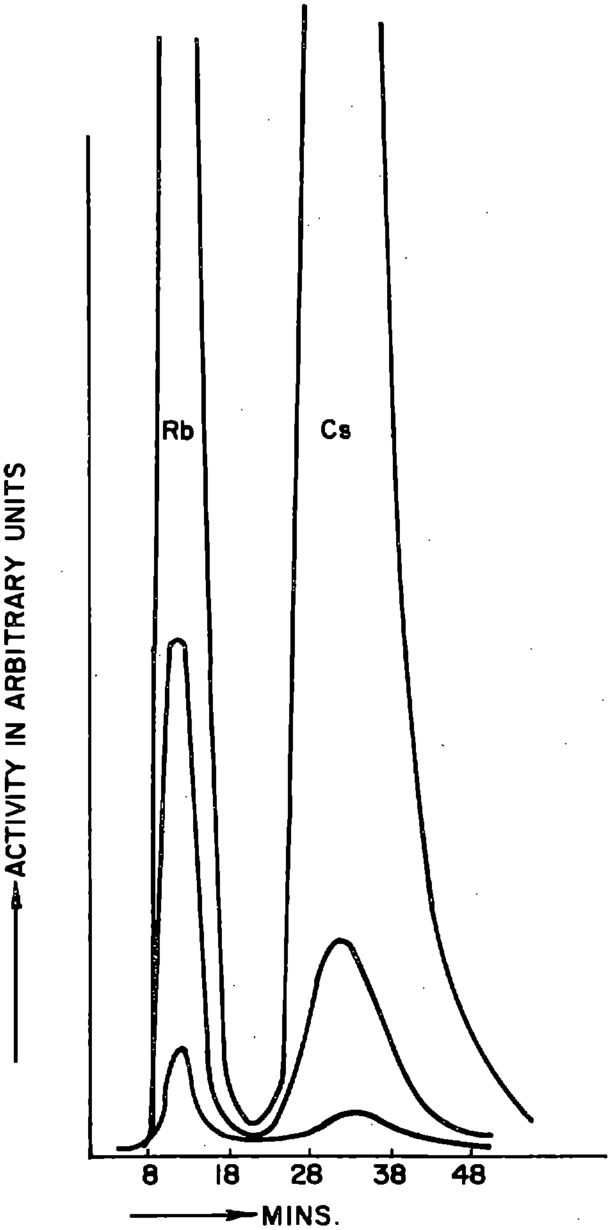

Figure 12. Ionic exchange separation of cesium and rubidium activities in fission product, by zirconium tunstate column (52).

Some of the experimental results are shown in Table I. Contaminations by other radionuclides are very small.

Table I. The Yield of Radioactive Silver from 1 M HNO_3 by the Isotopic Exchange Procedure

AgCl (mg)	Immersion Time	Temp. (°C)	Yield (%)
2	5 min.	25	97
2	2 min.	95	98
2	20 sec.	95	50
0.3	5 min.	25	85-90

Similar rapid exchange reactions were also found in the system between bromide ion and freshly prepared silver bromide (73,148), and it was shown that 96 to 98 percent of the bromide activity could be carried by the silver bromide precipitate in 10 to 45 minutes.

Another interesting isotopic exchange method in radiochemical work is the amalgam exchange technique (60). Separation of the radioisotope takes place by virtue of the rapid exchange which is known to occur between an element in the form of a dilute amalgam and its ions in solution. If there are many more inactive atoms of the element in the amalgam than there are of its radioisotope in solution, most of the activity will be incorporated in the amalgam.

Recent reports describe the successful application of this technique to the separation of Zn^{65} in reactor effluent (233), rapid radiochemical separation of cadmium activity in fission products (63), and the radiochemical separation of indium (217).

In the case of cadmium (63), the initial exchange with cadmium amalgam in the extraction step is followed by a back extraction or elution with thallous ion to selectively remove the cadmium from contaminants in the mercury. This method has proven to be rapid (8-10 minutes) and in addition, to give a high degree of separation from many elements. For most elements, this separation technique is at least 10 times better than the best separation technique previously used for cadmium, such as dithizone extraction or ion exchange. Of the 21 elements tested, only selenium, indium, and thallium contaminate to any extent through the overall procedure.

G. Electrochemical Methods

1. Electrical migration method. An interesting electrical migration method for rapid separation of carrier-free radioisotopes has been reported (94). The apparatus consists of a stack of filter papers (30 filter papers were used) moistened with electrolyte and placed between two platinum electrodes. In a typical separation, a sample of solution containing one or more carrier-free radioisotopes and possibly other stable elements in milligram amounts, was evaporated onto a circular filter paper and placed in the center of the stack. A direct current of 0.04 amp/cm^2 for a half hour was sufficient to move ions through 15 layers of filter paper.

By a proper choice of complexing ions and pH, the desired activity can be moved to either one of the electrodes or retained at the initial position in the center of the stack. The activity is usually concentrated in less than 5 adjacent papers.

The practical application of this technique is illustrated in Table II.

2. Polarographic method. Application of the polarographic technique to the detection of short-lived daughter activity has been reported (163). If the radioactive isotope is reduced to the zero oxidation state, the resulting metal forms a dilute amalgam with the mercury drop. This is then removed from the solution and its radioactivity measured very rapidly.

Table II.(*) Carrier-Free Radioisotope Separation Accomplished by Electrical Migration in Filter Paper

Carrier-Free Radioisotope		Separation From			Electrolyte
Element	Direction of Migration	Element	Amount	Direction of Migration	
Nb	Anode	Mn	mg	Cathode	1 N Ammonium oxalate
Nb	None	Mn	mg	Cathode	3 N HCl
Nb and Zr	Anode	Y and Rare Earth	(?)	None	1 N Ammonium oxalate
As	None	Cu	mg	Cathode	3 N HCl

(*) This table was cited from Wahl, A. C. and Bonner, N. A., "Radioactivity Applied to Chemistry", John Wiley and Sons, Inc., New York (1951).

Although the yield of the separation is extremely low, i.e., estimated as 6.88 x 10^{-4}% in the separation of Zr^{95}, this method is useful as a good qualitative radiochemical analysis technique for a mixture of radioactive isotopes, because it does not require prior knowledge for the addition of inactive carriers as do most other radiochemical procedures.

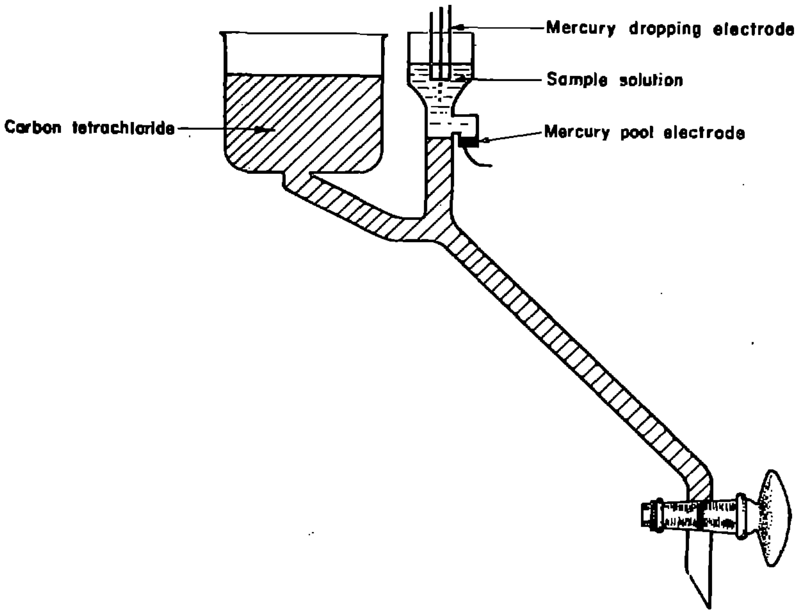

Figure 13. Polarographic cell used for rapid radiochemical detection of short-lived daughter nuclide (163).

The polarographic cell used is shown in Fig. 13. The active solution is placed above the carbon tetrachloride in contact with the mercury pool electrode or with the calomel electrode through agar gel. The dropping mercury capillary is placed in the solution. The purpose of the large volume of carbon tetrachloride in the other compartment is to keep the solution - carbon tetrachloride interface at a constant height.

The mercury drops fall through the solution and are removed by turning the stopcock. The distance through which the mercury drops fall through the carbon tetrachloride can be quite long when the solutions are highly radioactive and the cell compartment may require shielding. The size of the polarographic cell can be either large or small depending on the volume of the solution to be analyzed.

With this apparatus, an attempt was made to detect the 30-second Rh^{106} daughter in the Ru^{106}-Rh^{106} system. A sample solution of 500 λ in 1 M potassium thiocyanate solution was used. The mercury drops were collected at various potentials and it was found that about a constant radioactivity was separated at any voltage more negative than -0.7 volts vs. mercury pool electrode. The time required from the formation of one drop to the beginning of the counting of this drop was less than 30 seconds and the decay curve was traced.

By this means the half-life of Rh^{106} was estimated as 29.3 ± 0.65 seconds. The same technique was also utilized in the rapid analysis of Tc^{99m} and Ru^{106} in fission products (165) using the polarographic cell of Fig. 14. A 1 ml sample is

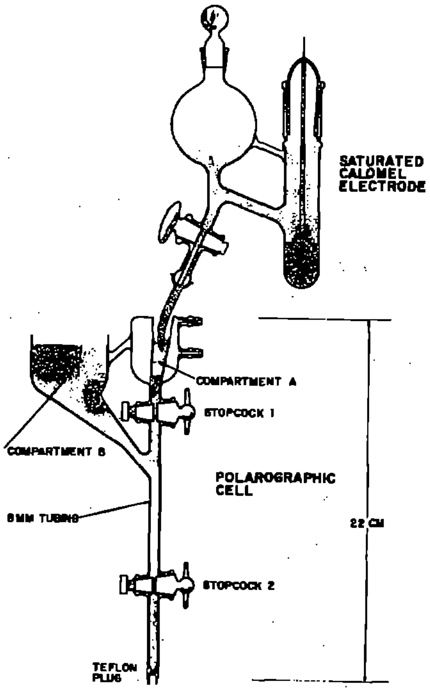

Figure 14. Polarographic cell (165).

placed in compartment A. The authors separated Tc^{99m} instead of Mo^{99} as a monitor of total fissions and describe a separation using this cell which requires 3 minutes and permits 48 analyses to be done in one day by 2 people. A disadvantage of the method is that the yield of the separation is only about 0.1% in 3 minutes but it is satisfactory for fission product mixtures where there is lots of activity.

3. "Focusing ion exchange". An ingenious application of inorganic paper electrophoresis has been described in detail by Schumacher in several articles in Helvetica Chimica Acta [40, 221, 228, 234, 2322 (1957); 41, 825, 1771]. He has used the simple apparatus shown in Figs. 15 and 16 to effect separations of carrier-free Sr-Y-Cs in 10 minutes and alternate rare earths in 8 minutes. By adjusting conditions he reports separation of Sr-Y in 5 seconds.

The disadvantage of the method as outlined by Schumacher is the lack of emphasis placed on the hazard of the lethal voltages involved in such a separation using the simple equipment shown in Figs. 15 and 16. 60 ma of current is required

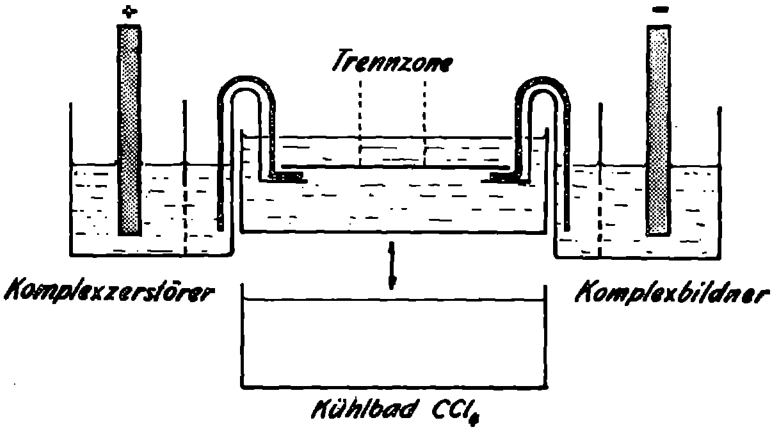

Figure 15. Equipment for focusing ion exchange.

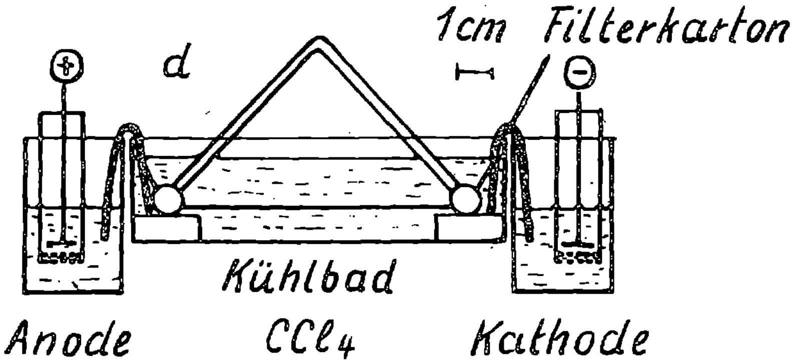

Figure 16. Equipment for focusing ion exchange.

at 1600 volts for a typical separation. However standard electrophoretic separations routinely utilize this combination of current and potential safely with specially designed equipment with interlock systems, and one would hope that adaptations of such equipment might become available for utilization of focusing ion exchange.

III. PRACTICAL EXAMPLES (ARRANGED BY ELEMENT)

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
He^6	0.82 s	$Be^9(n,\alpha)$	$Be(OH)_2$ powder	Distillation	During irradiation, a stream of H_2 is passed through a capillary tube to a thin-walled jacket around a thin-walled GM tube.		17
C^{10}	19.1 s	$B^{10}(p,n)$	H_3BO_3 $CaF_2 \cdot BF_3$ Boron metal	Distillation; precipitation	After or during irradiation, the radioactive gases formed are swept into Ascarite layer by a flushing gas; the CO_2 is absorbed in the Ascarite.	1 sec	228, 229
					The radioactive gas is bubbled into $Ba(OH)_2$ solution. CO_2 is ppted. as $BaCO_3$.	20 sec	228, 229
C^{11}	20.4 m	$B^{11}(p,n)$ $N^{14}(p,\alpha)$		Recoil; precipation	The recoil products are collected on paper. The paper is burned in atmosphere of O_2. The gas products are bubbled through $Ca(OH)_2$ soln. and CO_2 is ppted. as $BaCO_3$.		12
		Al(p)	Al foil	Distillation	The sample is dissolved in NaOH soln. in a small reaction vessel through which is passed a slow stream of air saturated at room temp. with ethanol vapor. The gas stream is dried roughly by bubbling into conc. H_2SO_4 and is then passed over CuO at 600°C to convert carbon compounds to CO_2. The gas is then bubbled through NaOH soln. to absorb CO_2 and the CO_2 ppted. as $BaCO_3$ by addition of $Ba(OH)_2$.		36, 37
N^{13}	10.05 m	Al(p)	Al foil	Distillation	The sample is dissolved in boiling NaOH soln. to which NH_4^+ carrier has been added. The nitrogen activity appears as NH_3 and is trapped on a filter pad moistened with dil. H_2SO_4.		37
		Al(p) Zn(p) In(p) Pb(p) U (p)	Metallic foil	Distillation	The Al is dissolved in 6 N HCl containing Fe^{++}, $PtCl_4$ and NH_4^+ carrier. After addition of 10 <u>N</u> NaOH, the evolved NH_3 gas is swept out and caught in 1 <u>N</u> NaOH + $KMnO_4$ soln. at 0°C. <u>Or</u> by heating the soln., the NH_3 gas is distilled by aid of He carrier gas and caught in sat. H_3BO_3 soln. containing a definite amount of HCl. After addition of NaOH to the boric acid soln., the NH_3 is distilled once again into a similar sat. H_3BO_3 soln.		66

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					The In is dissolved in 12 N HCl containing Fe^{++} and $PtCL_4$. The Pb is dissolved in 48% HBr containing Fe^{++} and $PtCl_4$. U is dissolved in 6 N HCl containing Fe^{++}.		
N^{16}	8 sec	$O^{16}(n,p)$	KNO_2	Distillation	A pellet of KNO_2 (about 300 mg) which is sufficient to produce about 75 cc of N_2 gas, is irradiated in the pile for 20 sec. It is then reacted with a hydrazine hydrochloride soln. The evolved N_2 gas is collected and measured.	7 sec	200
N^{17}	4.14 s		NH_4F aqueous soln.	Distillation	Using He carrier gas, the radioactive gas is swept through H_2SO_4 to remove H_2O, Ascarite to remove CO_2, CuO heated in a furnace to oxidize CO to CO_2, Ascarite again to remove this CO_2, Cu or Mg heated in a furnace to remove O_2 and finally through active charcoal at liquid air temp. to remove Ne. The gas that comes through is N_2 and is measured by a neutron counter.		2
O^{14}	72 s	$N^{14}(p,n)$	KSCN	Distillation	The gas released by bombardment is swept through $CaCl_2$. The oxygen activity appears as H_2O and is absorbed in the $CaCl_2$.		229
			Gas mixture of H_2(30%) and N_2(70%)		The bombarded gas is swept through a tube of platinized asbestos and then through $CaCl_2$ to catch the H_2O.		229
O^{15}	124 s	$N^{14}(d,n)$	N_2	Distillation	The irradiated gas is mixed with some O_2 and an excess of H_2, and is passed over heated platinized asbestos. The oxygen present as NO_2, N_2O, or O_2 is reduced to H_2O and collected in a $CaCl_2$ drying tube and measured.		162
Ne^{23}	40.2 s	$Mg^{26}(n,\alpha)$	$Mg(OH)_2$ ppted. together with $Fe(OH)_3$	Distillation	Air from an aspirator is passed at a suitable rate through the samples and then through a glass tube to the thin walled jacket around a counter.		18
Ne^{24}	3.38 m	$Ne^{22}(t,p)$	Ne	Distillation	The sample gas is passed through a trap of activated charcoal at dry ice temp. and then to the counting		67

					cell. All likely impurities such as Na and F (with the exception of He and Ar) are removed from the Ne by the trap.		
Na^{24m}	0.02 s	Daughter of $Ne^{24}\beta^-$ dis.		Distillation Gas electrolysis	The daughter activity of Ne^{24} is collected on a negative copper electrode (6 volt field) in which a detector is placed near the electrode. Before counting, the Ne activity is removed from the system by suction.	10 msec	67
Mg^{27}	9.45 m	$Al^{27}(n,p)$	Al metal	Precipitation	The Al is dissolved in NaOH and $Mg(OH)_2$ ppted. by addition of $MgCl_2$ carrier.		3
		$Mg^{26}(n,\gamma)$	Blood cell, blood plasma	Precipitation	The carriers of Mg (10 mg), Cu, Zn, and Mn are added to the irradiated sample. The soln. is swirled and stirred and allowed to stand for about 5 min. By adding conc. HCl (15 drops) to make 2 N acid, blood protein is ppted. After centrifuging and decanting, $Mg(OH)_2$ is ppted. from the resulting soln. by adding 3 ml 12 N NaOH. After centrifuging, the ppt. is dissolved in HCl (10 drops) and the soln. diluted to 10 ml. $Mg(OH)_2$ is ppted. once again and dissolved. To the soln. (8 ml) are added 10 drops of 2 MNH_4Cl. This is then made alkaline by addition of 6 N NH_4OH (3 drops excess from neutral) and heated gently. MnS and CuS are ppted. from the soln. by addition of sat. $(NH_4)_2S$ soln. (20 drops). After centrifuging and decanting, Mg is ppted. as $Mg(OH)_2$ from the soln. by addition of 2 ml 12 N NaOH. The $Mg(OH)_2$ is dissolved in conc. HCl (6 drops) and the soln. is diluted to 10 ml. After addition of Cu carrier and 5 ml 20% Mannitol soln., $Mg(OH)_2$ is ppted. by addition of 12 N NaOH. The above final dissolution and pptn. are repeated. The final ppt. is counted.		220
P^{34}	12.5 s	$Cl^{37}(n,\alpha)$	KCl or NaCl	Precipitation	To the irradiated KCl or NaCl soln., sodium phosphate and nitric acid are added. Then P^{34} is ppted. as phosphomolybdate.	~ 30 sec	273
S^{37}	5.04 m	$Cl^{37}(n,p)$	KCl or NaCl	Precipitation	The irradiated KCl or NaCl is dissolved and H_2SO_4 is added as carrier. The S^{37} is ppted. as $BaSO_4$.	40 sec	273

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
K^{38}	7.7 m	$Cl^{35}(\alpha,n)$	LiCl $NiCl_2$ NaCl	Precipitation	The sample is dissolved in a dil. KCl soln. K is ppted. as potassium cobaltinitrite.		124, 216
			LiCl		The sample is dissolved in H_2O (~ 5 ml). K is ppted. by addition of 10 ml naphthol-yellow-S (5% soln.). After centrifuging, the ppt. is recrystallized from about 5 ml NYS 5% in ice. $Fe(OH)_3$ scavenging is also done. Yield is ≥ 50%, depending on speed required.	≤ 40 min	22
K^{44}	22 m	$Ca^{44}(n,p)$	Ca metal or $Ca(OH)_2$	Precipitation	The sample is dissolved in dil. HCl. After addition of KCl carrier, Ca is ppted. as the oxalate in alkaline soln. After filtration, K is ppted. as the perchlorate by addition of $HClO_4$ and ethyl alcohol.		265
Ca^{45}	164 d	$Sc^{45}(n,p)$	Sc_2O_3	Extraction	The sample is dissolved in HCl and excess HCl is evaporated off. The soln. is diluted with H_2O to 0.5 mg Sc/ml concentration and extracted with an equal volume of 0.5 M TTA in benzene by stirring for 10 min (Sc and more extractable cations are removed). Then the aqueous phase is adjusted to pH 5 with NaOH and again extracted with 0.5 M TTA soln. (Sc and all cations extractable between pH 1 and pH 5 are removed). Then the aqueous phase is adjusted to pH 8.2 and extracted with 0.5 M TTA. The Ca^{45} is extracted into benzene phase and back-extracted with H_2O. Yield is > 99%.		21
Ti^{51}	5.8 m	$Ti^{50}(n,\gamma)$	Biological material, rocks, minerals, alloys		The sample, irradiated in a gelatin capsule, is put into soln. either by digesting with HNO_3 or by fusion with Na_2O_2 in a nickel crucible which contains 0.8 mg Ti carrier.	~ 10 min	140
				Extraction	1) The fused material is dissolved in H_2SO_4 soln. Ti is coppted. with $Fe(OH)_3$. The ppt. is dissolved in 20 ml of 10% H_2SO_4. The Ti is then extracted into 10 ml of isopropyl ether by addition of 10 ml of 6% aqueous cupferron. Contaminants, such as Cu, V, and Mo, are back-extracted into 20 ml of cooled 15% NaOH soln. The ether phase is counted by γ-spectrometry. Yield is ~ 70% and is determined colorimetrically.		

				Precipitation	2) The Ti in 2 $\underline{N}$ H_2SO_4 is coppted. with Zr-p-hydroxyphenylarsonic acid and the ppt. counted by γ-spectrometry. Yield is ~ 85%.		
V^{52}	3.76 m	$V^{51}(n,\gamma)$	Biological ash	Extraction	The sample is digested in 15 ml hot 4 $\underline{M}$ NaOH soln. containing V carrier (10 mg). Conc. HCl (5-10 ml) is added to the soln. to dissolve the remaining ash.		
					1) The V is reduced to V(+4) by addition of 10% $SnCl_2$ soln. (3 drops). After diluting the soln. to 50 ml and making pH ~ 5 by using 1 M NaOH, the V is extracted into an equal volume of 0.25 M TTA benzene soln. The V is back-extracted into 25 ml 6 $\underline{N}$ HCl from the benzene soln. After the V(+4) is oxidized to V(+5) by 1 M $KMnO_4$ (5-10 drops), the V is ppted. by 2 ml 6% cupferron aqueous soln. and filtered. Yield = ~ 30%.	~ 10 min	90
					2) The HCl acidic soln. is diluted to 50 ml with H_2O. The V is extracted into 10 ml $CHCl_3$ by addition of 2.5 ml 6% cupferron aqueous soln. and 10 ml $CHCl_3$. The extract is measured by γ-spectrometry. Yield = ~ 40%.	~ 4 min	90
			Cracking catalyst	Extraction	40-100 mg sample (finely ground) in a gelatine capsule, irradiated in pile for 2 min, is fused in 3 gm Na_2O_2 containing a known amount of V^{48} tracer. The melt is dissolved in 100 ml soln. containing 16.7 ml conc. HCl, 3 ml Al carrier, 0.5 ml V(+5) carrier, and 15 ml H_2O_2. From this soln., Al is ppted. as the oxinate by addition of 30 ml 10% oxine (1:4 acetic acid) and 60 ml conc. NH_4OH. After filtration of the Al ppt. by 120 mm medium glass frit filter, the filtrate is acidified with HCl to 1-2 $\underline{N}$ HCl and cooled with ice. The V is extracted into 5 ml $CHCl_3$ by addition of 6 ml 6% cupferron aqueous soln. The V extracted is measured by γ-spectrometry. Yield = Variable (up to 70%).	10 min	25
			Biological tissue	Extraction	The sample is fused in 10 g Na_2O_2 containing V^{48} tracer, 10 mg V carrier and 10 mg Cu holdback carrier. The melt is dissolved in 50 ml H_2O and 42 ml conc. HCl is added. After addition of 10 gm tartaric acid, Cu is ppted. by H_2S and filtered. The V is extracted into 10 ml $CHCl_3$ by addition of 10 ml 6% cupferron aqueous soln. The extract is measured by γ-spectrometry.	~ 5 min	132

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Cr^{51}	27.8 d	V^{51}(d,2n)	Vanadium metal	Distillation	The irradiated V target is transferred into a Pt dish and dissolved by heating in approx. 5 ml conc. HF. The excess HF is removed by fuming down with 10 ml of conc. H_2SO_4, whereafter the H_2SO_4 soln. is transferred to the distillation apparatus. 50 ml of conc. HCl is added dropwise at a temp. of 250°C. After complete distillation of As and Sb as their chlorides, a mixture of 50 ml HCl/HBr (1/3) is also added dropwise at the same temp. to insure removal of Sn as $SnBr_2$. The bromide formed in the soln. is swept from the soln. by a stream of dry gaseous HCl. 30 ml of 70% $HClO_4$ is added and the chromium activity distilled as CrO_2Cl_2 at a temp. of 250°C. A slow stream of dry HCl gas is maintained throughout the distillation. The chromylchloride is collected in ~ 5-10 ml H_2O. This soln. can be taken to dryness without Cr losses, after addition of a small excess of hydrazine hydrate. The excess hydrazine is finally destroyed with 6 N HNO_3. Yield = quantitative.		58
Cr^{55}	3.52 m	Cr^{54}(n,γ)	$Cr(OH)_3$	Precipitation	The sample (0.3 gm) is dissolved in conc. HCl. V carrier (5 mg) and H_2O are added to the soln. After boiling, V is ppted. and separated as V-cupferrate. After filtering, the Cr-activity is ppted. as silver chromate and filtered.		83
		Mn^{55}(n,p)	$MnSO_4$	Precipitation	The 0.3 g target is dissolved in 30 cc hot 0.1 N HNO_3. 70 mg $K_2Cr_2O_7$ and 30 mg $Cr(OH)_3$ are added as carriers. After cooling, the V-activity is ppted. by 60 mg cupferron. After a double V-separation, the soln. is made alkaline and H_2O_2 added. After boiling and separating Mn-hydroxide, Cr is ppted. as $BaCrO_4$ and filtered.		
				Extraction	To the irradiated $MnSO_4$, Cr and V carriers are added. The Cr in the soln. is oxidized by H_2O_2 to perchromate and extracted into ether at pH = 1.7. The organic layer is measured. Or the Cr activity can be back-extracted by 1 N KOH and ppted. as $BaCrO_4$ in acetic acidic soln., and filtered.		

Mn^{52m}	21.3 m	$Fe^{54}(d,\alpha)$	Iron metal	Precipitation	The sample is dissolved in a mixture of HCl + HNO_3 and small amounts of carrier of the appropriate elements are added. The soln. is adjusted to a concentration of 16 N in HNO_3 for the precipitation of Mn by addition of solid $KClO_3$ to the boiling soln. The MnO_2 is usually purified by redissolving it in an acid soln. of H_2O_2 and by again ppt'ing. from a boiling soln. of 16 N HNO_3 by the addition of solid $KClO_3$.		161
Mn^{57}	1.7 m	$Fe^{57}(n,p)$	Fe^{57} enriched sample				47
Mn		$Cu\begin{pmatrix}d\\p\\\alpha\end{pmatrix}$	Copper metal	Precipitation	The Cu is dissolved in a minimum amount of conc. HNO_3 and evaporated to near dryness. Carriers (Zn and below) including 5 mg Mn are added and the soln. is made to 1 N HCl. After pptn. of CuS, Mn is ppted. as MnS in ammonical soln. The MnS ppt. is dissolved in 16 N HNO_3 and volume made up to 4 ml with fuming HNO_3. Then MnO_2 is ppted. by addition of 2 or 3 crystals of $KClO_3$. The MnO_2 is dissolved in HNO_3 acid soln. containing H_2O_2 and re-ppted. by the above method. Yield = 75%.	30 min	15
Fe^{53}	8.9 m	$Cr^{50}(\alpha,n)$	Chromium metal	Precipitation	1) The bombarded Cr is dissolved in 5 ml of 8 N HCl. $FeCl_3$ carrier (Fe-15 mg), 12 ml conc. H_2SO_4, and H_2O to make 100 ml is added. After oxidation by addition of a small amount of $KMnO_4$, and cooling to 10°C, Fe is ppted. by addition of 6% cupferron. The ppte. is filtered and washed with 10% H_2SO_4 containing 0.2 g cupferron per 100 ml and measured.		216
				Extraction	2) The Cr is dissolved in HCl and made 6 N in HCl. The Fe activity is extracted as $FeCl_3$ in an equal volume of ether. The ether phase is washed by shaking with 4-5 successive portions of equal volume of 6 N HCl to remove all traces of radioactive Mn and Co.		160
Fe^{61}	5.5 m	$Ni^{64}(n,\alpha)$	Ni foil	Precipitation	The target (3-5 mg) is dissolved in 0.5 ml hot conc. HNO_3. After addition of 2 mg Co, 2 mg Cu, and 2 mg Fe (each in 2 ml chloride soln.), Fe is ppted. as the hydroxide by NH_4OH, digested in water bath and centrifuged. The ppt. is dissolved in conc. HCl (few drops) and the above $Fe(OH)_3$ pptn. is repeated and filtered. Yield = ~ 90%.	3-5 min	155

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Fe		As(d)	Arsenic	Extraction	The As is dissolved in a minimum of HNO_3 + HCl. After addition of 2 mg Fe and Ga, the soln. is adjusted to 6 $\underline{N}$ HCl. The Fe is extracted twice with ether and washed 5 times with 1/3 volumes 6 $\underline{N}$ HCl. The Fe is back-extracted into H_2O by washing twice with 1/4 volume H_2O and ppted. as $Fe(OH)_3$ in 1 $\underline{M}$ KOH. The $Fe(OH)_3$ ppt. is dissolved in minimum conc. HCl and diluted to 5 ml. After addition of 1 mg Ga hold-back carrier, the $Fe(OH)_3$ is ppted. This final dissolution and pptn. are repeated until the supernates (Ga fraction) become inactive.	30 min	120
Fe		Fe(n,γ)	Reactor coolant water containing activated process water, chemicals, activated corrosion products, and fission products. Aluminum metal	Extraction	The sample is dissolved in HNO_3 or aqua regia; 3 ml $HClO_4$ is added and evaporated to 1 ml (caution). 10 ml conc. HNO_3 is added. Soln. is heated to boiling and 2 ml H_2O added to bring the soln. to 12 $\underline{N}$ HNO_3 soln. The Fe is then extracted into an equal volume of 0.45 $\underline{M}$ TTA-benzene soln. by shaking for 15 min. The organic phase is washed 3 times with equal volumes of 3 $\underline{N}$ HNO_3 by shaking for 5 min each time. The washed organic phase is counted by γ-spectrometry. Yield = ~ 90%.		211
Co^{60m}	10.5 m	Co^{59}(n,γ)	Biological tissue	Extraction	The sample is fused in 10 g Na_2O_2 + 3 NaOH pellets containing Co^{60} tracer and 10 mg Co carrier. The melt is dissolved in 50 ml H_2O and cooled with 50-70 ml liquid N_2. After addition of 15-20 ml glacial acetic acid and cooled with liquid N_2, the Co is extracted by 25 ml oxine soln. in $CHCl_3$ (3%). The Co is back-extracted into 10 ml 9 $\underline{N}$ HCl, ppted. with Na_2O_2, and filtered. Yield = ~ 40%.	15 min	133
Co^{62}	13.9 m	Ni^{64}(d,α)	Nickel oxide enriched in Ni^{64}	Ion exchange	The nickel oxide target (about 50 mg) is dissolved in 10 $\underline{N}$ HCl and this soln. is then placed on an ionic exchange column (8 mm in diameter and 140 mm tall) with a Dowex-2 resin that has been previously washed with		92

					conc. HCl. The Ni was first removed by elution with 8 N HCl and then the Co removed with 4 N HCl. Cu from the (d,n) reaction remains on the column. The Co fraction is measured after evaporation on a counting cup.		
			Nickel foil	Extraction	The activated metal is dissolved in HNO_3, the nitrates are converted to chloride, and 2 mg Co and 2 mg Fe as chloride are added. After the pptn. of Fe by NH_4OH, the filtrate is acidified slightly with HCl, saturated with NH_4SCN, and Co is extracted with an equal volume of amyl alcohol-ethyl ether (1:1). Co is recovered from the organic layer with 6 N NH_4OH. After acidification with HCl, the above extraction procedure is repeated. Finally, Co is ppted. with NH_4HS and mounted for counting as dried CoS or as CoO. Yield = ~ 90%.	12 min	208, 156
Cu^{60} Cu^{62}	24 m 9.7 m	$Ni^{60}(p,n)$ $(d,2n)$ $Ni^{62}(p,n)$	Nickel foil	Precipitation	The foil (3-5 mg) is dissolved in 0.5 ml 6 N HNO_3 and evaporated to dryness. Conc. HCl (3-4 drops) is added and the soln. is evaporated to dryness. While warming, Cu and Co carrier (each 2 mg), 0.5 ml conc. NH_4HSO_3 soln., and 10% NH_4SCN soln. are added to ppt. CuSCN. The ppt. is filtered and washed with a 0.1 N NH_4SCN - 0.1 N NH_4HSO_3 soln. and a few drops of acetone. Yield = 50-75%.	4-5 min	157
Cu^{66}	5.1 m	$Cu^{65}(n,\gamma)$	Biological tissue	Extraction	The sample is fused in 10 gm Na_2O_2 + 3 NaOH pellets + 10 mg Cu carrier. The melt is dissolved in 50 ml H_2O. After addition of 30 ml conc. HCl and cooling by pouring on 50-75 ml liquid N_2, the Cu is extracted into 5 ml CCl_4 by addition of 10 ml 6% cupferron aqueous soln. (1.2 N HCl). The Cu is back-extracted to the aqueous layer with 10 ml conc. NH_4OH, ppted. as CuS with H_2S and filtered. Yield = 80%.	8 min	134
Cu^{62} Cu^{66}	9.7 m 5.1 m	$Cu\binom{N}{O}$	Copper foil	Precipitation	Sample is dissolved in dil. HNO_3. After the pptn. of $Fe(OH)_3$ by ammonia, Cu is ppted. by 1-α-benzoinoxine in ammonical tartaric acid soln. For more rapid separation, Cu is ppted. and counted as thiocyanate after the separation of Se and As in 9 N HCl by hypophosphite.	5 min	16
Cu^{68}	32 s	$Zn^{68}(n,p)$	$ZnSO_4$	Precipitation	20 g zinc sulphate in aqueous soln. is irradiated. After irradiation, Zn powder is added and filtered. The filtered Zn powder contains the Cu activity. In-		84

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					stead of Zn powder, Sn powder can also be used as a reductant for Cu. In this case, contamination of Ni can be avoided.		
Zn^{60} Zn^{61}	2.1 m 1.5 m	Ni(α,2n) Ni(α,n)	Nickel foil	Precipitation or extraction	The irradiated Ni foils are dissolved in HNO_3 and the Cu activity is separated by spontaneous deposition on Zn dust in weakly acidic medium or by pptn. of Cu(I) thiocyanate after reduction with sulfurous acid. Then the filtrate is counted for the zinc. In the other method, zinc is extracted with $CHCl_3$ as a Zn-di-β-naphthyl thiocarbazone compound which separates Zn from Cu, Ni, Co, and Fe.		159
				Ion exchange	The Zn is separated by anion exchange method from Ga, Cu, Ni, Co, Fe, and Mn.		53
Zn^{65}	245 d		Reactor cooling water	Isotopic exchange	A 100 ml sample of water is made 0.1 M in tartaric acid and is added to 6 ml of zinc amalgam (1 gm Zn in 5 ml Hg) in a 125 ml separatory funnel. The mixture is agitated for 8 min on a mechanical wrist shaker. The amalgam phase is separated into a suitable container and counted on a γ ray spectrometer. Yield = ~ 99%. Effective decontamination is obtained from all isotopes except Cu^{64} and Mn^{56}. Mn^{56} would not be a serious interference in monitoring application in waste streams. Cu^{64} can be removed by pre-extraction into cadmium amalgam (1 gm Cd in 5 ml Hg). The cadmium amalgam will reduce and extract the Cu, and leave the radiozinc in solution.	~ 10 min	233
Ga^{64}	2.6 m	Zn^{64}(p,n)	Zinc foil or zinc oxide enriched in Zn^{64}	Extraction	The sample is dissolved in HCl and made 6 N in HCl. The Ga is extracted as the chloride into ether. The ether phase is washed with 6 N HCl and evaporated.		50
Ga^{70} Ga^{72}	21.4 m 14.2 h	Ga^{69}(n,γ) Ga^{71}(n,γ)	Iron meteorite	Extraction	The sample is dissolved in hot conc. HCl. 4-5 mg Ga^{+3} carrier is added. The soln. is decanted to remove any insoluble residue (seldom more than 0.1% by weight). The soln. is adjusted to 5.5 to 6.5 M in HCl. The Ga is extracted with equal volumes of ether saturated		23

					with HCl and then back-extracted into H_2O from the ether phase. This procedure is repeated. $Fe(OH)_3$ scavenging is done by addition of NaOH (OH-concentration : 1-2 M) and one drop of aerosol. After centrifugation, the Ga is ppted. as Ga-oxinate by addition dropwise of 5 ml of a solution of 1% oxine in acetic acid and 3 M ammonium acetate. The ppt. is filtered, washed with water, and then with ether.		
Ge^{69}	39.6 h	Daughter of As^{69}(15m) by $Ge^{70}(p,2n)As^{69}$	GeO_2	Distillation	The As^{69} sample, isolated from the GeO_2 target (see section on As^{69}), is prepared as a soln. in HCl containing Ge carrier. The Ge is then quantitatively isolated for 2 min in a current of Cl_2. The Ge in the distillate is ppted. as the sulfide.		28
Ge^{73m}	0.53 s	Daughter of As^{73}(76d)		Distillation	When N_2 gas is swept through a soln. of As^{73} (+5 valence) in conc. HCl at 100°C, a rapidly decaying Ge^{73m} activity is found in the exit gas system. (Although the Ge activity could be separated by this technique at 25°C, better yields were obtained for the separation at the higher temp.) A 5 ml aliquot of soln. containing 0.4 mC As^{73} is placed in an 8 ml flask fitted with an inlet for introducing N_2 gas and an outlet leading to a lusteroid tube in a shielded well-type NaI counter. Small diameter plastic tubing (1.5 mm i.d.) is used between the flask and the tube in the counter to allow rapid transfer of the gas. A small trap is introduced into the line to prevent droplets of active solution from being carried over by the gas stream.		33
					This technique could be simplified (1) using low temperature (25°C) gas-sweeping techniques for removal of Ge(IV) from 12 N HCl solns. and (2) by absorbing Ge(IV) from the gas stream on an anion exchange resin (Dowex-1X8, 50-100 mesh). It was studied by using Ge^{77}(12 hr) → As^{77}(40 hr) tracer. The Ge absorbed in the resin can be easily recovered by washing the column with a few column volumes of water or, preferably, of dil. HCl soln. Since the first part of the effluent is rather concentrated in HCl, some care must be exercised to avoid loss of Ge(IV) by volatilization from the effluent.	The half-time of removal of Ge(IV) was about 2 min.	192

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
As^{68}	~ 7 min	$Ge^{70}(p,3n)$	GeO_2	Precipitation	20 gm of "specpure" GeO_2 were irradiated. The target is dissolved in 3 ml of 27 N HF. 5 ml of 6 N NH_4OH is added and the As ppted. as sulfide by passing in H_2S. After a second pptn. with inactive Ge holdback carrier, the arsenous sulfide is isolated for counting.		28
As^{69}	15 min	$Ge^{70}(p,2n)$					
As^{78}	91 min	$U^{235}(n,f)$	Uranyl nitrate	Extraction	1-2 g of finely ground U-nitrate is irradiated for 2-3 min and then dissolved in 6.5 ml H_2O to which is added 2.0 ml (20 mg) Ba carrier and 2.0 ml (20 mg) arsenic (V) carrier and 3 drops conc. HCl. The soln. is transferred to a separatory funnel containing 20 ml benzene. 4.5 ml 10.2 N HI (commercial 67%) is added and the funnel immediately shaken for 10 sec. The As in the benzene phase is back-extracted with 6.5 ml H_2O for 30 sec and the aqueous phase is washed with 10 ml benzene for 5 sec. The extraction and back-extraction are repeated. Sn and Sb and their decay product Te are found as contaminants. Therefore more purification steps must be included in order to achieve further decontamination. 1.0 ml (10 mg) Te(IV) carrier and 3.5 ml conc. HCl are added to the water phase. The soln. is heated nearly to boiling and reduced with SO_2. After filtering, the supernatant soln. is boiled to drive off excess SO_2. The soln. is cooled and 20 ml conc. HCl added. Arsenic sulfide is ppted. with H_2S, centrifuged off, decanted and washed with 6 N HCl soln. saturated with H_2S. The arsenic sulfide is dissolved in a solution of 0.5 ml conc. HCl, 0.5 ml conc. HNO_3, and 1.0 ml $HClO_4$, heated on a water bath for 5 min and thereafter heated strongly to fuming. After cooling, extraction and back-extraction, pptn. as sulfide and dissolution is performed as described above. The final As soln. is diluted to 20 ml with 6 N HCl. Metallic As is ppted. by adding 100 mg KI and 1 g NaH_2PO_2 and heating on water bath for coagulation. The ppt. is filtered and washed with alcohol and ether.	1.5-2.0 min	145

As^{79}	9.0 m	U^{235}(n,f)	Uranyl nitrate	Distillation	The irradiated uranium soln. is added to a standardized arsenite carrier in dilute H_2SO_4, oxidized to arsenate (V) with $KBrO_3$ and reduced again with potassium metabisulfite. (This oxidation-reduction procedure is carried out to insure the complete exchange of As-activity with the trivalent As carrier.) The reduced soln. is then placed in an arsine generator with Hg cathode as described by Rogers and Heron [Analyst <u>71</u>, 414 (1946)]. Arsine is generated for 15 min with the cell at about 80°C, at a cathode current density of about 2.5 amp/cm^2 and passed through Pb(OAc)$_2$ soln. before collection in $AgNO_3$. Sb carrier is added to the $AgNO_3$ soln. followed by NaCl, the soln. filtered and a $Fe(OH)_3$ scavenge carried out. Arsenic metal is then ppted. by ammonium hypophosphite in the presence of Sb holdback carrier from 1:1 HCl soln. The As is dissolved in brominated HCl and then distilled from conc. HBr in the presence of Sb, Te, and Sn holdback carriers, into conc. HCl. As is re-ppted., slurried with H_2O, filtered to a tared filter paper, dried, and counted. Yield = ~ 35%.	45 min	54
Se^{79m}	3.88 m	Daughter of As^{79}(9m) β-disintn.		Precipitation	The Se^{79m} which is grown from As^{79} is separated by direct pptn. from HCl soln. by addition of solid hydroxylamine hydrochloride.		54
Se^{81}	17 m	Daughter of Se^{81m}(57m) I.T.		Recoil	Radioactive selenic acid and HCl are mixed so that the final solution is 4 <u>N</u> HCl. The soln. is then saturated with SO_2. Under such conditions, selenic acid is reduced very slowly whereas selenious acid is reduced very rapidly to Se which can be filtered out of the soln. To the radioactive selenic acid, selenious acid is added at intervals of time, the elemental Se separated, and the decay curve of Se^{81m} measured.		153
Se^{81} Se^{83} Se^{84}	17 m 25 m 3.3 m	U^{235}(n,f)	Uranyl nitrate	Distillation	To 5 ml of neutron-irradiated uranyl nitrate, 2 ml of Se carrier and 10 ml of conc. HBr are added. The Se is distilled into 5 ml of H_2O contained in a 50 ml centrifuge tube placed in an ice bath. SO_2 is passed rapidly through the distillate in an ice bath until the	< 60 min	270

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					red precipitate of Se is coagulated (2 to 3 min). The ppte. is centrifuged and washed with 10 ml of H_2O. The ppte. is dissolved by heating with 5 to 10 drops of conc. HNO_3, evaporated nearly to dryness, and dissolved in 10 ml conc. HCl. The Se is ppted. once again by SO_2 in an ice bath, centrifuged, and filtered. It is washed 3 times with 5 ml ethanol and 3 times with 5 ml ether, dried and counted. Yield = > 80%.		
Se^{81} Se^{83}	17 m 25 m	$Se^{80}(n,\gamma)$ $Se^{83}(n,\gamma)$	Ammonium selenite	Recoil	After the neutron irradiation, the irradiated ammonium selenite is dissolved in 3 N HCl and extracted with CS_2. 20% of the Se activity is found in the CS_2 fraction. The CS_2 is evaporated to dryness, and the Se activity is taken up in conc. HNO_3. In another method, the 6 N HCl solution of the irradiated selenite is filtered through sintered-glass filter. Much of the activity is deposited on the filter. After the water wash, 20% of the original Se activity is removed by passing boiling conc. HNO_3 through the filter.		97
Se	$U^{235}(n,f)$			Precipitation	Se carrier is added as selenite to 3 g UO_2Cl_2 in 5 ml of 4 M HCl solution. The mixture is irradiated for 5 sec. After the irradiation, the active soln. is mixed with 25 ml 12 N HCl. $NaHSO_3$ is added and Se ppted., centrifuged, and washed by decantation. The activity of the ppt. was measured by a neutron counter. (The detection of a delayed neutron emitting isotope of Se was unsuccessful.)	~ 50 sec	247
Br^{78m}	6.4 m	$As^{75}(\alpha,n)$	Arsenous oxide	Precipitation	The target is dissolved in NaOH soln. containing a small amount of KBr carrier. The soln. is made slightly acid with HNO_3 and the Br is ppted. as AgBr.		234
Br^{80}	18 m	Daughter of Br^{80m}(4.5 hr) I.T., $Br^{79}(n,\gamma)$ Br^{80m}		Recoil	Ethyl bromide is irradiated with thermal neutrons and the active bromine is separated as HBr by Szilard-Chalmers process. Then, by saturating with tetrabutyl alcohol to the HBr, tetrabutyl bromide (5 g) is synthesized. The tetrabutyl bromide is dissolved in 200 ml of aqueous alcohol (75 ml %). The radioactive		224

					bromine, which is isolated as bromine ion by recoil effect, is ppted. as AgBr.		
Br^{85}	3.0 m	U^{235}(n,f)	Uranyl nitrate aqueous soln.	Extraction	The target soln. is irradiated for 10 sec in the pneumatic tube in the reactor. Carriers of Br^- and I^- are added to the irradiated soln. which is then transferred to a separatory funnel containing CCl_4 and $KMnO_4$ acidified with HNO_3. The liberated Br is extracted into the CCl_4 layer. This Br extraction is performed within 30 sec from the end of the irradiation. The CCl_4 extract is added to a funnel containing H_2O and $NH_2OH{\cdot}HCl$. The Br is transferred to the H_2O layer. Two oxidation and reduction cycles with $KMnO_4$ as oxidant and $NaHSO_3$ as reductant are then performed. The last aqueous $NaHSO_3$ layer is acidified with HNO_3, the soln. boiled to expel SO_2, and AgBr ppted.	7 min	248
Br^{87}	56.1 s	U^{235}(n,f)	Uranyl nitrate aqueous soln.	Extraction	To 1 ml of the activated soln., 30 ml of 8 <u>N</u> HNO_3 saturated with $KClO_3$ is added. The Br and I fission products are extracted with CCl_4 from the soln. and then the Br is back-extracted with KNO_2 aqueous soln. (weak HNO_3 acidic) from the CCl_4 soln.		235
Br^{88}	15.5 s	U^{235}(n,f)	Uranyl (U^{235}) nitrate soln.	Distillation	The soln. (15 ml) containing 2 g U^{235} in 1 <u>N</u> HNO_3 and 0.5 <u>M</u> $KBrO_3$ is irradiated. The air stream is flowed at a rate of about 1 liter/min through the irradiating soln., and to this air stream, a small flow (about 2 ml/min) of saturated Br_2 vapor is added. The resultant gas flows through a trap, to remove any active soln. which might be carried out of it by foaming or by an accident, and then a trap containing iodine crystals as hold-back carrier for iodine activity. After leaving these traps, the air stream passed through about 10 ml CCl_4, where the Br activity remained and from there through some traps to a vacuum pump, and on to the exhaust vent of the room. A neutron counter is situated adjacent to the CCl_4 solution.	Seconds	212
Br^{89}	4.51 s	U^{235}(n,f)	Uranyl nitrate soln.	Extraction	A solution of uranyl nitrate in 2 <u>M</u> Na_2CO_3, to which I^- and Br^- carriers are added, is irradiated for 5 sec. After irradiation, the soln. is mixed with 10	30 sec	247

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					ml 2 $\underline{M}$ Na_2CO_3 containing Br^- and BrO_3^- carrier and NaOCl in excess of that needed to oxidize I^- to IO_3^-. The soln. is then added to a separatory funnel containing 50 ml CCl_4 and 25 ml 6 N HNO_3. Thus, when the soln. becomes acid, Br_2 is liberated and extracted in the CCl_4. Whereas very little iodine is extracted since it is present as IO_3^-. (Hand shaking is better, rather than mechanical.) The CCl_4 phase is measured by neutron counters. Yield = 14–42%.		
Br^{90}	1.4 s	$U^{235}(n,f)$		Distillation	The previous method for Br^{88} is improved for the measurement of Br^{90}. The Br is swept out by air and is absorbed in a thin film of CCl_4 adhering to glass beads packed in a column. BF_3 counters are used in a suitable arrangement for counting fast neutrons emanating from the column.	2 sec	213
Br		U(n,f)	Uranyl nitrate	Extraction	To not more than 5 ml of a soln. of neutron-irradiated U-nitrate in a 50-ml centrifuge tube, 20 mg of bromine carrier as BrO_3^- and 10 mg of iodine carrier as IO_3^- are added. H_2S is passed into the soln. for 1 to 2 min. The soln. is boiled briefly to expel H_2S, cooled and transferred to a 60 ml separatory funnel. 1 ml of 6 $\underline{M}$ HNO_3 and a few drops of 1 $\underline{M}$ $KMnO_4$ are added and the Br extracted into 10 ml of CCl_4. The CCl_4 soln. is shaken with 10 ml H_2O containing 1 ml 1 $\underline{M}$ $NH_2OH \cdot HCl$ and the CCl_4 layer discarded. These extraction and back-extraction procedures are repeated. The CCl_4 in which the Br is extracted is shaken with 10 ml of H_2O containing a few drops of 1 $\underline{M}$ $NaHSO_3$ and discards the CCl_4. These procedures are repeated. In the aqueous phase, 1 ml of 6 $\underline{M}$ HNO_3 is added and the soln. is heated nearly to boiling and then the Br is pptd. as AgBr.	1 hr	106
Kr^{79m} Kr^{81m}	55 s 13 s	$Br^{79}(p,n)$ $Br^{81}(p,n)$	$PbBr_2$	Distillation	The target is coated onto Ni foil. The radioactive gas produced by proton bombardment is swept through a tube filled with glass wool at dry ice temp. (for removing Br activity) to the counting cell kept at liquid air temp. (for catching Kr-activity).		51

Kr^{89} Rb^{89}	2.6 m 15 m	$U^{235}(n,f)$	Uranium film	Recoil	A uranium film (0.3 mg/cm^2) deposited on a Pt disc (diameter: 1.6 cm) is used. The Kr activity recoiled from the target is collected in the recoil chamber and transferred into an evacuated cell. The Rb daughter is then collected on a negatively charged strip of Al.		202
Kr		$U^{235}(n,f)$	Uranyl nitrate (powder)	Distillation	The sample, in which uranyl nitrate powder layer and active charcoal layer are sandwiched between Kleenex paper pads in a polyethylene tubing, is irradiated for a short time (5-20 sec). After the irradiation, the radioactive gas is taken from the tubing with a syringe and a hypodermic needle in a manner such that the gas is pulled from the uranyl nitrate through the charcoal layer. The gas sample in the syringe is transferred to a second piece of tubing sealed under partial vacuum and then sent to the counter by a rapid transfer system.	20-30 sec	264
Rb^{84m}	20 m	$Rb^{85}(n,2n)$		Precipitation	The irradiated salt is dissolved in hot H_2O and the soln. boiled strongly for 2 min to remove Kr activities. Then the Rb is ppted. as the perchlorate.		82
Rb^{88}	17.7 m	$U^{235}(n,f)$	Uranium metal	Ion exchange	The irradiated metal (10 gm) is dissolved in a minimum amount of 6 $\underline{N}$ HNO_3 + 6 $\underline{N}$ HCl. Fe, Y, Ba, and Sr carriers (10 mg each element, as nitrate) and Cs and Rb carriers (each 1 mg) are added. The soln. volume is adjusted to 10 ml. Ag carrier (10 mg) is added and the AgCl ppt. is centrifuged off. By addition of NH_3 gas, Y, Fe, and U are ppted. and centrifuged off. The soln. is then scavenged with 10 mg Fe carrier and the $Fe(OH)_3$ ppt. is centrifuged off. $(NH_4)_2CO_3$ (5 ml saturated soln.) is then added and the Ba—Sr carbonate ppt. are centrifuged off. The whole soln. is evaporated to dryness. 5 ml aqua regia is added and the whole soln. is evaporated to dryness once again to destroy any residual NH_4-salt present. After addition of 5 ml conc. HCl, the soln. is evaporated to dryness. The dry residue is then dissolved in 2 ml 0.01 $\underline{M}$ NH_4Cl and ppted. on to a zirconium tungstate column (8 mm diameter, 3 mm long) and pressure (5 lb/sq in.) applied. The activity in the column is eluted with 1 $\underline{M}$ NH_4Cl soln. at a rate of not greater than 0.2 ml/min. The Rb fraction in the eluate is		52

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					evaporated to dryness with aqua regia and then ppted. as the Rb-chloroplatinate.		
Rb^{89}	15.4 m	Daughter of Kr^{89}(3.19m) from $U^{235}(n,f)Kr^{89}$	Saturated uranyl nitrate aqueous soln. (2.5 *l*)	Distillation Precipitation	The radioactive gas resulting from the uranium fission is removed from the soln. with a stream of air broken up into small bubbles by means of a sintered glass disc or a perforated tube. The decay products of the radioactive gases are collected on a platinum wire by an electrical field. (1000 volts negative with respect to the vessel) The Pt wire is then immersed in hot water. A soln. saturated in sodium bitartrate and containing RbCl is added. The rubidium bitartrate is ppted. by cooling the mixture in ice. This kind of pptn. is repeated five times. The fifth ppt. is dissolved in hot water to which is added inactive Ba and Sr chlorides. After two successive carbonate or sulfate pptns., the final filtrate is evaporated to dryness for the measurement of Rb activity.		102
Sr^{89}	50.5 d	$U^{235}(n,f)$	Aged fission products	Extraction	For the rapid separation of the radioactive Sr, a two step extraction is made. At first, the soln. of the gross fission product is treated with 0.05 M TTA-benzene at pH 7 to separate the rare earth activity. Then the remaining aqueous phase is shaken with 0.05 M TTA-hexanone at pH 8 to extract the Sr activity.		142
				Ion exchange	An aliquot of fission product soln. containing known amounts of Cs and Sr carrier is passed down a column of Amberite IRA-400 (OH form) and eluted with several column volumes of H_2O. The eluates are heated almost to boiling, and saturated ammonium oxalate then added to ppt. Sr. The Sr ppt. is centrifuged off and washed several times with H_2O, dried, weighed, and counted. (Ba^{140} should be a contaminant; but, if the fission product is at least several months old, the amount of Ba^{140} is negligible.)		55
Y^{89m}	14 s	Daughter of Zr^{89}(79 h)	Yttrium oxide	Extraction	Zr^{89} activity is separated from the yttrium target by repeated extraction of the TTA complex.		109

		from $Y^{89}(d,2n)Zr^{89}$			The Y^{89m} daughter is re-extracted into 2 M $HClO_4$ and measured.		
Y^{90}	64 h	Daughter of Sr^{90} (28 y) from $U^{235}(n,f)Sr^{90}$		Extraction	The Sr-carbonate ppt. (Sr is about 4 mg) is dissolved in 5 ml of 0.5 N HNO_3. The soln. is transferred to a separatory funnel which contains 10 ml TTA (10 gm per 100 ml benzene) washed previously with 5 ml 0.5 N HNO_3. 10 ml buffer soln. (0.5 M NaOAc, 0.1 M HOAc) is added to the soln. and the extraction made for 10 min. The extract is washed twice with 10 ml buffer soln., the Y^{90} is back-extracted into 10 ml 0.1 N HNO_3 from the benzene phase and the resultant solution evaporated to dryness.		232
				Extraction	To 1 ml of sample soln., 5 ml buffer soln. (0.5 M soln. consisting of HOAc, NH_4OAc, and HN_4OH) of pH 4 and 1 ml 5% cupferron aqueous soln. are added. The Y in the soln. is extracted into 2 ml $CHCl_3$ and washed with the above buffer soln. The Y in the $CHCl_3$ can be back-extracted with acidic aqueous soln. of pH 1.		141
				Extraction	The sample is dissolved in 2-5 ml of 0.1 M HNO_3 (HCl, $HClO_4$) and shaken with an equal volume of 0.1 M DBP in alcohol free $CHCl_3$. The two phases are withdrawn and the $CHCl_3$ phase is evaporated to dryness. 80% of the DBP in the $CHCl_3$ sample may be removed at 190°C. The Y^{90} in the CCl_4 phase can also be back-extracted into an equal volume of 5 M HNO_3. Yield = ~ 98%.		72
Y^{94}	16.5 m	Zr(d,α)	Zirconium metal foil	Precipitation	The foil is dissolved in dilute HF. Nb and Y carriers are added and the Y ppted. as the fluoride. This fluoride is metathesized to the hydroxide and then dissolved in acid. The resulting soln. is scavenged with zirconium phosphate and niobic acid ppts. to complete the separation. A final pptn. of YF_3 is slurried onto a Cu plate for counting.	25 m	221
Y^{94}	16.5 m	U(n,f)	Uranyl nitrate aqueous soln.	Precipitation	25 ml of saturated uranyl nitrate was irradiated for 20 min. A 2 ml aliquot of the soln. is diluted to about 7 ml by the addition of 20 mg of carrier Y^{+3} and 10 mg each of carriers La^{+3}, Ce^{+3}, and Zr as ZrO^{++}. The	65 m	65

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					soln. is then slurried with 1.5 g of $BaCO_3$ and centrifuged. This is repeated twice more to remove the uranium completely. The residual Ba^{++} is ppted. with dil. H_2SO_4, then the hydroxides are brought down with NH_4OH. The ppt. is dissolved in a minimum of dil. HCl and an excess of sat. K_2SO_4 is added. The soln. is heated to ppt. the K-La-sulfate. It is found that Ce and Zr are also removed at this point. More La carrier is added, and the sulfate pptn. is repeated. The Y, which remained in soln., is ppted. with oxalic acid and filtered through a 1-cm filter paper. The ppt. is washed with H_2O, alcohol, and ether, mounted on cardboard in the usual manner, and counted.		
Y		Sr(p)	Strontium carbonate	Precipitation	The $SrCO_3$ is dissolved in YCl_3 soln. containing H^+ to dissolve the carbonate (1/3 mg/ml of YCl_3 and H^+ = 0.025 N). To the soln., 25 ml of an oxine soln., made by dissolving 0.17 mg oxine in 12.5 ml acetone and diluting to 50 ml with H_2O, is added. Conc. NH_4OH is also added until the smell of NH_3 can be detected above the soln. After 10 min digestion on a water bath, the Y-oxinate ppt. is centrifuged. Yield = 100%.	20 min	35
Zr^{89m}	4.5 m	$Y^{89}(p,n)$		Precipitation	The Zr activity is separated from the bombarded yttrium as the iodate in HNO_3 soln., with a trace of zirconium nitrate added as carrier. The ppt. is filtered and measured.		68
Zr^{95}	65 d	$U^{235}(n,f)$	Fission product	Extraction	A suitable aliquot of the fission product soln. is adjusted to 2 M in HNO_3 and approx. 0.5 M in hydroxylamine hydrochloride. The soln. is then extracted for 10 min with an equal volume of 0.5 M TTA-xylene. After the extraction, the organic phase is washed for 3 min with an equal volume of 1 M HNO_3 soln., transferred into a suitable centrifuge cone, and centrifuged for 3 min in a clinical centrifuge. Recovery of Zr^{95} = 99 ± 2.4%. The Zr-95 radioactivity is then determined by counting a suitable aliquot of the organic phase.	20 min	181

				This method is rapid and can be performed in about one fourth of the time required for the barium fluozirconate.	
Zr	$U^{235}(n,f)$	Uranyl nitrate	Precipitation	The irradiated sample is dissolved in H_2O, and HNO_3 added to make about 5 ml of 6 <u>M</u> HNO_3 soln. 2 ml conc. HF and 20 mg Zr carrier are then added. 10 mg of La carrier soln. is added as a scavenge to remove rare earth and alkaline earths. The ppt. found is centrifuged off and discarded. This scavenging process is repeated. 1 ml of $Ba(NO_3)_2$ carrier soln. (50 mg Ba) is then added to the soln. After 1 min standing, the $BaZrF_4$ ppt. is centrifuged. It is then dissolved in 5 ml H_2O and 2 ml 5% H_3BO_3 and 1 ml conc. HNO_3. 1 ml $Ba(NO_3)_2$ soln. (50 mg Ba) and 1 ml conc. HF are added, the ppt. is formed and then centrifuged. This $BaZrF_4$ ppt. is dissolved with 5 ml H_2O, 2 ml H_3BO_3 and 3 ml conc. HCl. H_2SO_4 (1 drop) is added and the $BaSO_4$ ppt. is centrifuged off. To the supernatant soln., 2 ml 6% cupferron soln. is added and the Zr-cupferrate is ppted.	123
Zr	Nb(p)	Niobium foil	Extraction	The Nb target foil is dissolved in a mixture of conc. HNO_3 and conc. HF. 10 mg of La-nitrate is added and ppted. as LaF_3 which co-ppts. Zr activity and separates Zr from Nb. The LaF_3 ppt. is contacted with conc. KOH to metathesize the fluoride to the hydroxide compound. Then the hydroxide is dissolved in 2 <u>M</u> $HClO_4$. Any Nb, which might remain, is scavenged by MnO_2 which is ppted. from the soln. by adding Mn^{++} and $KMnO_4$. The Zr is then extracted from the $HClO_4$ soln. by contacting it intimately for 15 min with an equal volume of a 0.2 M soln. of TTA in benzene. $Al(NO_3)_3$ is added to the aqueous phase to complex any traces of F ion which might remain from the metathesis step, since the F ion interferes with the TTA extraction. The Zr-TTA complex in benzene soln. is washed by 2 <u>M</u> $HClO_4$ soln. The benzene soln. is diluted 10-fold and contacted with one tenth its volume of conc. HCl to effect the return of the Zr to conc. aqueous medium.	128

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Nb^{90m}	24 s	Daughter of Mo^{90}(5.7 h)	Carrier-free Mo^{90} sample	Recoil	An intense carrier-free Mo^{90} sample is placed on the shelf of the scintillation counter for a 1 min growth period. The Al front cover on the NaI crystal is not grounded, and carries a potential of —500 volts with respect to the sample. The sample is then quickly withdrawn and the recoil daughter atoms of Nb^{90m} collected from the sample are counted.		171
				Ion exchange	The Mo^{90} activity in 6 N HCl soln. is run through a column (4 cm x 0.5 cm) of Dowex-1 anion exchange resin previously equilibrated with 12 N HCl. The Mo on the column is washed with 5 N HCl. After a growth period of 1 min, the Nb activity formed during this time is quickly stripped from the column by elution with 5 N HCl and measured by the scintillation counter.		171
Nb^{94m}	6.6 m	$Nb^{93}(n,\gamma)$	Rock (rutile)	Precipitation	The 100 mg irradiated sample contained in a gelatin capsule is fused with 3-4 gm Na_2O_2 containing known amounts of Nb^{95} tracer. The melt is dissolved in a solution of 25 ml H_2O, 20 ml conc. HNO_3, and 3 ml T_2^{1+4} carrier. The soln. is filtered to remove any undissolved material. 25 ml of conc. HNO_3 is added to the filtrate, followed by addition of 0.5 gm SiO_2 gel, and the mixture is boiled for 1 min. The soln. is filtered through a medium stainless steel filter funnel. The SiO_2 gel on which Nb^{94m} is absorbed is dried with acetone and counted. Yield = 30-35%.	10 min	26
Nb		$U^{235}(n,f)$	Uranyl nitrate, aqueous soln.	Extraction	10 mg Zr carrier is added to the sample soln. (1-10 ml) and an equal volume of conc. HNO_3 is added. 1 ml saturated oxalic acid and 2 ml Nb carrier soln. are also added and the solution is stirred gently for 10 min. The soln. is then heated nearly to boiling and 0.5 g $KBrO_3$ in small portions is added, boiling for a few seconds after each addition. The soln. is evaporated to small bulk and the niobic acid ppt. formed is centrifuged and washed with 10 ml hot 2% NH_4NO_3 solution.	1.5 hr	184

					The ppt. is dissolved in 2.5 ml 40% HF in a polyethylene tube and 0.2 ml 12 M H_2SO_4 and 4-5 ml TBP are added. The Nb is extracted into the TBP phase by stirring for 2 min and centrifuged. To the separated TBP phase, 5 ml petroleum ether and then 4 ml 6 N NH_4OH are added. After stirring for 1 min and centrifuging at high speed for 1 min, the TBP phase is discarded. After washing the aqueous phase with 5 ml petroleum ether and centrifuging it, the organic and aqueous supernate are discarded. The niobic acid ppt. is washed with 10 ml hot 2% NH_4NO_3 soln., ignited and counted as Nb_2O_5. Yield = ~ 60%.	
Mo^{91m}	75 s	$Mo^{92}(\gamma,n)$		Precipitation	The Nb activity is carried down by a $Fe(OH)_3$ ppt. from alkaline $(NH_4)_2MoO_4$ soln. and then the Mo is ppted. as ammonium phosphomolybdate from acidic soln.	69
Mo^{99}	67 h	$U^{235}(n,f)$	Fission product	Precipitation	10 mg of Mo(V) are added to the sample, the volume adjusted to 30 ml, and the acidity to approx. 1 M. 5 ml of 2% α-benzoin oxime in ethanol are added, the mixture stirred, and centrifuged. The ppt. is washed with 30 ml of H_2O. The ppt. is dissolved in 3 ml fuming HNO_3, the soln. diluted to 25 ml with H_2O, partially neutralized with 1-2 ml of NH_4OH, and cooled in an ice bath. The Mo is re-ppted. by adding 5 ml of the oxime reagent. The mixture is stirred and centrifuged. The dissolution and pptn. procedures are repeated. The final ppt. is dissolved in 3 ml of fuming HNO_3. 3 ml $HClO_3$ is added and the soln. boiled down (CAUTION) until the $HClO_3$ starts fuming and 30 sec longer. Then the mixture is cooled. 10 ml of H_2O and 1 ml of Fe^{+++} are added; then NH_4OH or NaOH is added until the molybdic oxide dissolves and $Fe(OH)_3$ has ppted. The mixture is filtered through any fast filter paper such as Whatman 4 or 41. The Mo is then ppted. as $PbMoO_4$ in the usual manner. Yield = 70-75%.	218
Tc^{92}	4.3 m	$Mo^{92}(d,2n)$	Enriched Mo^{92}	Distillation	The Tc activity is separated from the bombarded Mo isotopes by means of a volatility separation method and then the Tc is prepared as a thin sample for counting by co-pptn. with platinum sulfide.	187

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
				Extraction	The extraction method using methyl-ethyl-ketone can be applied.		76
Tc^{101}	14.0 m	Daughter of Mo^{101}(14.6m)	Biological tissue	Extraction	The irradiated sample is dissolved in 3 ml hot 12 N NaOH. The soln. is acidified with 10 ml HCl. 5 mg Re carrier and 5 ml saturated Br_2 is added to the soln. After boiling to expel Br_2, 7 gm $NaHCO_3$ is added and then the soln. is diluted to 30 ml with H_2O and adjusted to pH 8 by a few drops of 4 N NaOH. Tc is extracted together with Re carrier into 20 ml $CHCl_3$ by addition of 2 ml 1% tetraphenylarsonium chloride soln. After extraction, 20 ml 1 N HCl is added to the organic layer and $CHCl_3$ is boiled off from the soln. After oxidation of Re by 2 ml Br_2-water and boiling off the Br_2, Tc is co-ppted. with Re by 2 ml tetraphenylarsonium chloride soln. while hot. The soln. is cooled with liquid N_2 and the ppt. is filtered and counted. Yield = 60%.	30 min (including waiting period of ~ 15 min)	262, 91
Tc^{102}	5 s	Daughter of Mo^{102}(12m)		Precipitation	The Mo parent activity is purified as a lead molybdate ppt. The ppt. is dissolved in a mixture of HCl and tartaric acid in the presence of ReO_4^- carrier. (The tartaric acid forms a complex with Mo and inhibits its pptn. by tetraphenylarsonium chloride.) The soln. is transferred to a Büchner funnel fitted with a membrane filter and a detachable top. The filter is placed directly beneath a GM tube. Tetraphenylarsonium chloride soln. is added, and the suction is turned on. As soon as the soln. has drained, the sample is counted.	6 sec	85
Tc		U^{235}(n,f)	UO_3	Extraction	The sample is dissolved in H_2SO_4 containing a few μg of I^- hold-back carrier. After addition of $S_2O_8^{=}$, I_2 is extracted into toluene. The aqueous phase is heated to boiling to remove traces of iodine and neutralized with $(NH_4)_2CO_3$. More $S_2O_8^{=}$ is added and the soln. heated to boiling. TcO_4^- is extracted into $CHCl_3$ by addition of tetraphenylarsonium chloride soln., and back-extracted into $HClO_4$ or H_2SO_4.		262

Tc		U^{235}(n,f)	Uranyl nitrate soln.	Distillation	To 5 ml of an active soln., 10 ml of conc. HBr is added and evaporated almost to dryness. The evaporation with HBr is repeated twice. The residue is transferred to a distillation flask with a minimum amount of H_2O, and 10 ml of H_2SO_4 is added. The soln. is heated gently to boiling and distilled into a centrifuge tube containing 20 ml of H_2O until less than 2 ml of H_2SO_4 remains in the flask. 10 mg of Re carrier is added to the distillate, and Re_2S_7 is ppted. by H_2S. The sulfide ppt. in which Tc is co-ppted. is dissolved in 1 ml of conc. HNO_3 and evaporated to dryness with 5 ml of conc. HBr. The evaporation first with 5 ml of HNO_3 and then with 5 ml of HCl is repeated. The residue is diluted to 20 ml with H_2O. After scavenging with $Fe(OH)_3$, in which about 2 mg of Fe is used, Tc is co-ppted. with a tetraphenylarsonium perrhenate ppt. by addition of 1 ml of 0.05 M $(C_6H_5)_4AsCl$. Yield = ~ 95%.		104
Tc				Precipitation	Separation of Tc and Mo: Mo is ppted. with oxine from neutral soln., filtered and made to 0.6 N HCl soln. (50 ml). After addition of 0.3-2.0 mg Cu^{++} carrier and heating, H_2S is passed into the soln. for 15 min. The CuS on which Tc is co-ppted. is filtered and washed with H_2S water.	40 min	130
				Precipitation	Separation of Tc and Re: The sample is dissolved in a small volume of conc. HCl and heated to about 75°C for 30 min ($Tc^{+7} \to Tc^{+4}$). After addition of Fe^{+++} ion, the soln. is diluted and made ammoniacal to ppt. $Fe(OH)_3$. Tc^{+4} is co-ppted. on $Fe(OH)_3$. The ppt. is filtered, washed with a hydrazine sulfate soln., and dissolved in a small amount of conc. HNO_3. ($Tc^{+4} \to Tc^{+7}$) The Fe^{+++} ion is removed by pptn. with ammonia.		95
Ru^{93}	50 s	Mo(α)	Molybdenum metal	Distillation	The Mo is dissolved in a mixture of HNO_3 and HF. The Ru activity is distilled off after the addition of H_2SO_4 and $KMnO_4$.		10

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Ru^{108}	4 m	U^{235}(n,f)	Uranyl nitrate	Distillation	The Ru activity in the fission product is isolated by distillation with $NaBiO_3$ in acid soln. The Ru in the distillate is absorbed in 6 N NaOH soln. and ppted. by boiling with alcohol. The black oxide is centrifuged into the bottom of a lusteroid tube and removed for mounting and counting by cutting off the bottom of the tube.	4 min	103
Rh^{98}	9 m	Ru(d)	Ruthenium metal	Precipitation	The Ru is distilled by heating the soln. with H_2SO_4 and bromate. The Rh in the remaining soln. is ppted. as Rh([illegible]th alkali, followed by several extractions of tet[illegible] arsonium perrhenate (261) to remove Tc activity. Finally, Rh is ppted. as $K_3Rh(NO_2)_6$. According to this procedure, the Rh was not quite free from Tc, but this contamination did not interfere with the decay measurement of Rh^{98}.		7
		Daughter of Pd^{98}(17.5m)		Precipitation	The purified Pd-dimethylglyoxime containing Pd^{98} is allowed to stand an appropriate growth time. After it is dissolved, Rh(IV) carrier is added and the Pd is removed by a series of pptns. with cupferron. Finally, the Rh is ppted. as $K_3Rh(NO_2)_6$.		138
				Ion exchange	The purified Pd-dimethylglyoxime ppt. containing Pd^{98} is dissolved in a few ml of 3 N HCl. This soln. is then poured in at the top of a Dowex-1 anion exchange column (7 mm in diameter and 4 cm long). The Rh^{+3} activity is eluted quantitatively from the column with 4 ml of 6 N HCl while 60 ml of 6 N HCl moves the Pd(II) down the column only 2-3 cm.	2 min for the elution of Rh^{+3} on ion exchange resin	138
Rh^{104m}	4.4 m	$Rh^{103}(n,\gamma)$	Meteorite	Precipitation	1 g of irradiated sample is fused with 8 g Na_2O_2. The melt is dissolved in a soln. containing 100 ml H_2O, 25 ml HCl, 2 ml Rh carrier and 2 ml Fe carrier. After slow addition of sat. sodium nitrite soln. to ppt. $Fe(OH)_3$, the soln. is filtered. 5 g each of KCl and KNO_2 are added to the soln. and the ppt. produced is filtered and dissolved with hot 0.5 N HCl. Metallic zinc is added to this soln. and filtered. Yield = 50-70%.	9 min	230

			Rock	Extraction	The irradiated sample is fused with 5 g Na_2O_2. The melt is dissolved with 20 ml conc. HCl and 5 ml $RhCl_3$ carrier (1 mg/ml in 1 N HCl). 1 ml 10% tartaric acid and 8 ml pyridine are added. After filtration to separate silica, 15 ml of 12 N NaOH is added and Rh is extracted into the pyridine layer. Yield = 95-100%.	7 min	237
Rh		U^{238}(n,f)	Uranyl nitrate aqueous soln.	Precipitation	10 or 20 mg of Rh carrier is added to the irradiated sample. The acidity is adjusted by neutralizing with 6 N NH_4OH and acidifying by adding 2 to 3 drops of 6 N HCl until all the ppt. dissolves. Then $K_3Rh(NO_2)_6$ is ppted. by addition of 10 ml of 60% KNO_2 to the soln. and centrifuged. After washing the ppt. with 2% acid washing soln., the ppt. is dissolved in 1 ml of aqua regia and diluted to 10 ml. Scavenging pptns. of zirconium arsenate and silver chloride are carried out by adding 10 mg each of Zr and Ag carriers and then adding 3 ml of phenylarsonic acid in a little 6 N HCl. The zirconium arsonate scavenging is repeated. Then $K_3Rh(NO_2)_6$ is ppted. and counted.		225
Pd^{98}	18 m	Ru(α)	Ruthenium metal	Precipitation	The normal method of isolation consists of two scavenging pptns. of $Fe(OH)_3$ followed by one or two Pd pptns. with dimethylglyoxime and one with furfuraldoxime. As hold-back carriers, Cu^{++}, Fe^{+++}, Ni^{++}, Co^{++}, Zn^{++}, and Mn^{++} are present.		9
Pd^{98} Pd^{99}	17.5 m 21.6 m	Ru(α)	Ruthenium metal or chloride	Precipitation	Shortly after each irradiation, the target is dissolved in dil. HCl. For the Ru metal targets, a preliminary fusion in a mixture of NaOH and Na_2O_2 is necessary. Pd^{+2} carrier is added, and then Pd dimethylglyoxime is ppted. This ppt. is dissolved in hot HNO_3, and then several successive $Fe(OH)_3$ and AgCl scavenging pptns. are made. The Pd is ppted. again as the Pd dimethylglyoxime.		138
Ag^{106}	24.0 m	Pd^{105}(d,n)	Palladium metal	Precipitation	After the bombardment, the Pd targets are dissolved in HNO_3 and a small quantity of solns. of the neighboring elements (Ag, Rd, and Ru) are added. The Ag is ppted. from HNO_3 soln. as the chloride, then dissolved in NH_4OH and re-ppted.		149, 214

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Ag^{108}	2.3 m	$Ag^{107}(n,\gamma)$	Meteorite	Precipitation	The 1 g irradiated sample is fused with 8 g Na_2O_2 (containing known amounts of Ag carrier). The melt is dissolved in 100 ml H_2O and 25 ml HCl. The AgCl produced is filtered and dissolved in hot NH_4OH. Ag is reduced by Zn and ppted. from the ammoniacal soln. Yield = 80%.	5 min	231
			Rock	Precipitation	The 0.5 g irradiated sample is fused with 5 g Na_2O_2 containing 40 mg Ag and 50 mg Mn carrier. The melt is dissolved in 100 ml of 4 <u>N</u> HNO_3 containing 5 ml of 30% H_2O_2. The soln. is filtered through a $PbCl_2$ ppt. layer (Pb: 1.5 gm). After the $PbCl_2$ layer is washed with hot 4 <u>N</u> HNO_3 and then with acetone, the AgCl remaining on the filter paper is counted. Yield = ~ 60%.	5 min	151
Ag^{109m} Ag^{111m}	39 s 74 s	Daughter of Pb^{109}(13.6h) from $Pd^{108}(d,p)$ Daughter of Pd^{111}(22m) from $Pd^{110}(d,p)$	Palladium foil	Ion exchange	A Pd foil (50 mg) is bombarded and allowed to decay for 3 hr. The Pd is separated from Ag and Rh activities by standard procedures (pptn. of Pd with dimethylglyoxime in 1 <u>M</u> HCl and then with H_2S in 6 <u>M</u> HCl). The Pd activity, mainly 13.3 hr Pd^{109}, is absorbed from 10 <u>M</u> HCl on the top of the anion exchange column (1/4 in. diameter, 2 in. long, filled with Dowex-1 100-200 mesh, 10% cross-link). Short-lived Ag^{109m} in equilibrium with the Pd parent can be quickly eluted with 5-10 ml of 10 <u>M</u> HCl. The eluent is forced by compressed air through the column, is collected on a Cu planchet, and evaporated to dryness for counting. Prior to the ion exchange separation, the oxidation of $PdCl_4^{-2}$ to $PdCl_6^{-2}$ with chlorine gas gives better results for the separation of Pd activity.	< 60 sec (The time between elution and beginning of counting)	219
Ag^{110}	24.2 s	Daughter of Ag^{110m}(253d)		Recoil	The separation of Ag^{110} from Ag^{110m} obtained from Oak Ridge is studied. The separation is affected by collection of activity on Cu electrodes immersed in an ether soln. of silver (Ag^{110m}) tetraphenyl-porphin. Two Cu electrodes separated by about 3 mm are immersed in the soln. and a potential difference of 600 volts		178

					is applied. After about 3 min, one of the electrodes is removed, washed with ether, and then counted. Separation occurs at both the anode and cathode with about the same efficiency although the background is generally lower in the anode. Separation is also obtained when a Cu electrode is immersed in the soln. with no potential applied, but the separation is not as clean. No separation is observed when an uncharged Pt electrode is introduced.		
Ag^{114}	3 m	Cd(γ) Cd^{114}(n,p)	Cadmium nitrate	Precipitation	The Ag activity is ppted. as AgCl, dissolved and reppted. in the presence of Cd^{++} hold-back carrier.		70
Cd				Extraction	1 ml of sodium tartrate soln. (20%) is added to the sample soln. and the pH of the soln. adjusted to 13.5 by dil. NaOH. Cd is extracted by 10 ml dithizone soln. (0.75 mg/ml $CHCl_3$). Cd is back-extracted into 10 ml of 0.1 N HCl soln. from the $CHCl_3$ soln. After adjustment of HCl concentration to 3 N, the soln. is passed through an ion exchange column (Dowex II, 300 mesh), which has been equilibrated with 3 M HCl. After washing the column with 10 ml of 3 N HCl, Cd is eluted by 0.1 N NH_4OH. (The first 0.5 ml is discarded and the next 3 ml of eluent is collected.) Yield = 62%.	25 min	59
				Extraction	0.2 gm solid sodium tartrate is added to the sample soln. The soln. is adjusted to pH 13-13.5 with 0.1 N NaOH and diluted with H_2O to 10 ml. The Cd is extracted with 10 ml dithizone-$CHCl_3$ soln. (0.75 mg/ml) and back-extracted into 10 ml 0.1 N HCl (shaking for 2 min in each case). These procedures are repeated, depending upon the desired decontamination factor. Yield = ~ 77%.	~ 10 min	61
Cd		U^{235}(n,f)	Uranyl nitrate	Isotopic exchange (Amalgam)	100 mg sample is irradiated for 10 min and dissolved in 1 ml H_2O (< 0.1 n HNO_3 acidic). ~ 25 mg of Cu pellets are added to the soln., the soln. shaken for 1 min and filtered. To the filtrate is added 1.5 ml chromous sulfate soln. (~ 100 mg chromium sulfate per ml in 0.1 N H_2SO_4 is reduced by stirring with 5% Zn amalgam), to reduce U^{+6} to U^{+4}. 50 λ (~ 0.68 gm) Cd amalgam (2% Cd by weight) is then added. After stirring vigorously for 5 min, the supernate is removed by suction and the amalgam washed twice with	~ 15 min	63

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					2 ml portions of 0.1 N HNO_3 and twice with 2 ml portions of distilled H_2O. Then the amalgam is transferred to a stoppered bottle containing 2 ml thallous acetate soln. (75 mg Tl^+ per ml of 0.1 N HNO_3) and shaken for 1 min. Radioactive cadmium is now in the aqueous phase and can be counted.		
In^{119}	17.5 m	$Sn^{120}(\gamma,p)$	Tin foil (95.4% enriched Sn^{120})	Precipitation	After dissolution of the foil, In is ppted. as $In(OH)_3$ from hot 2 N NaOH.		71
In		Cd(d) Cd(p)	Cadmium metal	Precipitation	The sample (a few mg) is dissolved in a few drops of hot conc. HNO_3. Ag and In carriers (each 2 mg) are added to the soln. and the volume of the soln. adjusted to 5 ml. By addition of NH_4OH, the soln. is made slightly basic (methyl orange → yellow). The $In(OH)_3$ produced is filtered, washed with a small amount of 1% NH_4NO_3 soln., and then dissolved in HNO_3. The pptn. and dissolution procedures for $In(OH)_3$ are repeated. The final $In(OH)_3$ ppt. is washed by 1-2 ml acetone to remove the residual H_2O. Yield = 50-75%.	10 min	158
In				Extraction	(1) The In is extracted as the bromide into dimethyl ether from an aqueous soln. which has been adjusted to be 4.5 M in HBr. After washing with 4.5 M HBr twice, the In is back-extracted into 6 N HCl. The HCl is evaporated to ~ 1 ml and the pH adjusted to 2-3 by addition of NaOH. Potassium acid phthalate buffer (pH 2.4) is then added to bring the soln. volume to 20 ml. In is extracted into 20 ml of 0.5 M TTA in benzene and counted. Yield = 70-80%.		252
					(2) The irradiated sample is dissolved in about 10 ml of conc. HBr. 10 mg of In as a soln. of the bromide and several mg of Fe as the nitrate are added, and the soln. is evaporated just to dryness. The residue is then taken up in 1 M HBr, washed into a separatory funnel, and		121

					made up to ~ 8 ml with 1 M HBr. Two successive 30 ml portions of isopropyl ether are equilibrated with the acid phase and discarded. Enough concentrated HBr (48%) is added to make the HBr concentration 4.5 M. The total volume is about 20 ml. A fresh 30 ml portion of isopropyl ether is added and shaken with an aqueous soln. for one min, and the acid phase separated. This step is repeated and after the second equilibration with ether the acid phase is discarded. The two ether phases are combined and washed with three successive 5 ml portions of 4.5 M HBr. The washings are discarded. The ether phase is then equilibrated with three successive 5-ml portions of 5 M HCl. The aqueous phases are combined and washed with another fresh portion of isopropyl ether. The HCl soln. is then diluted to 50 ml, 5 gm of NH_4NO_3 added, and the soln. just neutralized with NH_4OH. The mixture is centrifuged, and the ppt. is washed with H_2O and dried.		
In				Isotopic exchange (Amalgam)	2 ml sample soln. (0.1 N HBr) containing μg amounts of In carrier are purged with N_2 gas for 1 min. 75 λ (~ 1.0 gm) In amalgam (0.2% In by weight) is added and shaken for 4 min in a stoppered bottle. The aqueous layer is decanted. The amalgam is transferred to a new bottle, washed with a 2 ml portion of H_2O, and then transferred to another new bottle. The radioactive In is back-exchanged into the aqueous phase by contact with a higher concentration of In ion. Cobaltous ion can also be used for this back-exchange process.	~ 11 min	217
Sn^{125}	9.5 m	$Sn^{124}(n,\gamma)$ $Sn^{124}(d,p)$	SnO_2 Tin metal	Precipitation	Chemical separations are made by pptn. and by fractional distillation. Sb is ppted. as Sb_2S_5, In as $In(OH)_3$, and Sn is ppted. by phenylarsonic acid.		154
Sn^{130} Sn^{131} Sn^{132}	2.6 m 3.4 m 2.2 m	$U^{235}(n,f)$	Pyridine hexachloro-uranate (solid)	Extraction	The irradiated sample is dissolved in 10 ml aqueous NH_4OAc (10%) soln. to which ~ 0.5 mg Sn^{++} carrier has been added. 10 ml of 10% K-Na-tartrate is added to this soln. and the soln. is adjusted to pH 8.5 with 2-4 ml 4 M NH_4OH. Sn is extracted by shaking for 10-15 sec with 10 ml	45 sec	207

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					0.01% dithizone-CCl_4 soln., and the organic phase washed once with 5% NH_4OAc—5% Na tartrate (pH = 8.5) soln. With this procedure, Pd, Cd, In, Pb, Bi, and Po are also extracted under these conditions, but the former three elements have much lower fission yields than the Sn isotopes and the latter three elements do not occur as products of low energy fission, so these contaminants do not interfere in this method.		
Sn		$U^{235}(n,f)$	UO_2Cl_2 aqueous soln.	Distillation	An aliquot of the sample is added to 10 mg Sn^{+2}, 10 ml conc. H_2SO_4 and 1 ml H_3PO_4, and evaporated to fumes of SO_3 over a burner. The soln. is then introduced into a distilling apparatus and the temp. raised to 220°C. 10 ml 48% HBr is added dropwise and $SnBr_4$ is distilled into 10 ml conc. HCl in a 50 ml centrifuge tube. A current of air carries the $SnBr_4$. The distillate is evaporated to 5 ml to insure complete removal of Br_2. The Sn is ppted. by addition of 2 ml 5% cupferron and extracted into 15 ml ethyl acetate. The organic phase is washed with 10 ml portions of 1 N HCl. The Sn is back-extracted into 10 ml saturated oxalic acid soln. The oxalic acid soln. is diluted to 25 ml, and heated to boiling. The Sn is ppted. by addition of 5 ml sat. phenylarsonic acid soln. and washed with 4% NH_4NO_3, alcohol, and ether. Yield = 60%.	20 min	269
Sb^{122m} Sb^{124m-2} Sb^{124m-1}	3.5 m 21 m 1.3 m	$Sb^{121}(n,\gamma)$ $Sb^{123}(n,\gamma)$ $Sb^{123}(n,\gamma)$	Sb_2O_3, antimoney metal	Precipitation	The bombarded Sb is dissolved in HCl and the Sb is plated out on Fe. Sn is added as a carrier and is plated out on Zn after the Sb is removed.		57
Sb^{126} Sb^{128}	19 m 10.3 m	$Te(d,\alpha)$ $Te(n,p)$	Elemental tellurium, Na- or K-tellurite	Precipitation	The irradiated tellurite is dissolved in HCl and Sb^{+3} and Sb^{+5} carriers added. The antimony activity is distilled as SbH_3 and caught in $AgNO_3$ soln. In the case of an elemental Te target, the large amount of Te is reduced by hydrazine hydrochloride. Sb is then ppted. as Sb_2S_3 and dissolved in HCl. Finally, Sb is distilled as SbH_3.		88, 87
Sb^{128}	10.3 m	Daughter of $Sn^{128}(57m)$		Precipitation	The Sn activity in fission products is purified radiochemically and dissolved in HCl as the fluoride complex.		87

					Sb is reduced to Sb(III) from (V) by hydrazine chloride in hot soln. By this method, the weak fluoride complex of Sb(V) is destroyed, whereas the tin(IV) complex remains. In the presence of hydrazine chloride, Sb is ppted. as Sb_2S_3, which separates it rapidly from Sn activity. The Sb_2S_3 ppt. is dissolved in HCl. SbH_3 is produced by addition of Zn and is caught by a $AgNO_3$ soln.		
Sb^{127} Sb^{129}	94 h 4.2 h	$U^{233}(n,f)$ $U^{235}(n,f)$ $Pu^{239}(n,f)$		Distillation	The sample (free of nitrate) is transferred to a distillation flask in which 4 ml Sb^{+5} carrier (10 mg/ml) and 4 ml 6 N H_2SO_4 are contained. After addition of a drop of Br_2, the soln. is boiled. Then 5 gm granulated Zn is added and the SbH_3 generated caught in 20 ml 6 N HCl containing a few drops of liquid Br_2. 10 mg Te and 1 gm hydrazine hydrochloride are added to the soln. H_2S is passed through the soln. during heating to ppt. Te and centrifuged off. After addition of 5 ml conc. NH_4OH and dillution to 30 ml, Sb_2S_3 is ppted. by passing in H_2S gas. The ppt. is centrifuged and washed, and then dissolved in 2 ml conc. HCl. After addition of 0.5 gm $NaHSO_3$ dilution to 10 ml and boiling, Sb is ppted. as the oxinate by addition of 3 ml oxine soln. (3% in 2 N HCl), 1 ml conc. NH_4OH, and 3 M NH_4OAc (at pH = 5). The ppt. is washed with H_2O, alcohol, and ether. Yield = ~ 25%.	< 40 min	86
Sb^{130} Sb^{131}	10 m 23.1 m	Daughters of Sn^{130}, Sn^{131}, and Sn^{132}, from $U^{235}(n,f)$		Extraction	The Sb daughter activity is back-extracted into 5% NH_4OAc—5% K-Na-tartrate aqueous soln. (pH 8.5) from the dithizone-CCl_4 soln. into which the tin parent activity has been extracted.	30 sec	207
Sb^{131} Sb^{132} Sb^{133} $Sb^{134,135}$	23.1 m 2 m 4.4 m 50 s	$U^{235}(n,f)$	Uranium oxide	Extraction	The fast chemical separation of Sb is accomplished by solvent extraction. The Sb is extracted as $SbCl_5$ into isopropyl ether in the presence of Cl_2, and back-extracted as $SbCl_3$ into the aqueous phase containing N_2H_4. After addition of NaSCN, any Sn contaminant is extracted into the ether phase.		205

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
				Distillation	A 10 gm sample of uranium oxide irradiated with neutrons is dissolved in 4 N H_2SO_4. The soln. is introduced into a H_2 generator (employing Zn and 4 N H_2SO_4). Under these conditions, SbH_3 is formed, which is carried along in the stream of H_2. The SbH_3, on coming in contact with $AgNO_3$, forms a black ppt. of silver antimonide. The only element which reacts similarly is Ge.		1
Sb			Tellurium	Extraction	The target is dissolved in 5 ml 9 N HCl to which is added 2 mg Sb(V) carrier. 2.5 ml $SnCl_2$ soln. is added per 100 mg target, the soln. stirred vigorously and then allowed to stand for 0.5 min. The Te ppt. is filtered and washed with 2 ml 9 N HCl. The filtrate is transferred to a separatory funnel containing 10 ml benzene, 1 ml isopropyl ether and 0.05 ml Br_2 (or as little as possible). The organic phase is shaken for about 10 sec, and washed with 1 ml 9 N HCl. The Sb is back-extracted into 5 ml 9 N HCl and 0.5-1.0 ml $SnCl_2$ soln. The above extraction—back-extraction procedures are repeated. The final aqueous soln. is used for liquid β counting or the Sb is ppted. as the metal by addition of 2-3 mg $SbCl_3$ carrier and approximately 15 ml boiling hot Cr^{++} soln. Yield = 60-70%.	< 5 min	146
Sb		U^{235}(n,f)	Fission product	Distillation	Zinc metal (40 mesh) is placed in a distilling flask and warmed to 100°C. Sb^{+3} carrier + fission product + 30% H_2SO_4 are dropped onto the Zn. The SbH_3 generated is passed through a $CaCl_2$ drying tube and then through a heated small-diameter quartz tube. The SbH_3 is converted to Sb metal, part of which collects as a mirror on the cool portion of the quartz tube and the rest (~ 50%) on a fine fritted glass filter. As is expected to follow the Sb; however, most of it collects at a different portion of the tube. The As can be removed from the Sb metal by leaching with Na-hypochlorite. Yield = 80%.	10 sec	164

Te^{131}	24.8 m	Daughter of Te^{131m}(30h)	Telluric acid containing Te^{131m}	Recoil	The method depends upon the changes of a molecule of telluric acid to one of tellurous acid when the Te nucleus in that molecule undergoes an isomeric transition to its ground state. (In order to synthesize telluric acid, the chemically purified radioactive Te is dissolved in HNO_3 and Ag-tellurite ppted. from the neutralized soln. The Ag-tellurite is suspended in H_2O and oxidized with liquid Br_2 to telluric acid. The excess Br_2 is boiled off and the product AgBr is filtered out, leaving a soln. of pure telluric acid.) To the radioactive telluric acid, small amounts of inactive Na-tellurite, iodide, and sodium iodate are added as carriers. At suitable intervals, aliquots of this sample are taken and the isomer separation performed. The soln. is first freed of I^{131} (8 day), which had grown from the Te^{131}, by reducing the iodate present with an equivalent amount of iodide and extracting the iodine with $CHCl_3$. Sufficient conc. HCl is added to make the soln. 3 <u>N</u> and SO_2 is introduced in order to reduce the tellurous but not the telluric acid to elementary Te. (Under these conditions the reaction between telluric acid and SO_2 is extremely slow and may be neglected.) This Te ppt. contains the separated daughter isomer with no detectable amount of parent isomer as contaminant.		223
Te^{131} Te^{133} Te^{135}	24.8 m 2 m < 2 m	U^{235}(n,f)		Precipitation	A fairly specific pptn. is obtained through reduction by H_2SO_3. When SO_2 is passed into a boiling 3 <u>N</u> HCl soln. containing appropriate carriers, Au, Pd, Te, and Se are ppted. completely in the elemental state. A large fraction of the Po is likewise carried down. But Au, Pd, Se, and Po are not among the strongly active products of U fission. Thus, Au and Pd are separated from Te by reduction with formic acid. Se is ppted. from 12 <u>N</u> HCl by SO_2. Te is not ppted. under these conditions. On the other hand, hydrazine HCl ppts. Te but not Po.	10 min	1

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Te^{133}	2 m	Daughter of Te^{133m}(63m), from fission	Telluric acid containing Te^{133m}	Recoil	From the irradiated uranium oxide, Te fission products are first isolated. When the parent isomer (Te^{133m}) is held in acid soln. in the valency state +6, most of the daughter will be formed in the lower valence +4. A fast daughter separation by selective reduction with H_2S is made from the Te^{133m} soln. in the +6 state.		206
Te		U^{235}(n,f)	Uranyl nitrate		In the irradiated uranyl nitrate soln., 10-50 mg of Te carrier is added. The soln. is fumed to the chloride with HCl (3 times). The residue is dissolved in 3 <u>N</u> HCl. The soln. is heated and Te ppted. by passing SO_2 into the soln. The ppt. is dissolved in several ml of 6 <u>N</u> HCl + a few drops of 6 <u>N</u> HNO_3. After expelling HNO_3 with several portions of conc. HCl, the residue is dissolved in 3 <u>N</u> HCl and the Te is ppted. by SO_2 method. This ppte. is dissolved and reppted. as before. Yield = 80-90%.	1.5-2.0 hr	198
I^{137}	22 s	U^{235}(n,f)	Uranyl nitrate aqueous soln.	Extraction	About 150 ml sample soln. is irradiated for 2 min in a separating funnel. A few mg of KI and KBr are present to act as carrier, and a few cc of CCl_4 are present. The concentration of the U-nitrate soln. is held down so that the CCl_4 would settle promptly after the funnel has been shaken. 1 ml of conc. HCl is present in the soln., and after irradiation 10 ml of 5% $NaNO_2$ soln. are added. Following shaking and settling, the CCl_4 layer is drawn off and measured with a neutron counter.		235
I^{128}	25 m	$I^{127}(n,\gamma)$	Sodium iodate	Recoil	The hot atom produced from the iodate is ppted. as AgI, in the presence of NH_4OH to prevent pptn. of $AgIO_3$.		3
I		U^{235}(n,f)	Uranyl nitrate		The irradiated U-nitrate (1-10 g) is dissolved in 25 ml of H_2O and boiled gently for 3 or 4 min to remove active noble gases. After cooling and then addition of 5 ml of 6 <u>M</u> HNO_3, Br^- carrier (15 mg) and I^- carrier (20 mg), the soln. is transferred to a separatory funnel.	1.5 hr	30

					By addition of 4-5 drops of 0.1 M $NaNO_2$, the iodine is extracted into 15 ml of CCl_4. From the CCl_4 extract, the iodine is back-extracted into 10 ml of H_2O by adding 0.5 M $NaHSO_3$ drop by drop until no color of the I_2 remains in the CCl_4 layer. Such extraction and back-extraction procedures are repeated and finally the iodine is ppted. as AgI for counting. Yield = 90%.		
I		Sb(α)	Antimony metal	Distillation	The target (500 mg) is dissolved in a cold soln. containing 10 mg I^- carrier, 10-15 ml saturated tartaric acid, and 10-15 ml HNO_3. I_2 is distilled and collected in 0.5-1.0 N NaOH. The soln. is acidified with H_2SO_4 until the iodine turns brown and is then oxidized with HNO_2 ($NaNO_2$ in acid soln.). I_2 is extracted with an equal volume of CCl_4 and then back-extracted into NaOH soln. These NaOH-CCl_4 cycles are repeated as many times as wished. The last NaOH soln. is acidified with H_2SO_4 and reduced with SO_2 gas. Air is blown through the soln. to expel most of the SO_2. The iodide is then ppted. as AgI with excess Ag^+ and a few drops of conc. HNO_3. Yield = ≥ 80%.	40 min	168
Xe^{127m}	75 s	$I^{127}(p,n)$	PbI_2 KI	Distillation	The procedure closely parallels that with Kr^{79m} and Kr^{81m}.		51
Xe^{135m}	13 m	Daughter of I^{135}(6.7 h) from $U^{235}(n,f)I^{135}$	Uranyl nitrate	Distillation	The iodine activity is extracted into CCl_4 from the fission product soln. The CCl_4 soln. is placed in a flask on a vacuum line and the air above the soln. removed. The soln. is allowed to stand for 5 min. Air is then bubbled through the soln. and into an evacuated chamber equipped with a thin mica window. This chamber is immediately placed a few cm away from the window of a GM counter tube and the measurement made immediately.		199
Xe^{139}	41 s	$U^{235}(n,f)$	Uranyl nitrate powder	Distillation	The uranyl nitrate powder is contained in a polyethylene tubing (1/4 inch, i.d.) and irradiated. The radioactive gas fission products are taken with the help of a syringe from the tubing into another polyethylene tubing in which Norite (activated charcoal) is placed. The Xe activity is absorbed in Norite, and sent to the counting equipment through a rapid transfer system.	20-30 sec	264

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Cs^{123}	6 m	$I^{127}(\alpha,8n)$	CaI_2	Precipitation	The target is dissolved in H_2O and then saturated with HCl while being cooled with ice water. The Cs is ppted. as silicotungstic acid and centrifuged. The ppt. is dissolved in 2 drops of H_2O and the Cs is re-ppted. as silicotungstic acid. Finally the ppt. is dissolved in 0.5 ml H_2O and the soln. passed through a cation exchange (1 cm x 4 mm column of Dowex-50) to separate silicotungstate ion. The resin is washed with H_2O, and then the Cs eluted from the resin with a few drops of 6 N HCl.		169, 125
Cs^{126}	1.6 m	Daughter of Ba^{126}(96 m)	Indium metal or oxide	Precipitation	A pure Ba parent activity is isolated from the indium target bombarded by nitrogen ions in a cyclotron. The method involves the pptn. of $Ba(NO_3)_2$ with fuming HNO_3 followed by pptn. of $BaCl_2$ with "ether-HCl" reagent. The rapid "milking" of Cs from solns. containing the Ba^{126}-Cs^{126} equilibrium mixture is done by pptn. of Cs-cobaltinitrite. The ppt. is quickly dissolved in fuming HNO_3 and several $Ba(NO_3)_2$ scavenges are performed.	< 3 min for Cs separation	135
Cs^{128}	3.7 m	Daughter of Ba^{128}(2.4d) from $Cs^{137}(d,7n)$	Cesium nitrate	Precipitation	After a Ba fraction is isolated from the target, the Cs^{128} daughter is separated from the Ba^{128} parent by addition of chilled absolute alcohol to a previously fumed and chilled $HClO_4$ soln. of the Ba to which Cs carrier had been added. The resultant ppt. of $CsClO_4$ is collected in a sintered glass filter and washed with alcohol.	< 1 min for separation of daughters	
Cs^{137}	26.6 y	$U^{235}(n,f)$	Fission product	Ion exchange	Anion exchange resin (De-Acidite F.F., 80-100 mesh) is used. The resin is converted to the carbonate form by passing 3 M Na_2CO_3 through the column and then is washed with demineralized water. The system consists of 1 gm of resin in a 25 cm x 0.7 cm glass column. (Care must be taken never to allow the column to run dry at any time during the procedure.) An aliquot of fission product soln. is taken and adjusted to pH 1 by addition of alkali or by dilution with H_2O. (Salt conc. must not be greater than 3 M in the finally prepared sample.)		271

					Cs, Ru, Zr, and Ba carriers are added to the soln. and 1 ml of the mixture is added to the resin column. The eluent is collected in a small polyethylene cup at about 6 drops/min (0.3 ml/min) speed. The column is washed with 4 ml demineralized H_2O and the eluent collected in the same cup. The eluent is measured by γ-spectrometry. Such an ion exchange separation can also be done by using a Dowex-1 anion exchange resin column in the carbonate form at pH of about 6. The Cs elutes in the first 3 ml. The column is then washed with 5 ml H_2O. (The resin serves as a solid precipitant for ppting. Zr, Nb, Ru, and Ce.) Yield = ~ 100%.		186
Cs^{139}	9.5 m	$U^{235}(n,f)$	Uranyl nitrate	Precipitation	After 1 min irradiation in the pneumatic tube, the irradiated material is dissolved in 10 ml H_2O containing 2 ml Cs carrier (~ 10 mg/ml) and 1 ml Rb carrier (~ 10 mg/ml). A pptn. of the mixed Cs-Rb cobaltinitrites is performed about 1.5 min after the end of irradiation. The centrifugation of the ppt. is finished by 2.5 min after the end of irradiation. The purpose of these rapid operations is to minimize the activity of 32 min-Cs^{138} growing from 17 min-Xe^{138}. The cobaltinitrite ppt. is dissolved in dil. HCl and Cs is separated from Rb by pptn. of Cs with BiI_3-HI reagent. This ppt. is dissolved and a $Fe(OH)_3$ scavenging pptn. is made. The Cs is finally pptd. as the silicotungstate and counted.	12 min	249
Cs^{139}	9.5 m	$U^{235}(n,f)$	Fission product	Extraction	An aliquot ($\leq$ 5 ml) of the fission product soln. is taken and 10 ml buffer soln. (1 $\underline{M}$ $Na_3C_6H_5O_7$ in 0.5 $\underline{M}$ HNO_3, pH = 6) is added in a separatory funnel. The total volume of the soln. is adjusted to 15 ml with H_2O. After addition of an equal volume of 0.05 $\underline{M}$ Na-tetraphenyl borate in amyl acetate, the Cs is extracted into the organic phase by shaking for about 30 sec. The aqueous phase is transferred to another separatory funnel and the extraction is repeated. The organic phases are combined in the first separatory funnel and the Cs activity is back-extracted into two successive 10 ml portions of 3 $\underline{N}$ HCl from the amyl acetate phase. All the above procedures are repeated once again. Yield = 100%.	30 min	81

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
		Daughter of Xe^{139}(41 s) from U^{235}(n,f)	Uranyl nitrate aqueous soln.	Distillation for Xe, precipitation for Cs	Air is bubbled through an irradiated uranyl nitrate soln. at a rate of 600 ml/min. It is then passed through a tube filled with cotton wool (length 15 cm) and through a wash bottle containing 40 ml of H_2O with a small amount of HNO_3 and 1 drop of NH_4OH. After adding Cs, Rb, Ba, Sr, and La carriers, Cs is ppted. with antimony chloride dissolved in HCl.	15 min	116
Cs		U^{235}(n,f)	Fission product	Precipitation	Cs and Rb are separated from most of the other fission product cations by the following procedure. First appropriate carriers and then NaOH and Na_2CO_3 are added. The alkali metal ions remain in the soln. and are purified further by a $La(OH)_3$ scavenging pptn. The Cs is then separated by pptn. of $Cs_3Bi_2I_9$ with BiI_3 in HI. After re-pptn., the ppt. is dried and measured		75
Ba^{137m}	2.63 m	Daughter of Cs^{137}(26.6y)		Precipitation	Sulfate ions are added to a soln. containing the active Cs, and the soln. is then placed in contact with solid $BaSO_4$. The $BaSO_4$ is washed and measured.		260
Ba^{141} Ba^{142}	18 m 6 m	U^{235}(n,f)		Precipitation	The sample soln. is placed in a centrifuge tube and Ba carrier (~ 20 mg) added. To the cool soln. (~ 5 ml), 30-35 ml of HCl-ether reagent is added and the soln. is stirred for 1-2 min. Thereafter the ppt. is centrifuged, dissolved in 1 ml H_2O and re-ppted. in 15 ml of HCl-ether mixture. Such dissolution and pptn. is repeated. The final ppt. is washed with alcohol-ether.		105
Ba		U, Th, Bi (fission)		Precipitation	An aliquot of HNO_3 or HCl soln. of the target is taken and Ba carrier is added to the soln. If necessary, the soln. is evaporated to less than 1 ml. By addition of 10 ml ether-HCl reagents, $BaCl_2 \cdot 2H_2O$ is ppted. in ice temperature. After stirring and digesting for 2 or 3 min, the ppt. is centrifuged and dissolved in 0.5 ml H_2O. This pptn. procedure is repeated. The ppt. is dissolved in 0.5 ml H_2O, the soln. is diluted to 7-8 ml, and made up to a pH > 10 with carbonate and free ammonia. The scavenging with $Fe(OH)_3$ is done twice. Then $BaCO_3$ is ppted. by addition of 3 drops sat. Na_2CO_3. This	30-45 min	117

					$BaCO_3$ ppt. is dissolved in 1 ml of 6 N HCl and the $BaCl_2 \cdot 2H_2O$ pptn. by the above procedure is repeated twice. The ppt. is centrifuged and washed with 5 ml absolute alcohol and then 3 portions of 5 ml ether. Yield = ~ 100%.		
La^{134}	6.5 m	Daughter of Ce^{134}(72 h), La(p,6n)	Lanthanum oxide	Precipitation	The bombarded material is dissolved in HNO_3 and Ce^{+3} and Ba carriers are added. The material is then subjected to fluoride-hydroxide cycles. This consists in ppting. the fluoride from warm HNO_3 soln., dissolving the fluoride in 8 N HNO_3 saturated with H_3BO_3, ppting. the hydroxide by NH_4OH, and dissolving in HNO_3. This method is specific for rare earths and Y. Separation of the La daughter activity is effected by pptn. of the fluoride from the Ce^{+4} soln. containing Ce^{134}.		242
La^{143}	19 m	U^{235}(n,f)	Uranyl nitrate	Precipitation	After 20 min irradiation in the reactor, U-nitrate is immediately dissolved in 1 M HNO_3 containing La carrier. Lanthanum fluoride is ppted., centrifuged, and washed with H_2O. This LaF_3 is dissolved in a mixture of H_3BO_3 and HNO_3. At intervals after preparation, this soln. is sampled, Ce carrier is added, the Ce is oxidized with $KClO_3$ and $Ce(IO_3)_4$ is ppted. By means of the measurement of the long-lived Ce^{143} (33 h) activity (daughter of La^{147}) the half-life of La^{143} is determined.		96
Ce^{145}	3.0 m	U^{235}(n,f)	Uranyl nitrate (10-100 mg)	Extraction	Ce^{+4} is separated by solvent extraction from 10 N HNO_3 with methyl isobutyl ketone, while the trivalent rare earths as well as the other fission products remain in the aqueous phase. $NaBrO_3$ is used as the oxidizing agent for the Ce. The organic layer containing the Ce^{+4} is washed with 10 N HNO_3 and then the Ce is back-extracted into an aqueous phase by reduction to Ce^{+3} with H_2O_2. This extraction and back-extraction cycle is repeated 2 or 3 times. Finally the Ce is ppted. as ceric iodate.	~ 9 min	167
Ce		U^{235}(n,f)	Uranyl nitrate	Precipitation	Ce, La, Y, and Zr carriers are added to the dilute uranyl nitrate soln. (UO_2^{++} conc. of ~ 10 mg/ml). The soln. is diluted to 10 ml; 10 ml of conc. HNO_3 is added; and $Zr(IO_3)_4$ is ppted. by addition of 10 ml of 0.8 N HIO_3.	20-30 min	222

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					The supernatant soln. is then oxidized with 1 g of $KBrO_3$ [no heating is necessary to oxidize Ce(III) to Ce(IV)], and $Ce(IO_3)_4$ ppts. The $Ce(IO_3)_4$ is dissolved in H_2O_2, a small amount of HNO_3 and H_2O, and the cycle of $Zr(IO_3)_4$ and $Ce(IO_3)_4$ pptn. is repeated. The oxidation of Ce(III) proceeds more rapidly with BrO_3^- than with ClO_3^-. In addition, the danger of incomplete oxidation of the Ce is avoided by the use of BrO_3^-.		
Pr^{135}	22 m	$Ce^{136}(p,2n)$	Enriched Ce^{136}	Ion exchange	An ion exchange method (139) is applied. The elution with α-hydroxy isobutylic acid should be more advantageous than the citrate elution (43).		114
Pr^{144}	17.5 m	Daughter of Ce^{144}(235d) from $U^{235}(n,f)$	Ce^{144} sample	Precipitation	(1) The ceric oxide containing Ce^{144}, which is purified from the irradiated uranyl nitrate, is dissolved in H_2SO_4 and H_2O_2. La carrier is added to the soln. to carry the Pr and ppted. as the hydroxide by NH_4OH. The ppt. which co-ppts. Pr^{144} is filtered and dissolved in HNO_3. Ce is oxidized to the ceric state with $KBrO_3$ and ppted. as $Ce(IO_3)_4$ by addition of KIO_3. The ppt. is centrifuged off. The supernatant soln. is made basic with NH_4OH, and $La(OH)_3$ ppted. Pr^{144} is co-ppted. in the $La(OH)_3$ and measured.		197
					(2) To the Ce^{144} sample, Pr and La carriers are added. The Ce activity is ppted. as $Ce(IO_3)_4$ and then the Pr ppted. as the hydroxide with 6 N NaOH. The hydroxide is dissolved with 6 N HCl and the Pr ppted. as oxalate by addition of sat. $H_2C_2O_4$.		226
				Leaching	Ce^{144} is ppted. as iodate by the usual method. The iodate is mixed with glass wool and placed in an ice-cooled column (10 mm i.d., 15 mm height). When this column is eluted with HNO_3, Pr^{144} is eluted together with Ce and the amount of Ce increases with increase of HNO_3 acidity. However, by use of 0.005 N HNO_3 (flow rate: 3 ml/min), the amount of Ce eluted is negligible and the radiochemical purity of P^{144} obtained is ~ 97%.		272

Pm^{141}	20 m	$Nd^{142}(p,2n)$	Enriched Nd^{142} oxide	Ion exchange	The chemical procedures are designed to isolate the rare earth elements as a group but not separate them from each other. The bombarded oxides are dissolved in HNO_3 from which the rare earth fluoride is ppted. This is dissolved and the hydroxide ppted. and dissolved in conc. HCl, which is sucked through a column of anion exchange resin (Dowex A-1). The rare earth hydroxide is then prepared for counting sample. In those cases where complete identification is desirable, rare earth separations are made using a cation exchange resin column (Dowex-50, citrate elution at pH 3.5) and elevated temperature as described by Thompson et al., as follows: The fluoride ppt. is converted to a hydroxide by treatment with 6 M KOH followed by centrifuging and washing. The hydroxide ppt. is dissolved in dil. $HClO_4$ (0.5 M) and the mixture of radioactivities absorbed on a small amount of ammonium form Dowex-50 resin (spherical fines). This is then transferred to a 20 cm length, 2 mm diameter column packed with the same resin. The column is surrounded by a vapor jacket through which passes trichloroethylene vapor to maintain the temp. at about 87°C. The elution is performed by passing amm. citrate (buffered with citric acid to pH 3.5— total citric acid conc. 0.25 M) through the column at a rate of 1 drop (~ 0.030 cm^3) about every 2 min and the drops are collected separately. The use of α-hydroxy isobutyric acid (pH 4.6, 0.4 M) as the eluting agent should be more advantagous, rather than the elution using citric acid.(43).		144 254
Ho^{160}	~ 22 m	Dy(p)	Dysprosium oxide	Ion exchange	The ion exchange method (139) is applied. As the eluting agent, the use of α-hydroxy isobutyric acid should be more advantageous (43).		112
Yb^{167}	19 m	$Tm^{169}(p,3n)$	Thulium oxide	Ion exchange	In order to obtain a separation rapidly, the separation method is somewhat modified for the Ketelle-Boyd method (139). The ion exchange column is 35 cm long and 1.8 cm i.d. and filled with Dowex-50 (200-500 mesh). It is heated by a steam jacket. The elutriant, 0.25 M citric acid is adjusted to a pH 3.18 with NH_4OH, and	2.1 hr	113

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					is preheated with steam and an infrared heat lamp. The storage bottle for the elutriant is placed 20 feet above the column to produce sufficient hydrostatic pressure for a flow rate of 180 ml/hr. As the eluting agent, the use of α-hydroxy isobutyric acid (0.2 M, pH 4.0) should be more advantageous than the previous one (43).		
Ta^{178}	8.35 m	Daughter of W^{178}(21.5d), Ta^{181}(p,4n)		Precipitation	The Ta daughter activity is separated by co-pptn. with Ni(OH)$_2$ from the boiling strongly alkaline tungstate soln. The washed hydroxide is dissolved in acid, excess tungstate soln. added and the boiling soln. made alkaline once again. After 3 cycles of these procedures, the active Ta is finally collected on a small amount of Zr-hydroxide by pptn. from HCl soln. with addition of NH$_4$OH. A large amount of Ni remains in soln.		268
Ta^{182m}	16.5 m	Ta^{181}(n,γ)	(NH$_4$)$_2$TaF$_7$	Precipitation	The target is dissolved in HF soln. and the Ta ppted. as the hydroxide by addition of NH$_4$OH. Re-pptns. are made to increase purity.		227
Ta		Hf(p,n)	Hafnium oxide	Extraction	The target oxide (~ 350 mg) is dissolved in 6 N HCl-1 N HF in a Pt dish and evaporated to 0.5-0.75 ml. The ppt. of Hf is centrifuged off. The supernatant soln. is cooled in an ice bath and centrifuged to remove any Hf that ppts. Ta in the supernatant soln. is extracted with an equal volume of di-isopropyl ketone which had been previously equilibrated with 6 N HCl-1 N HF. The Ta in the organic phase is back-extracted into an equal volume of H$_2$O.		77
				Precipitation	The target oxide is dissolved in a mixture of HNO$_3$ and HF. Excess HF is removed by evaporation with 10 N HNO$_3$. KMnO$_4$ is added to the soln. and MnO$_2$ is ppted. The MnO$_2$ ppt., in which the Ta activity is co-ppted., is dissolved in oxalic acid and Mn^{++} is removed by adsorption on a cation exchange resin (Amberite IR-1 or Dowex-50).		188

W^{178}	30 m 5.2 m	Ta(p,3n)	Tantalum metal	Extraction	The target is dissolved in the minimum amount of a mixture of HNO_3 and HF. To the hot solution, hydrazine sulfate is added to reduce any HNO_3 present. Sufficient strong HCl to give a 6 N solution is added followed by $SnCl_2$ soln. Then NH_4SCN soln. is added followed by excess boric acid to complex the fluoride. The green thiocyanate complex of W^{+4} formed is then extracted into ethyl acetate. The washed solvent layer is evaporated, hold-back carriers for various elements are added and W^{+4} ppted. by boiling with strong HNO_3. The oxide is dissolved in HN_4OH, thiocyanate added to the soln. and the reduction-extraction process repeated after acidification.	268
Re^{177}	17 m	W(p)	Tungsten metal or oxide	Extraction	The tungsten target is dissolved in a NaOH and H_2O_2 soln. by heating, in the presence of K-perrhenate as carrier. The soln. is scavenged with Zr and La hydroxides and then made 4 N in NaOH. The resulting soln. is equilibrated once with an equal volume of pyridine previously shaken with 4 N NaOH (108). The pyridine layer, which contains the Re, is washed with an equal volume of 4 N NaOH, and then the Re is back-extracted to 4 N HCl soln. The HCl soln. is heated to boiling, and saturated with H_2S. The Re_2S_7 ppt. is separated, washed with H_2O, and dissolved in 6 N NaOH + 30% H_2O_2 (1:1). The resulting soln. is heated, diluted, and scavenged with Zr and La hydroxides. After neutralizing the soln. with HCl and cooling in ice water, tetraphenylarsonium chloride is added. The Re ppt. is separated, washed twice with ice water and counted.	111
Re Re^{180}	9.8 m 2.8 m	Os(n,p) $Os^{182}(d,\alpha)$	Osmium metal	Precipitation	The target is dissolved in an alkaline soln. of persulfate, containing Re and W as carriers. After addition of bromate and sulfuric acid, Os is distilled off as OsO_4. The remaining soln. is made alkaline, Fe^{+3}, Cu^{+2}, and Mg^{+2} are added and their hydroxides ppted. Then $CaCl_2$ is added to the filtrate to ppt. Ca-tungstate. The soln. is boiled and filtered. An excess of HCl is added to the filtrate, bromine is expelled by boiling and the Re ppted. as tetraphenylarsonium perrhenate.	8

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Re		Ta, W $\begin{pmatrix}\alpha\\ d\\ p\end{pmatrix}$	Metal	Precipitation	The targets are dissolved in HF with a minimum amount of HNO_3 in a Pt dish and Re carrier added. H_2S is passed into the boiling soln. for 15 min to ppt. black Re_2S_7. The ppt. is centrifuged and washed in a lusteroid test tube. The ppt. is dissolved in 3 ml 1 <u>N</u> NaOH + 1 drop 30% H_2O_2, and the soln. transferred to a glass centrifuge cone. After scavenging with $Fe(OH)_3$ (1 mg Fe^{+3}) two or three times and then dissolving in 10 ml of conc. HCl, the Re is ppted. as Re_2S_7 from strong HCl soln. The ppt. is centrifuged and washed. Yield = ~ 100%.	30 min	118
Re			Molybdenites	Extraction	After the preliminary attack of the ores, Re is extracted quantitatively between pH 8 and pH 9 by $CHCl_3$ in the form of tetraphenylarsonium perrhenate. In this pH region the Mo remains in the aqueous phase.		261
Os^{190m}	9.5 m	Daughter of Ir^{190}(11d), Re(α,n) from Os(d,n) Os(d,2n)	Rhenium or osmium metal	Distillation	The Re target is dissolved in dilute HNO_3. The Os target is dissolved in aqua regia. These dissolutions are performed in all glass distilling flasks. Chemical separations are made after the addition of the proper inactive carrier. Os is distilled as OsO_4, followed by pptn. of the sulfide with Na-thiosulfate.		46
			Rhenium metallic powder	Distillation	The target is dissolved in 3 ml HNO_3. 10 mg Os carrier is added and the Os is distilled as OsO_4 by heating. The distillate is caught in ice-cold 6 <u>N</u> NaOH and then neutralized and acidified with 6 <u>N</u> HCl. The Os is ppted. as the sulfide by addition of 3 ml 10% $Na_2S_2O_3$ while the sample soln. is heated on water bath. Yield = ~ 100%.	~ 20 min	45
Ir^{191m}	4.9 s	Daughter of Os^{191}(16 d), from $Os^{190}(n,\gamma)$	Osmium metal	Distillation for Os^{191}	The irradiated Os is converted to ammonium perosmate, $(NH_4)_2OsO_5$, dissolved in dil. NH_4OH. To prepare the Ir activity, portions of this soln. with		190

					added Ir carrier are dried on 1 mil Pt foils and then flamed over a Bunsen burner to distill off the Os, leaving the Ir on the foil.		
			Osmium oxide	Ion exchange	The irradiated sample (0.1 gm OsO_4) is dissolved in 6 N HCl containing 1 ml ethyl alcohol, and the resulting soln. refluxed for 5 hr near 100°C to convert the Os to the red $OsCl_6^{-2}$. An aliquot containing about 0.01 mc Os^{191} is added to a small column (1 ml) of anion exchange resin which had been treated with 6 N HCl. The Os complex absorbs as a reddish brown band at the top of the resin bed. When 6 N HCl is rapidly forced through the bed with a hypodermic syringe, a short-lived activity of Ir^{191m} is found in the effluent.		32
Ir^{197}	7 m	Pt(γ,p)	Platinum chloride	Precipitation	The target $PtCl_4 \cdot XH_2O$ is dissolved in conc. HNO_3. Os and Ir carriers are added to the soln. and Os is distilled as OsO_4. To the remaining HNO_3 soln., $NaBrO_3$ is added. The soln. is heated and neutralized by addition of $NaHCO_3$. The IrO_2 ppt. is filtered, dissolved, and re-ppted. This second pptn. removes the Pt activity beyond the limits of detection.		44
Pt		Ir(p)	Iridium metal (foil)	Extraction	The Ir target is dissolved in a Pt flux of KOH and KNO_3 (1:1) (for a small strip of 1 mil foil this should take no longer than 5-10 min). After cooling, the melt is leached for 5 min with conc. HCl, and Au and Pt carriers added in small amounts. (Ir gives a strong blue colored soln.) Au is extracted twice with ethyl acetate. A little $SnCl_2$ soln. in HCl is added to the aqueous soln. to reduce Pt^{+4} to Pt^{+2}. The red coloration (Pt) is extracted into ethyl acetate and washed twice with an equal volume of 3 N HCl. Yield = ~ 70%.	30 min	256
Au^{188}	4.5 m	$Ta \begin{pmatrix} C^{12} \\ C^{13} \\ C^{14} \end{pmatrix}$	Tantalum metal (foil, 40 μ thick)	Extraction	The target is dissolved in a minimum of 27 N HF and 16 N HNO_3 with addition of 100-100 μG quantities of Hg, Au, Pt, and Te carriers (and if necessary, Ir). These elements are then ppted. as the metals by $SnCl_2$. The ppt. is dissolved in aqua regia and the Au extracted into amyl acetate.		38

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Au^{203}	55 s	$Hg^{204}(\gamma,p)$	Mercury	Distillation for Hg	A full radiochemical identification of the 55-sec activity as Au was not possible in the time available. This activity is prepared by irradiating very pure Hg which is then poured onto a few mg of Au foil, distilled off, and the residue heated to redness, thus leaving only Au and Pt.		29
Au		Au(p)	Gold metal (foil, 0.004 mil)	Extraction	The Au is dissolved in 1-2 ml of warm aqua regia (2 $\underline{N}$ $HNO_3 \cdot 4$ $\underline{N}$ HCl) and hold-back carriers for Hg, Pt, Ir, and Os (~ 1 mg each) added. The Au is extracted into ethyl acetate and the organic phase is washed with 6 $\underline{N}$ HCl. Yield = ~ 100%.	4-10 min	257
Au		$Ir\begin{pmatrix}\alpha\\p\\d\end{pmatrix}$	Iridium metal (foil)	Extraction	The target Ir is fused with a melt of KOH and KNO_3 (1:1). The flux is allowed to cool and then leached for ~ 5 min with conc. HCl after adding Au and Pt carriers in small amounts. Au is extracted twice with ethyl acetate and the organic phase is washed twice with equal volume 3 $\underline{N}$ HCl. Yield = ~ 70%.	20-30 min	258
Au		Daughter of Hg activity	Tracer Hg	Extraction	The Au daughter nuclide in 4 ml 3 $\underline{N}$ HCl soln. of the Hg tracer is extracted with 500 λ of isoamyl acetate. (The presence of at least 0.1 $\underline{N}$ Cl^- is required for separation from Hg.) The organic layer is washed with equal volumes of sat. NH_4Cl to insure separation from Hg and evaporated on a counting plate. Yield = quantitative if several extractions made.	~ 10 min	78
Hg^{189}	20 m	$Ta\begin{pmatrix}c\\N\end{pmatrix}$	Tantalum metal (foil 40 μ thick)	Ion exchange Distillation	The target is dissolved in a minimum amount of 27 $\underline{N}$ HF and 16 $\underline{N}$ HNO_3 with addition of 100-1000 μg Hg, Au, Pt, Te, and, if necessary, Ir carriers. These elements are then precipitated with $SnCl_2$. The metallic ppt. is dissolved in aqua regia and the Au is separated by extraction into amyl acetate. The aqueous phase is evaporated to a small volume and then passed through an anion exchange column (Amberite IR-400, 250 mesh, 13-15 mm long, 0.055 cm^2 cross section). 0.1 $\underline{N}$ HCl is used as the eluent at a flow rate of 0.1-0.2 ml/min.		38

					The recovery of Te is complete in 2 min or less. The Hg is then eluted with 2 N HNO_3. Decontamination from Pt is, however, not good; about 1/3 of the Pt initially present would be eluted with the Hg. Final decontamination from the Pt can be achieved by vacuum distillation of Hg. But this decontamination step is not so necessary because it is possible to distinguish easily between the Hg and Pt isotopes on the basis of half-life.		
Hg		Pt Au Tl $\begin{pmatrix}\alpha\\ p\\ d\end{pmatrix}$	Metals	Distillation	The target is placed in the chamber of a stainless steel vaporizer. A clean Pt collecting plate is cemented to a stainless steel water-cooled finger. The bottom of the apparatus is heated for about 5 min with a Fisher burner to not more than ~ 400°C. Because of the large difference of the vapor pressure between Hg (1574.1 mm at 400°), Au (0.001 mm at 1292°), and Tl (0.001 mm at 413°) at this temperature, the Hg activity (carrier free) can be separated on the collecting plate very quickly. Yield = enough activity to work with but by no means quantitative. For more yield heat longer.	1-5 min	79, 80
Tl^{185m} Tl^{187m}	3.5 s 0.6 s	Daughters of Pb^{185m}(17m) and Pb^{187m}(42m), from Tl(p)	Thallium metal	Precipitation	Separation for Pb parent: After irradiation, the Tl target is dissolved in conc. HNO_3 containing about 5 µg Pb^{+2} and 250 µg Fe^{+3} as carriers. In order to reduce Tl to the monovalent state, the soln. is boiled with H_2O_2 until excessive peroxide has disappeared. Fe and Pb are ppted. with NH_3, the role of the Fe being to carry down the more soluble $Pb(OH)_2$ as a coprecipitate. After centrifuging, the ppt. is dissolved in HNO_3, reppted., and washed in H_2O, 3 times. Finally the hydroxides are dissolved in the smallest possible amount of HCl (about 10 drops of 0.1 N acid).		5
				Extraction	Separation for Tl daughter: The Pb activity is kept in 6 N HCl. Three ether extractions are first made in order to get rid of the Fe carrier. To oxidize Tl(I) to Tl(III) in the soln., a drop of $KMnO_4$ soln. is added immediately before each ether portion (about 3 ml). The soln. is stirred with a glass rod for some seconds in order to bring the ether into intimate contact with the aqueous phase.	5 sec	

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					Then that part of the ether phase which is formed first (nearest to the surface) is pipetted off and transferred to a test tube which is put into counting position as fast as possible.		
Tl^{206} (RaE")	4.19 m	Daughter of Bi^{210}(RaE, 5.0d)	RaD-E-F	Extraction	100 mg Pb, Bi, Tl, and Te carriers are added to the RaE-D-F stock soln. and then 5 ml aqua regia is also added. The soln. is evaporated, HCl is added and the soln. evaporated once again to expel HNO_3. The soln. is made up to 6 N with HCl and extracted with 5 ml ether (pre-equilibrated with 6 N HCl). The ether phase is washed twice with Pb-Bi-Tl carrier soln. 1 ml of ethyl alcohol is added to the ether layer and ether is vaporized.		266
			$BiCl_3$ soln. containing RaE	Extraction	To the soln. of Bi chloride containing RaE, about 12 mg $TlNO_3$ is added, together with excess Br_2 to oxidize the Tl and the mixture heated on a water bath. After the $TlNO_3$ has dissolved and the color of the Br_2 has more or less disappeared, the soln. is chilled with ice and stirred quickly with 2 cc of ether for about 10 sec. The ether phase is then pipetted off, washed 4 times with 1:1 HCl in a separatory funnel, and evaporated on a counting glass disc.		20
			RaE source on Pt plate	Recoil	A brass button collector (2.0 cm diameter) is held at a distance of 6 mm from a Pt disc (7 cm^2) carrying the RaE (about 21 mc). The collecting voltage of 450 volts is applied for 12 min. Preparation of the RaE source: A soln. of RaD-E-F of about 50 mc strength is freed from most of the Po by agitation of 6 Cu foils. Thereafter, 2 Ni foils are agitated in the soln. for 45 min each; the acidity being kept at N/10 HCl and the temperature constant. The Ni foils are dissolved in HNO_3, and about 200 mg didymium nitrate (mixture of Pr and Nd) is added. The rare earths and RaE are ppted. as the hydroxide with		20

					NH_3. The ppt. is washed with NH_3 to remove all traces of Ni and is dissolved in trichloracetic acid. The soln. is evaporated to dryness, the residue again taken up in trichloracetic acid (2%), and electrolyzed for 12 hr using Pt electrodes, each of about 7 cm^2, at 1.9 V and 1 mA. About 21 µC of RaE is found on the cathode after the electrolysis. The weight of deposit is certainly not more than 0.1 mg and may have been considerably less, since the surface of the Pt showed no discoloration.		
Tl^{206}	4.19 m	$Tl^{205}(n,\gamma)$	Rock, mineral, meteorite, alloy	Extraction	The irradiated sample (200-1000 mg) is fused with Na_2O_2 (3.5-4 gm). (For samples containing large amounts of silica, digest with H_2SO_4 and HF in Pt.) The melt is dissolved in 20 ml 1 $\underline{N}$ HBr and then 20-25 ml conc. HBr is added to the soln. 10 mg each of Al, Cu, Mn, K, and Zn carriers plus 5 ml Br_2—H_2O are added to the soln. The Tl^{+3} is extracted 2 times into isopropyl ether (each 20 ml). After washing the organic phase with 15 ml portions of 1 $\underline{N}$ HBr (3 times) and evaporating off the ether, the Tl is ppted. as TlI by reduction with SO_2 gas and addition of excess saturated KI. The ppt. is filtered and washed with water. [The Tl can also be ppted. by thionalide after pH adjustment by 20% Na-tartrate and addition of 20% KCN, or the Tl can be ppted. as $TlB(C_6H_5)_4$ by addition of 2% $NaB(C_6H_5)_4$ after reduction by SO_2]. Yield = 45-80%.	10-14 min	27
Tl^{209}	2.2 m	Daughter of Bi^{213}(47m), from Ac^{225} decay chain	Ac^{225} aqueous soln.	Extraction	10 mg Tl carrier and a small amount of Br_2 are added to 2 ml of 6 $\underline{N}$ HCl containing Ac^{225}. The soln. is heated to oxidize the Tl to Tl^{+3} and excess Br_2 boiled out. The soln. is cooled, 2 ml of ether added and the mixture stirred for 30 sec. The ether phase is transferred to another vessel and washed for several seconds with an equal volume of ether saturated with 6 $\underline{N}$ HCl. Such washing is repeated 2 or 3 times with fresh portions of acid. After final washing, the ether is transferred to a Pt dish and ignited. Preparation of Ac^{225} solution: Ac^{225} is separated from a stock soln. of U^{233} which has decayed for one year, as follows: Ac^{225} is separated by co-pptn. with LaF_3 and is thor-	3-5 min	110

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					oughly purified by a procedure involving extraction of the Ac^{225} with a benzene soln. of thenoyltrifluoroacetone to separate from Ra and Th isotopes. This is followed by addition of Pb carrier and pptn. of PbS to remove any extraneous Pb, Bi, or Po activities.		
Tl		Daughter of Pb	$PbCl_2$ HCl soln.	Extraction	The Pb parent soln. is kept in 10 ml of 6 N HCl. Tl carrier may be added if desired, but it is not necessary. 3 or 4 drops of $KMnO_4$ soln. are added to oxidize Tl^{+1} to Tl^{+3}. The soln. is stirred until loss of brown color. After addition of 10 ml HCl-saturated ether, Tl^{+3} is extracted into the ether. A second extraction by ether is made by the same method and the ether phases are combined. The mixture is then washed with 10 ml of 6 N HCl. Yield = ~ 96%.	10 min	193
Pb^{199m} Pb^{201m} Pb^{203m} Pb^{207m}	12.2 m 61 m 6.1 s 0.8 s	Daughter of: Bi^{199}(25m), Bi^{201}(111m), Bi^{203}(13.3h), Bi^{207}(27y), from Pb(d)	Lead metal	Ion exchange	With the application of pressure by a syringe, an anion exchange column (Dowex-1, 200 mesh), which holds Bi parent activity in 0.3 N HCl is eluted with 0.3 N HCl. The eluate is rapidly transferred to a counter and measured. Preparation of the parent Bi: The target Pb is dissolved in HNO_3, and the bulk of the Pb is ppted. by addition of HCl in an ice bath and centrifuged. The remaining soln. is then boiled with addition of more HCl until no trace of nitrate remains. After dilution to about 0.3 N HCl, the soln. is slowly passed through Dowex-1 resin column (200 mesh), which is heated to the boiling point of isopropyl alcohol (82.3°C) (191). This results in a very effective separation of Pb and Bi, the Bi adhering strongly to the resin.	3 sec	240, 6
Pb^{207m}	0.8 s	Daughter of Bi^{207}, Pb(d)		Ion exchange	The Pb^{207m} is eluted with 0.5 N HCl from Dowex-1 resin (about 1 cm^3) on which Bi^{207} is absorbed. A few drops of the eluate are transferred rapidly to a counter.		31, 32
Pb		Daughter of Bi	Bi metal	Precipitation	The parent Bi is purified by the BiOCl pptn. method (as described below).	15 min	194

					This ppt. is dissolved in a few drops of conc. HNO_3, 10 mg Pb [as $Pb(NO_3)_2$] and 3 drops of conc. HNO_3 added and the soln. diluted to about 8 ml. NH_3 is added dropwise until the soln. becomes just clear. At the desired time for the milking of Pb daughter activity, the soln. is diluted to 45 ml and heated in a water bath for a few minutes. The fine crystals of BiOCl produced is centrifuged out. To the supernant, 2-3 ml of $Na_2Cr_2O_7$ soln. is added to ppt. Pb as $PbCrO_4$. The $PbCrO_4$ ppt. is centrifuged, and dissolved in 2 ml 4 N HCl + one drop of 30% H_2O_2. The soln. is heated in a water bath to destroy excess H_2O_2. By addition of 20 mg Bi carrier, a BiOCl pptn. is performed by a method similar to above. Finally $PbCrO_4$ is ppted. once again. Yield = about 85%.		
$Bi^{<198}$	1.7 m	$Pb\binom{d}{p}$	Lead metal	Precipitation	The lead is dissolved in a minimum amount of 6 N HNO_3. 8 N NaOH soln. is added to the soln. until a $Pb(OH)_2$ ppt. just appears and then HNO_3 is added dropwise until the soln. becomes just clear. A Ni foil is immersed in the soln. for several min, stirring continuously to obtain highest yield of the Bi activity on the Ni foil. The Ni foil is washed, dried and measured. Yield depends upon the immersion time of Ni foil. 10% in 5 min, 25% in 15 min, 65% in 45 min.	15 min	195, 196
Bi		Daughter of At	At plated on Ag foil	Recoil	The At, plated on Ag foil, is placed in a vacuum chamber with a collecting plate maintained at a few 100 volts negative potential. Some At volatilizes across the gap and contaminates the Bi daughter caught by α-particle recoil. To remove this, some H_2SO_4 is placed on the Pt collecting plate and the plate is heated until the H_2SO_4 is fumed off. This step converted the Bi to its sulfate which remains on the plate while the At is removed by the flaming.		14
Po^{204-x}	2-4 min	$W^{184}(Ne^{20},xn)$	Tungsten metal (foil)	Distillation	Immediately after the cyclotron bombardment, the target (size: 13 x 19 mm - thickness: 45μ) is placed under an infra-red lamp to be heated. A few drops of a	2-3 min	201

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					soln., which is about 9 N in HF and 2 N in HNO$_3$, are pipetted onto the target surface and spread over the entire bombarded area. In the minute during which the acid evaporates, dissolution of the W surface occurs, producing a thin salt layer. The dried target is then placed on a support. A half-cm-thick asbestos ring, lying around the W plate, acts as a heat insulator and collimator, while a Pt plate, lying on the ring, catches the quickly distilled Po from the heated target. It is necessary to cool the Pt catcher foil with a small amount of moist tissue. The yield of Po = < 20%.		
				Electro-deposition	The target (0.184 g W) is dissolved in a mixed solvent of 1.0 ml of 10.7 N HF and 0.2 ml of 14.2 N HNO$_3$. It is then diluted to make 20 ml soln. of 0.54 N HF, 0.14 N HNO$_3$ and 0.1% tartaric acid. The soln. is electrolyzed as follows: the cathode: round disc, painted on one side with nail polish the anode: Pt wire distance between electrodes: 2 cm voltage: 5 volts current: 50 mA/cm^2 time: 10 minutes Yield = about 30%.		201
Po^{200}	11 m	Bi^{209}(p,10n)	Bismuth oxide	Extraction	The target is dissolved in HCl and the soln. adjusted to about 6 N HCl. The Po in the soln. is extracted into 20% TBP in dibutyl ether. The organic phase is washed several times with 6 N HCl and evaporated onto Pt counting discs.	30 min	136
Po^{210}	138.4 d	Daughter of RaE	RaD·E·F aqueous soln.	Extraction	The sample soln. is made 0.25 M in KI and 3 N in HCl. 1 ml of the soln. is transferred to a separatory funnel containing 4 ml of isopropyl ether. Po is extracted into the organic phase with gentle swirling, while Pb(RaD) and Bi(RaE) remain in the aqueous phase. Po is then back-extracted into 1 ml of 3 N HCl. Yield = ~ 80%.	< 20 min	143

Po^{211}	0.52 s	Daughter of At^{211}(7.2h), from Bi(α,n)	Bismuth metal	Distillation for At	The bombarded thick Bi target is heated in a vacuum and the evaporated At is caught in a liquid air trap. The At in the trap is dissolved in HNO_3. A few drops of the At^{211} (HNO_3 acidic) soln. are put on the Pt strip (thickness: 1.2-1.7 mils, about 1 cm wide and 5 cm long) and allowed to evaporate to dryness. This strip is connected through a switch to a transformer in order that it can be brought rapidly to a dull red heat by the passage of a large current pulse. This strip is mounted on a sliding holder so that it can be positioned rapidly over the thin window of a gas flow proportional counter. The procedure is to position the strip away from the counter, passing a single pulse of current through it to drive off the At and then slide it over the counter window in order to measure the Po^{211} activity.		236
Po^{211m}	25 s	Pb^{208}(α,n)	Lead carbonate	Precipitation	(1) The $PbCO_3$ is dissolved in hot 6 N HCl containing 1.0 mg/l of Bi as a hold-back carrier. A piece of Ag foil is inserted in the soln. and stirred for about 1 min. Then the Ag foil is removed, washed in distilled H_2O, and finally counted in the proportional counter.	30 sec	236
					(2) The $PbCO_3$ is dissolved in about 5 cc of hot HNO_3 and about 20 mg of Bi in soln. is added. The soln. is boiled to concentrate the HNO_3, at which point, a large part of the $Pb(NO_3)_2$ precipitates as $Pb(NO_3)_2$. The soln. is transferred into a centrifuge cone containing NaOH to neutralize the HNO_3 and then 2 drops of a 1% soln. of thionalide (thioglycolic acid β-amino naphthalide) are added. This precipitates the Bi as an organic complex which carries the Po with it. The soln. is centrifuged and the liquid poured off. The ppt. is first washed and then completely dissolved in acetone. The acetone soln. is then dropped onto a hot Pt disc to evaporate the liquid. The disc is then flamed and finally placed in the counter.	4 min	
Po		Daughter of At		Extraction	The parent At activity is dissolved in 1-5 ml organic solvent (benzene, CCl_4, di-isopropyl ether, etc.). (1) The soln. is washed with 0.5 ml of 2 N H_2SO_4-0.25 N $FeSO_4$ soln. This keeps At in zero valency state and back-extracts Po into the aqueous phase.	40 min	13

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
					The H_2SO_4-$FeSO_4$ phase is washed twice with di-isopropyl ether and Po is then extracted into 20% TBP-80% dibutyl ether mixture. The organic phase is evaporated on a plate with a heat lamp. (Do not flame plate or the Po will be lost.) Yield = ~ 90%. (2) To the original soln. is added one-tenth volume of 20% TBP-isobutyl ether and back-extracted with 2 M HNO_3-4 M HCl aqueous soln. (Po, Bi, and Pb leave the organic phase and At remains quantitatively in the organic layer.) The Po can then be separated from Bi and Pb by extracting into TBP soln. after destroying the nitrate ion and making the aqueous soln. 6 M HCl.		14, 137
$At^{<202}$ $At^{<203}$ At^{203} At^{204} At^{205}	43 s 1.7 m 7 m 25 m 25 m	Bi(α) Bi(α) Bi(α,10n) Bi(α,9n) Bi(α,8n)	Bismuth metal or oxide	Extraction	The Bi target is dissolved in such manner as to end up with a soln. in conc. HCl to which $FeSO_4$ is added to make sure that At is reduced to the zero state. The At^0 is extracted into di-isopropyl ether. The ether soln. is washed with dilute H_2SO_4 or HCl. (So far as could be told by absence of Po and Bi α activity, the At is pure.) The ether soln. is then evaporated on Pt or stainless steel discs.		58
				Distillation	This method gives At of somewhat uncertain purity but which is considered as adequate for determination of short-lived α emitters. The Bi target is put into a stainless steel crucible fitted on the top with a water-cooled steel finger to which a cooling Pt disc is clamped. When the Bi is kept slightly above its melting point (as measured by a thermocouple fitted into a well in the crucible), within a few seconds At distilled onto the collecting plate. Po does not distill in appreciable quantities until considerably high temperatures are reached.	< 90 sec	14
At^{219}	0.9 m	Daughter of Fr^{223}(21m), from Ac^{227}		Distillation	The purified carrier-free Fr fraction is evaporated onto a Pt filament. A Pt disc is mounted 1-2 mm above this filament, and an electric current is passed	< 30 sec	126

					through the filament to warm it and volatilize the At onto the collector disc. Since Fr, Pb, and Bi will so volatilize if the temperature is sufficiently high, a number of tests were made to determine at what current setting for the particular apparatus the volatilization of At would occur without contaminant volatilization of the others.		
				Extraction	At is extracted from 3 N HCl soln. of the Fr parent soln. into an equal volume of 10% soln. of TBP in butyl ether. After a quick washing of the organic phase with 3 N HCl, the organic phase is evaporated to dryness on a Pt plate. Preparation of Fr^{223}: The parent Fr^{223} is separated from Ac^{227} by a method consisting of pptn. of free silicotungstic acid from a soln. of Ac^{227} in saturated HCl, followed by separation of the Fr from the carrier on a cation exchange resin (Dowex-50).	Several minutes	
Rn^{204} Rn^{206} Rn^{207} Rn^{208}	3 m 6.2 m 10 m 21 m	$Au^{197}(N^{14},xn)$	Gold metal (5 mil foil)	Distillation	The extraction of the emanation is accomplished by amalgamating the Au foil with Hg in a vacuum system. The amalgam is heated with an induction heater to speed up the dissolution of the Au. The heating also causes agitation of the sample. The gases evolved are passed through a U-tube maintained at the temperature of a dry ice-acetone mixture and condensed in another U-tube cooled by liquid nitrogen. Samples for counting are prepared by the glow discharge procedure as outlined by Momyer and Hyde (179).	15-20 min	179, 241
Rn^{212}	23 m	$Th^{232}(p)$	Thorium metal (foil)	Recoil	The target consists of thin Th metal strips sandwiched with thin Al foils to act as catchers for the recoiled atoms during irradiation. After the irradiation, these Al foils are heated at a very low temperature in a vacuum system. A slow stream of Ar carries the emanation through two cold traps at -50°C and into a final trap at -90°C where the emanation is frozen out. From this storage trap, the emanation can be easily transferred to the counting system.		99

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
				Distillation	The emanation activity can be fixed on a Pt plate by the glow discharge method (179).		180
Rn		$Th^{232}(p)$	Thorium metal (foil)	Distillation	The strips are heated to red heat with an induction heater for 1-2 min. Ar carrier gas is then passed through the flask and then through a trap cooled with an ice bath. Finally the Rn is frozen out in another trap cooled with a liquid N_2 bath and then introduced into a sealed counting chamber. Yield = small from metal; up to 50% from soln.	5-15 min	100
Fr^{212}	19.3 m	$Th^{232}(p)$	Thorium metal (foil)	Precipitation	The target foil is dissolved in hot 6 N HCl containing 0.1 N $(NH_4)_2SiF_6$. After the dissolution, 2 mg of Cs carrier is ppted. as Cs-silicotungstate from 6 N HCl, in which Fr is co-ppted. The ppt. is washed with HCl, dissolved in NaOH, and $Fe(OH)_3$ scavenging performed. Finally, the Cs is ppted. as $CsClO_4$ co-ppting. the Fr from anhydrous alcoholic $HClO_4$ soln. It is possible to partially separate the Fr from Cs by passing a 1 N HNO_3 soln. through a short resin columns of colloidal Dowex-50, the Fr coming off at the last of the other alkali elements.		127
				Precipitation	The method involves co-pptn. of Fr with free silicotungstic acid from cold saturated HCl soln. To obtain carrier-free samples of Fr, the silicotungstic acid ppt. is dissolved in water and passed through a Dowex-50 column. The Fr adheres to the column and can later be removed with 6 M HCl.	< 30 min	179
Fr^{221}	4.8 m	Daughter of Ac^{225}(10d)		Precipitation	If the actinium parent is purified carefully, a single pptn. of silicotungstic acid and a single HCl wash are enough to get a nearly quantitative separation of the Fr^{221} from its parent. If a small amount of inert solid matter is tolerated in the final Fr soln., the following procedure can be	10 min	125

					used. The ppt. of the silicotungstic acid which co-ppts. Fr is dissolved in 500 µl of distilled H_2O and stirred with 1 or 2 ml of ether. In the three phase system produced, the bottom layer consists of a liquid compound of silicotungstic acid and ether, the uppermost phase is ether and the intermediate phase is a water soln. of Fr. This method is not as satisfactory as the ion exchange method since a small part of the silicotungstic acid remains in the aqueous phase, but is useful when the most rapid separation is necessary.		
Fr^{223} (AcK)	21 m	Daughter of Ac^{227} (22y)		Solvent extraction	AcK can be extracted into nitrobenzene from an aqueous soln. adjusted to pH 9 and 0.05 <u>M</u> in Na-tetraphenyl boron. 99% of the Fr is extracted. If Ra is present in tracer amounts, 90% of it is also extracted. However, if the Na salt of EDTA is present to a concentration of 1%, the extraction of the Ra is suppressed, while that of the Fr remains quantitative. The authors state that AcK can be separated from all members of the Ac^{227} decay chain with the exception of Ac-C" (Tl^{207}). The AcK can be removed from the nitrobenzene by contacting the organic phase with two volumes of 1 <u>N</u> HCl. In the reported experiments, a chloride soln. containing AcK and AcX was prepared. Na-borate was added until the pH was 9. The soln. was then made 0.05 molar in sodium tetraphenyl boron. (Before it was added this reagent was purified by contact with $Al(OH)_3$ and filtration.) The soln. was then stirred 3 min with an equal volume of nitrobenzene.		189
Ra^{213}	2.7 m	Th(p)	Thorium metal (foil)	Precipitation	To remove Ra from Th targets, the usual method of co-pptn. with $BaCl_2$ is used. The target is dissolved in HCl (with ammonium fluosilicate catalyst added), the soln. is saturated with HCl gas and cooled in ice, and the $BaCl_2$ ppt. is brought down within 5-10 min after the end of bombardment. The pptn. is made from about 10 ml of HCl using about 1 mg of Ba carrier. The emanation daughters of Ra^{213} can be studied by dissolving the $BaCl_2$ and sweeping the emanation activity from the soln.	5-10 min	179

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
		Pb(C^{+6})	Lead metal	Precipitation	The Pb target is dissolved in a minimum amount of hot 2 to 3 M HNO_3, and the soln. is saturated with HCl gas and cooled in ice. In saturated HCl the Pb forms a soluble chloride complex. Ba carrier (a few tenths of a milligram) is then added to precipitate $BaCl_2$ and carry the Ra. With sufficient Ba carrier and time to complete the pptn., one would expect quantitative yields. The pptn. is not instantaneous, however, and $BaCl_2$ is slightly soluble even in saturated HCl. To obtain thin samples for pulse analysis of the α rays in a short time, incomplete pptn. of $BaCl_2$ is tolerated, and yields are sometimes as low as 20%, judging from tracer runs with Ra^{226}.	5-10 min	179
Ra^{222}	38 s	Daughter of Th^{226}(30.9m), from Th(α,6n)U^{230}		Recoil	To get a maximum amount of recoil from a Th^{226} sample, Th is isolated with a minimum of carrier from a soln. containing 100,000 cpm of U^{230} and mounted on a Pt disc. By inserting this disc in a standard α-counting chamber for a few minutes it is possible to introduce several thousand cpm of Ra^{226} into the chamber, because of recoil phenomenon. The Th^{226} sample is then removed and the recoil activity is measured.		245
Ac^{222}	5.5 s	Daughter of Pa^{226}(1.8m), from Th^{232}(d)	Thorium nitrate	Recoil	Pa^{226} is separated rapidly from the $Th(NO_3)_3$ by TTA solvent extraction method (as described in the section on Pa^{226}). The recoil atoms from a thin plate of the Pa fraction are collected in a vacuum in one section of a fast sample changer.	8 sec	176
Ac				Extraction	To the Ac tracer in mixed activities, about 5 mg of pure Lu carrier is added and ppted. as $Lu(OH)_3$ with NaOH at pH > 13. The $Lu(OH)_3$ which co-ppts. Ac is washed with H_2O and dissolved in 5 ml of buffer soln. (0.1 M HOAc and 1 M NaOAc) of pH 5.7.		129

					Then the Ac and Lu are extracted into 30 ml of 0.40 M TTA benzene by 3 min shaking. The organic layer is washed twice with ~ 15 ml of pH 5.7 buffer soln. The Ac (but not the Lu) is back-extracted into ~ 10 ml of pH 4.5 buffer soln. made of 0.1 M formic acid and 1 M Na-formate. This back extraction is repeated. After adjusting the pH of the soln. to about 5.7 with saturated NaOAc, the Ac is extracted once again into a new batch of 0.40 M TTA in benzene. After washing with pH 5.7 buffer soln. and then with water, the Ac is back-extracted twice into a few ml of 0.05 M HCl.		
Th^{234} (UX_1)	24.1 d	Daughter of U^{238}(4.5×10^8y)	Uranyl nitrate	Extraction	To the sample soln. (10 ml) is added sat. $(NH_4)_2CO_3$ and H_2O until the yellow ppt., which first forms, dissolves. Sufficient $(NH_4)_2CO_3$ is added to make the final pH of the soln. 8.0-8.5. By addition of 1-2 ml of 6% aqueous cupferron reagent and 10 ml of $CHCl_3$ to the above aqueous soln., Th^{234} is extracted into the C $CHCl_3$ layer. The extraction prodedure is repeated. The combined $CHCl_3$ extracts are washed with 20 ml of H_2O to which has been added 1 ml of cupferron reagent and sufficient $(NH_4)_2CO_3$ soln. to make the pH 8.0-8.5. Then the Th^{234} is back-extracted into 10 ml of 3 M nitric acid containing a few ml of saturated Br_2 water which serves to decompose the UX_1 cupferrate. This allows extraction of all the organic material and excess bromine into the $CHCl_3$ phase. The aqueous phase is washed twice with $CHCl_3$ and evaporated.		49
Pa^{226}	1.8 m	$Th^{232}(\alpha)$	Thorium metal (5 mil foil) Thorium nitrate	Extraction	A simple extraction with a soln. of 0.4 M TTA in benzene is sufficient to separate the isotope from all other α emitters. The Th metal is dissolved in conc. HNO_3, in which a few drops of 0.2 M ammonium fluosilicate had been added to accelerate the dissolution process. [The $Th(NO_3)_4$ target can be dissolved directly in 4 N HNO_3.] The soln. is diluted to about 4 N acid and extracted with an equal volume of TTA-benzene soln. The phases are separated and an aliquot of the organic layer containing the Pa is evaporated on a Pt plate. Yield = 40-80%.	~ 1 min	175, 173, 172

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Pa^{234m}	1.18 m	Daughter of $Th^{234}(UX_1$, 24.1 d)	Th^{234} in HCl soln.	Extraction	The Th^{234}-HCl soln. is evaporated under an infrared lamp to a volume of 3 ml. This soln. is extracted with TBP to remove traces of U (Pa is also extracted). Thereafter the Th^{234} soln. in aqueous phase is kept in a 10 ml centrifuge tube and the Pa^{234m} daughter is extracted with an equal volume of TBP di-isopropyl ether soln. by shaking vigorously for 2 min and then centrifuging to separate the phases. The organic phase is washed with an equal volume of 6 $\underline{N}$ HCl for 15 sec and then counted.	1-2 min	34
Pa				Extraction	Pa can be extracted from solns. of irradiated Th by di-isobutylcarbinol (DIBC) (2.6-dimethyl-4-heptanol). Pa is extracted from a soln. containing 1.0 $\underline{M}$ Th, 0.6 $\underline{M}$ $Al(NO_3)_3$, and 4.0 $\underline{M}$ HNO_3. Impurities are scrubbed from the organic phase with a soln. containing 0.6 $\underline{M}$ $Al(NO_3)_3$ and 2.0 $\underline{M}$ HNO_3. Feed/organic/scrub volume ratios are 1/0.4/0.75; there are 4 extraction and 5 scrub stages. Pa is stripped from the organic with 1.0 $\underline{M}$ HNO_3 containing 0.05 M fluoride. The extraction is made at 50°C to decrease emulsion formation. Yield = > 99.5%.		204
U^{227}	1.3 m	Th(α,9n)	Thorium nitrate	Extraction	For the measurements of α rays, the extraction of U from 10 $\underline{N}$ NH_4NO_3 or 3 $\underline{M}$ $Mg(NO_3)_2$ (0.1 $\underline{N}$ HNO_3) soln. is found satisfactory.	~ 1 min	175, 173, 176
U^{228}	9.3 m	Th(α,8n)	Thorium metal (foil) or nitrate		After washing the ether with several portions of the nitrate solns., the U is finally back-extracted into the water phase, evaporated on Pt plates and measured. In the case of a $Th(NO_3)_3$ target, the irradiated target is dissolved directly in a mixture of the aqueous medium and the organic extracting agent, so that the stirring of 10 sec for dissolution also serves as the stirring for extraction. For the measurement of short-lived U isotope, the ether phase is rapidly removed from the extraction system and ignited on the Pt plate to leave a weightless U fraction on the plate.	~ 1 min	

U^{235m}	26.5 m	Daughter of Pu^{239}(24,360y)		Extraction	The U is separated by an ether extraction from Pa^{239} soln. reduced with Fe^{+2} and saturated with NH_4NO_3. After several washing steps, the ether containing U is evaporated on a Pt plate.	Few min	122
U^{239}	23.5 m	$U^{238}(n,\gamma)$	Thorium nitrate	Extraction	In the case of Th-nitrate samples, the irradiated sample is dissolved in 10 ml 0.1 N HNO_3 containing 20 mg U carrier. After extraction by 10 ml 0.25 M TTA in benzene, the aqueous phase is adjusted to 2 N HNO_3 and extracted again with TTA. After the extraction and washing of the aqueous phase by an equal volume of benzene, U is ppted. as ammonium diuranate by addition of NH_4OH. The ppt. is centrifuged. The ppt. is dissolved in dil. HNO_3, and 5 ml of 0.1 M EDTA added. The soln. is made neutral for methyl orange with NH_4OH. By addition of 10 ml of 1% oxine-$CHCl_3$ soln., U is extracted as the oxinate. The extraction is repeated. The combined extract is washed with 5 ml 0.02 M $(NH_4)_2$EDTA. The U is back-extracted into dilute acid and ppted. as U-oxinate from NH_4OH-NH_4OAc buffer soln. Yield = 60-80%.	~ 30 min	56
			Biological ash		The irradiated ash sample is dissolved in 4 ml conc. HNO_3 containing 20 mg U carrier and evaporated. The residue is dissolved in small amounts of 2 N HNO_3, 1 g of $Al(NO_3)_3$ added, and U extracted by the ether. The U is back-extracted by H_2O and ppted. as diuranate. The latter procedure is the same as above. Yield = ~ 60%.	~ 30 min	56
			Silicate materials		The irradiated powder sample is dissolved in 1 ml of a soln. of conc. HNO_3, ~ 0.5 ml 40% HF and 20 mg U carrier and evaporated to near dryness. The residue is dissolved in dil. HNO_3 and NH_4OH added. The ppt. is dissolved in 10 ml HCl (2:10) and cooled in ice. After addition of 10 ml 6% cupferron, the cupferrate is extracted with 10 ml $CHCl_3$. The U remaining in the aqueous phase is extracted as the oxinate, as described.above. Yield = 60-80%.	~ 30 min	56

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Np^{232}	13 m	$U^{233}(d,3n)$	Uranium oxide	Precipitation	The U oxide is dissolved by 8 $\underline{N}$ HNO_3 containing a trace of Fe^{+3}. The soln. is adjusted to 1 $\underline{N}$ HNO_3. A few mg of MnO_2 are ppted. from the soln., by the addition of $KMnO_4$ soln. and then of $NaNO_2$ soln. to reduce the permanganate. This MnO_2 pptn. acts as an effective decontamination step for Pa, usually present in large yield. The MnO_2 ppt. is discarded. The nitrite reduces Np^{+6} to Np^{+5}. The Np is co-ppted. with LaF_3, by addition of La^{+3} (1 mg or less) and HF to the soln. The LaF_3 is then metathesized to hydroxide by KOH. The U remains in the supernatant soln. This $La(OH)_3$ containing Np is dissolved by HNO_3 and diluted. H_2SO_4 and $NaBrO_3$ are then added to the soln. to make 1 $\underline{N}$ HNO_3, 1 $\underline{N}$ H_2SO_4, and 0.1 $\underline{M}$ $NaBrO_3$, and the mixture heated. LaF_3 is ppted. from this soln. following the addition of HF to a conc. of about 0.1 $\underline{M}$. The supernatant containing Np^{+6} is then diluted a factor of five by addition of excess SO_2 soln. so that all BrO_3^{-1} is reduced to Br^-. Then Np^{+4} is co-ppted. with LaF_3 from a soln. which has been made 1-3 $\underline{M}$ in HF. This LaF_3 is dissolved in hot saturated $Al(NO_3)_3$ aqueous soln., adjusted to 1 $\underline{M}$ HNO_3, 0.1 $\underline{M}$ $NaBrO_3$ ($Np \rightarrow +6$) and heated for 2-3 min. Solid NH_4NO_3 is then added to 10 $\underline{M}$ concentration. This mixture is extracted by twice its volume of diethyl ether and the ether washed by 10 $\underline{M}$ NH_4NO_3 soln. The ether containing Np is then evaporated on a counting plate.	45 min	166
				Precipitation	The uranium target is dissolved in 6 $\underline{M}$ HNO_3 containing 10^{-3} $\underline{M}$ Ce^{+4}, 0.02 $\underline{M}$ Zr^{+4}, and Np^{+6} tracer. After 1 min heating ($Np \rightarrow +6$), H_3PO_4 is added to the soln. and Zr-phosphate is ppted. To the filtrate containing Np(+6), $N_2H_2 \cdot H_2SO_4$ and Fe^{+2} are added $(N_2H_2 \cdot H_2SO_4 \rightarrow 0.01\ M)$ $(\quad Fe^{+2} \rightarrow 0.005\ M)$ and Np^{+6} is reduced to Np^{+4} . 1 mg Zr^{+4} is added to		166

					this soln. and Zr-phosphate is ppted. The Zr-phosphate containing Np^{+4} is washed with 3 $\underline{N}$ HNO_3 and 0.5 $\underline{M}$ H_2SO_4, and then dissolved in 1 $\underline{N}$ HF^- 1 $\underline{N}$ HNO_3 soln. By the addition of 0.1 mg La^{+3}, LaF_3 is ppt. and Np is co-ppted. in LaF_3.	
Np^{240}	7.3 m	Daughter of U^{240}(14.1h), from $U^{238}(n,\gamma)U^{239}$, $U^{239}(n,\gamma)U^{240}$		Precipitation	After formation of the U complex with NH_2OH, the Np daughter is co-ppted. with LaF_3.	147
Pu			Uranium aqueous soln. (150 mg U/ml) containing tracer concentrations of Cs, Zr, Nb, Ru, and rare earths	Extraction	To 1 ml of the sample soln., 3 ml 2 $\underline{N}$ HNO_3 and 1 ml 1 $\underline{N}$ HCl are added and heated at 80° for 5 min. The volume of the soln. is adjusted to ~ 4 ml by addition of 1 $\underline{N}$ HNO_3. The soln. is transferred to a separatory funnel using 2 ml of 1 $\underline{M}$ $NaNO_2$ and allowed to stand until gas evolution ceases. The aqueous phase at the time of extraction should be about 1 $\underline{N}$ in HNO_3. The soln. is extracted for 10 min. with an equal volume of 0.5 $\underline{M}$ TTA-xylene. The organic phase (Pu) is washed with 1 $\underline{N}$ HNO_3 for 3 min and then the Pu is stripped from the organic phase by mixing thoroughly for 2 min with an equal volume of 10 $\underline{N}$ HNO_3. This nitric acid soln. is washed with an equal volume of 0.5 $\underline{M}$ TTA-xylene to remove the last traces of Zr and Pa activities and centrifuged for 1 min. Yield = ~ 99%.	183
Am^{246}	25 m	Daughter of Pu^{246}(10.85d)		Ion exchange	The purified Pu parent is absorbed on a column of Dowex-1 anion exchange resin which had been previously washed and graded and equilibrated with a $HCl-HNO_3$ mixture (about 30 µl conc. HNO_3 per 5 ml conc. HCl). The Am daughter activity can be milked by passing the $HCl-HNO_3$ mixture through the column.	24
				Extraction	The Am daughter can be milked from the Pu activity by washing the TTA benzene soln. with 0.5 $\underline{N}$ HCl.	74
				Precipitation	The Am daughter is co-ppted. with LaF_3 from a soln. containing Pu parent which have been oxidized to the +6 state.	

Nuclide	Half-Life	Production Reaction	Target	Type of Separation	Procedure for Separation	Time for Separation	Reference
Bk			An aqueous soln. containing lanthanides and actinides	Extraction	The soln. is evaporated to near dryness two or three times with 16 M HNO_3. After the last evaporation, the sample is made up to a soln. 10 M in HNO_3 and 1 M in $KBrO_3$. This soln. is shaken for 3 min with equal volumes of 0.15 M HDEP [di(2-ethylhexyl)orthophosphoric acid]-heptane soln. which has been previously washed with 10 M HNO_3-1 M $KBrO_3$ soln. The organic phase is washed twice with an equal volume of 10 M HNO_3-1 M $KBrO_3$ aqueous soln. The Bk in the organic phase is then back-extracted into an equal volume of freshly prepared 10 M HNO_3-1.5 M H_2O_2 soln. The aqueous phase is washed with 0.15 M HDEP-heptane for 3 min and then with heptane for 3 min. Yield = ~ 97%.		210
Cf^{244}	25 m	$Cm^{244}(\alpha,4n)$	Cm^{244}(95%) containing small amounts of heavier Cm isotopes	Recoil Extraction Ion exchange	The product nuclei from the target are collected on thin Au foils. These foils are dissolved and the bulk of the fission product and induced radioactivity are removed by a combination of solvent-extraction and ion exchange techniques. The actinide fraction is separated into the components by rapid elution from Dowex-50 cation-exchange resin, with α-hydroxy isobutyric acid as the eluent (42).		39
		$Cm^{242}(\alpha,2n)$		Ion exchange	The separation is accomplished through the use of ion exchange method employing the resin Dowex-50.		259
E^{246}	7-7.3 m	$U^{238}(N^{14})$		Precipitation Ion exchange	The method is accomplished by the co-pptn. on LaF_3 ppt. and by the elution method from a Dowex-50 ion exchange column.		101
E^{248}	25 m	$Cf^{249}(d)$	About 10^{13} atoms of Cf^{249} containing 2 x 10^{13} atoms of Bk^{249} parent	Recoil Ion exchange	The recoil products in deutron bombardment are collected on recoil collectors. After bombardment, the Cf and E reaction products are separated by the ion exchange method (255) and electrodeposited on Pt foil.		40

Fm^{250}	30 m	$U(O^{16})$	Uranium metal	Precipitation	The surface layer of the U target is dissolved and carriers are added. The separation of actinide and lanthanides are performed by LaF_3 pptn. and ion exchange methods (244).	11
		$Cf^{248}(\alpha,xn)$	About 10^{13} atoms of Cf^{249} containing 3 x 10^{13} atoms of Bk^{249} parent	Recoil Ion exchange	The recoil products in bombardment of the thin target are caught on a separate Au foil. The chemical purification and separation of the products involve mainly ion exchange techniques and electroplating as described before.	4
Mv^{255}	~ 30 min	$E^{253}(\alpha,n)$	About 10^{9} atoms of E^{253} electrodeposited on the backside of Au foil (2 mil) in beam area	Recoil Ion exchange	From the thin target the nuclear transmutation recoils are ejected in a narrow spray and caught on 0.1 mil Au foil adjacent of the target. The Au foil is quickly dissolved in aqua regia and the Au extracted with ethyl acetate for 1 min. The aqueous phase is then eluted through a Dowex-1 anion resin column (2 mm diameter, 1 cm long) with 6 M HCl to complete the removal of Au and other impurities. The drops containing the actinide fraction are evaporated and the activity is then eluted through a Dowex-50 resin cation column with 0.4 M soln. of ammonium α-hydroxy-isobutyrate, which is adjusted to pH = 4.0 with NH_4OH to separate the various actinide elements from each other. (Mv-Fm-E-Cf)	98

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