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ESOL FACILITY FOR THE GENERATION AND RADIOCHEMICAL SEPARATION OF SHORT HALF-LIFE FISSION PRODUCTS[a]

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ABSTRACT

A facility has been developed at the Idaho National Engineering Laboratory (INEL) for the generation and rapid radiochemical separation of short half-life mixed fission products. This facility, referred to as the Idaho Elemental Separation On Line (ESOL), consists of electroplated sources of spontaneously fissioning ^{252}Cf with a helium jet transport arrangement to continuously deliver short half-life, mixed fission products to the radiochemistry laboratory for rapid, computer controlled, radiochemical separations.

1. INTRODUCTION

Nuclear structure and radiochemical separations have been of long standing interest (>30 years) to the Nuclear Physics personnel located at the Materials Test Reactor Area at the INEL. The study of fission products, because of their relevance to reactor safety, and of the deformed rare-earth radionuclides has been of special interest. When the Materials Test Reactor was permanently shut down in 1970, another source of short half-life fission products and rare-earth radionuclides had to be found. The other materials test reactors at the INEL were designed to be very efficient for materials testing with in-pile loop experiments but they left no flexibility for the installation of rapid irradiation facilities for nuclear structure studies. Hence, a new radionuclide production facility specifically designed for nuclear structure studies was required. The facility selected utilizes electroplated ^{252}Cf spontaneously fissioning sources that can be coupled via a helium-jet transport system to either an on-line elemental separation (ESOL) or an on-line mass separation (ISOL) facility. In this paper we discuss only the ESOL facility. With this facility a number of experiments can be performed, including the measurement of radionuclide half-lives, gamma-ray energies and relative intensities, decay energies (Q_β) and gamma-gamma coincidences. A description of the ISOL system can be found in reference 1.

The helium jet recoil method for the rapid transportation of radionuclei from the production target to remotely located counting instrumentation was first developed in the early 1960's.[2-4] As illustrated in Figure 1, nuclear reaction products escape from the target or source by recoil and become thermalized in a gas-filled chamber surrounding the target or source. If the gas (other gases besides helium may be used with ESOL), which flows continuously through the target/source chamber, is seeded with aerosols,[b] the thermalized radionuclei rapidly attach themselves to the aerosols and are entrained in the gas flow from the target/source chamber through a small diameter capillary. If the gas flow is laminar in the capillary the radionuclei can be transported, via the aerosol carriers, with high efficiency, to a remote laboratory where they can be collected, purified and studied. The gas exits the capillary at sonic velocity into an evacuated

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[a] This work was performed under DOE contract DE-AC07-76ID01570.

[b] Solid aerosols (vs. liquid), and especially NaCl[5] became the aerosol of choice for many facilities employing the gas jet transport technique because of their stability and the ease of generating aerosols of an optimum size for transport (~0.05 to ~1.0 micrometer diameter).

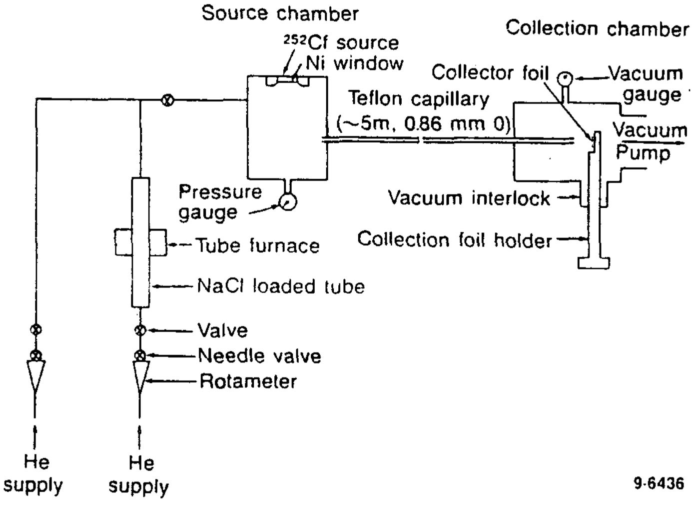

FIG. 1 BLOCK DIAGRAM OF A GAS JET RECOIL TRANSPORT SYSTEM.

collection chamber in the form of a gas jet. The carrier gas is exhausted from the collection chamber by a high volume mechanical vacuum pump, and the relatively massive aerosols emerge from the capillary with a small divergence angle and impact upon a collection surface located a few millimeters from the capillary.

II. DEVELOPMENT OF THE INEL ESOL

The idea to apply the gas-jet technique at the INEL to a spontaneously fissioning ^{252}Cf source was proposed by one of the authors (RCG) in the mid 1970's. Sufficient quantities of ^{252}Cf were available to the DOE laboratories on a loan basis from Oak Ridge National Laboratory (ORNL), and the technique of electrodepositing tens of micrograms of californium with good adhesion onto a platinum backing was already being developed at ORNL for other applications. A special hot cell to accommodate the large ^{252}Cf sources was designed and built[6] in a radiochemical laboratory at the INEL which was already equipped to handle large quantities of actinide materials. This hot cell (see Figure 2) was completed in the late 1970's and within six months of completion the first electroplated ^{252}Cf source (50 micrograms) was received from ORNL. The proposal for this new facility was to couple on-line, via the helium jet transport system, the ^{252}Cf produced fission products to a mass separator. (The mass separator was located in the same laboratory wing and had been used to obtain highly enriched stable and radionuclide target materials for neutron cross section studies.) It soon became clear that the effort required for a ^{252}Cf spontaneously fissioning source to be coupled via a helium-jet recoil transport system to an on-line mass separation system would take several years of research and development. In order to perform nuclear structure studies far from the line of beta stability prior to the completion of the on-line mass separator system, several of the authors proposed that a rapid radiochemical separation facility could be developed in parallel.

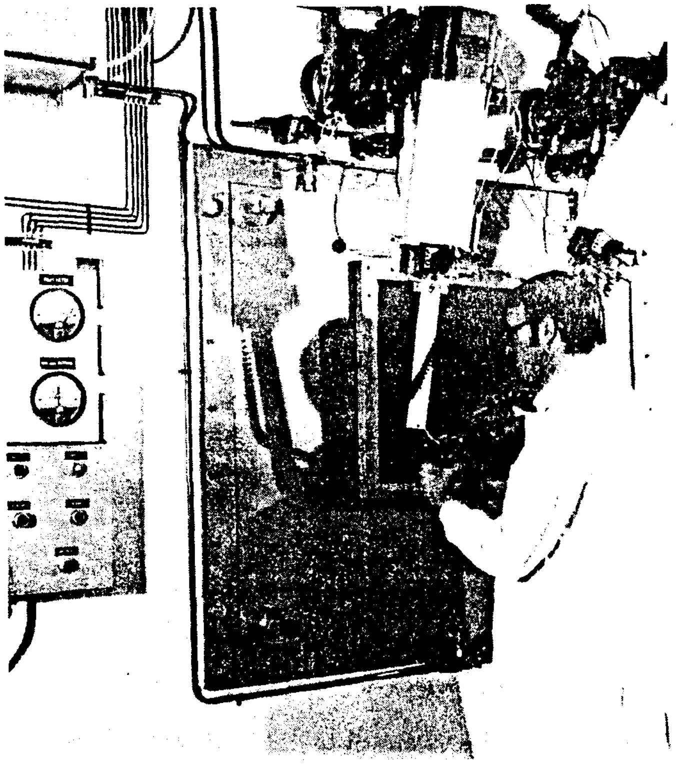

FIG. 2 HOT CELL DESIGNED TO CONTAIN UP TO 2 mg OF ^{252}Cf.

The INEL ESOL facility became operational in early 1979 with a computer-controlled radiochemical separation system, based on HPLC, for the rapid separation of individual rare-earth fission product fractions. Less than one year later the rapid, computer-controlled solvent-solvent extraction radiochemical separation system, based on centrifugal contactors, was in operation. The following timely advances in separation chemistry played an important role in the implementation of a rapid radiochemical separation facility at INEL.

A. Extractants for Removal of Actinides from Processed Spent Fuel Effluent

Development of special solvent extractants for the separation of actinides from commercial light water reactor wastes advanced significantly during the late 1960's and early 1970's.[7,8] These new extractants, unlike the old, allowed the extracted actinides to be back-extracted into an aqueous phase under mild chemical conditions. Our laboratory developed a commercial purification method for the bidentate compound dihexyldiethylcarbamylmethylene phosphate (DHDECMP).[a,9] Because of the chemical similarity of the rare-earths and actinides, DHDECMP can be used to separate within seconds the rare-earth fission products as a group from ^{252}Cf mixed fission products by solvent extraction or extraction chromatography.

B. High Pressure Liquid Chromatography

Advances in high pressure pumps, computer-controlled gradient elution and newly developed anion and cation exchange resins of a 5 to 20 micron particle diameter during the 1960's and 1970's allowed separation of individual rare-earth fission products within minutes by high pressure liquid chromatography (also referred to as high performance liquid chromatography, HPLC).

C. Liquid-Liquid Extractions

Laboratory scale models of plant-size in-stream centrifuges for solvent extraction were developed during the 1970's to allow studies of extraction techniques for radioactive waste processing.[10,11] Laboratory-scale in-stream centrifugal contactors were built for the Idaho ESOL system based on the design developed at Argonne National Laboratory, Argonne, Illinois.[12]

III. ^{252}Cf GAS-JET FISSION PRODUCT GENERATION AND TRANSPORT

Other research groups have also studied short half-life fission products, but have generally carried out these studies by mass or elemental separation using thermal neutron fission in ^{235}U. The facility at the INEL has been designed to complement these other facilities in that the fission products are generated from spontaneously fissioning ^{252}Cf. With this source the yields of fission product radionuclides between ~110 and ~125 atomic mass units (AMU) and above ~145 AMU are enchanced relative to ^{235}U or ^{239}Pu as shown in Figure 3. Direct yield ratios of fission products in the rare-earth region, relative to those of ^{235}U, exceed 1000 for some radionuclei.

A. Fission Product Generation

The ^{252}Cf sources are located in a hot cell (Figure 2) which has been designed to contain up to two milligrams of ^{252}Cf.[6] The interior of the cell is 1.2 m deep x 1.2 m wide x 1.5 m high and lined by a stainless steel box. Between the inner and exterior sides of the cell is a completely enclosed shield structure containing 3.8 cm thick lead sheet and 90 cm thick borated gypsum. The cell has a large 52 cm x 52 cm window providing a view of 80% of the cell interior, two master-slave manipulators and a shielded air-lock door. In addition, it is equipped with service access tubes, internal cell lighting, a HEPA filtered ventilation system and appropriate monitor and control instrumentation.

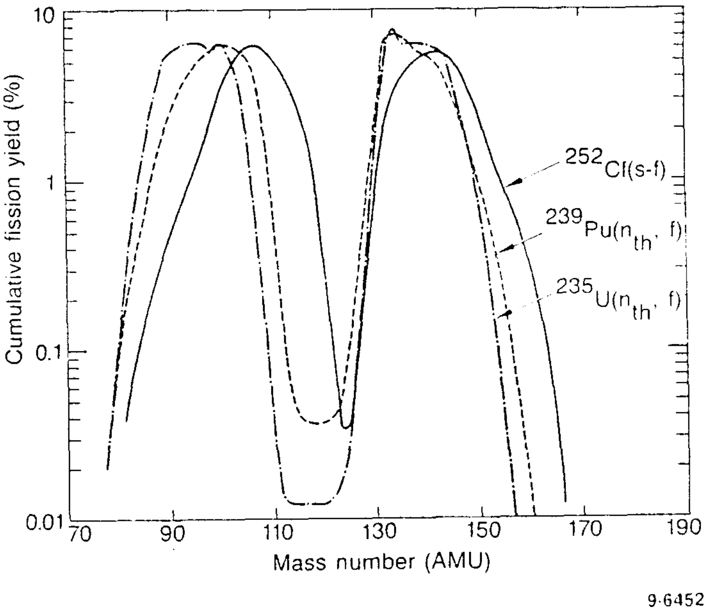

FIG. 3 COMPARISON OF THE CUMULATIVE YIELDS FOR THE SPONTANEOUS FISSION OF ^{252}Cf AND THERMAL NEUTRON FISSION OF ^{235}U AND ^{239}Pu.

The spontaneously fissioning ^{252}Cf sources are electrodeposited as the hydroxide onto platinum disks (~2 cm diameter deposit). They were prepared at ORNL.[13] Each source has a deposit of up to 1000 micrograms of ^{252}Cf. The sources are each mounted in a stainless steel holder and these holders can be screwed into the ends of the cylindrical stainless steel source chamber (8.25 cm x 7.6 cm long). To reduce the possibility of transport of recoiling actinide nuclei from the source chamber to the collection chamber in the chemistry laboratory, a replaceable, thin (1.3 μm) nickel foil window covers each source and is located ~0.6 cm from each ^{252}Cf electrodeposit. The nickel foil is held in place with a retaining ring fastened to the source holder as shown in Figure 4. The recoiling actinide nuclei lack the energy to penetrate the nickel foil and are thereby segregated from the main source chamber. The chamber is pressurized with helium to ~2 atmospheres (2 x 10^5 Pa).

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[a] Dihexyldiethylcarbamylmethelene phosphate (80% purity) obtained from Bray Oil Company, Los Angeles, California, U.S.A. Further purified at the INEL.

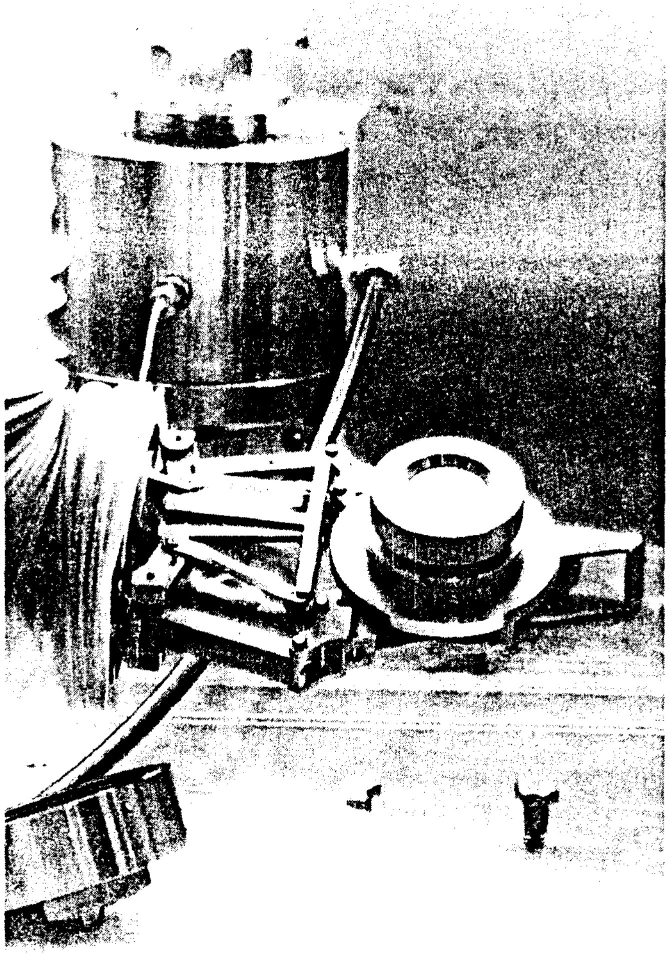

FIG. 4. THE ^{252}Cf SOURCE CHAMBER AND A ^{252}Cf SOURCE HOLDER WITH ATTACHED Ni WINDOW HELD IN PLACE BY AN ANNULAR RETAINING RING.

B. Gas Jet Transport

A NaCl aerosol loaded helium gas stream is used in the ESOL setup. The helium flow rate is ~40 cc/sec at STP. The solid NaCl aerosols are produced by passing helium gas over a NaCl filled ceramic boat that is placed inside a tube furnace operated at 600°C (a variety of other solid aerosols have also been produced using this same technique, e.g., AgCl, Ag, Sm, etc.). By operating the furnace at ~600° a sufficient number of NaCl aerosols are produced to provide high transport efficiencies (~79%) without causing the gas-jet transport capillary to become plugged over extended use.

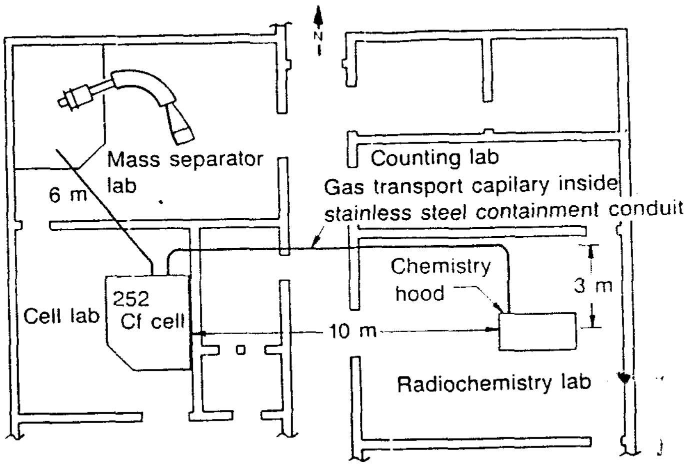

FIG. 5 SCHEMATIC FLOOR PLAN OF THE LABORATORIES CONTAINING THE ^{252}Cf HOT CELL, THE MASS SEPARATOR AND THE CHEMISTRY HOOD.

The teflon capillary, 1.35 mm ID x 25 m long, is contained in a sealed stainless steel conduit that runs from the hot cell to the fume hood in the radiochemistry laboratory as shown in Figure 5. The conduit provides secondary containment of the fission products during transfer between laboratories and protects the fragile capillary from damage. The fission products exit the capillary at sonic velocity into an evacuated collection chamber located inside the fume hood. The carrier gas is exhausted by a large 76 L/S mechanical pump while the relatively heavy aerosols impact and stick to a collection tape. Magnetic computer tape has been found to serve as a convenient and inexpensive collection tape. Figure 6 is a diagram of the collection chamber assembly. The moving tape mechanism allows the transfer of the collected aerosol and fission products from the collection chamber to a dissolution vessel.

C. Dissolution of Aerosols and Attached Fission Products.

At the end of a collection period, the collection tape section containing the aerosol deposit (~3 mm diameter) travels under microprocessor control from the evacuated collection chamber through a sliding vacuum seal to a small dissolution vessel where the activity is washed from the tape. The vacuum seal is simply a piece of thin flexible rubber gasket with slits cut to allow the tape to pass through it. To protect the aerosol deposit during transfer through the sliding seal, a second tape covers the deposit during movement of the collection tape from collection point to dissolution point. In the dissolution vessel the individual layers of tape separate exposing the collected radioactivity to the wash solution as illustrated in Figure 7.

IV. AUTOMATED RADIOCHEMICAL SEPARATIONS

The movement of the collection tape at the end of a collection period and the sequential radiochemical separation steps are computer controlled to assure rapid, accurate and

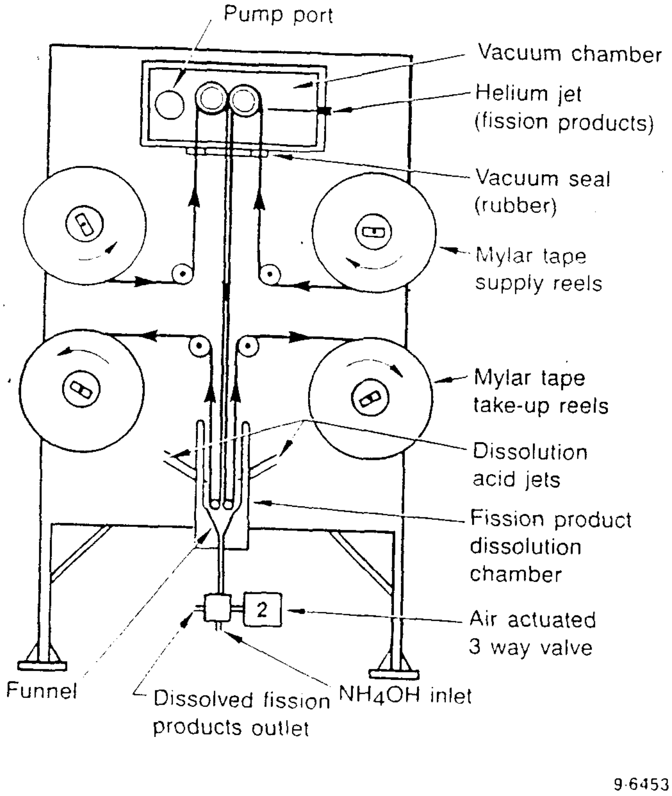

FIG. 6 AUTOMATED FISSION PRODUCT COLLECTION AND DISSOLUTION APPARATUS.

reproducible timing sequences. The microcomputer can be programmed with a set of simple time sequenced commands that automatically control the subsequent chemical separation steps at precise times predetermined relative to the end of the collection period. The Idaho ESOL facility is primarily based on two types of radiochemical separation schemes: 1) column chemistry, and 2) solvent-solvent extractions.

A. Column Chemistry

Columns that can be effectively used in purification schemes include columns to carry out oxidation or reduction, extraction chromatography and anion/cation exchange. Column chemistry is rapid, simple, and elementally specific. High purification factors can be realized so that the fission product element of choice can often be selected with one column.

The system used for rare-earth purifications is illustrated in Figure 8. This system is composed of two HPLC's coupled in series to perform a two-step purification of individual rare-earth elements. Collection times from 3 to 5 minutes were chosen to optimize the yield. The fission products, dissolved in ~1 mL of warm 3.0 M HNO_3, are placed onto an extraction chromatography column using an injection valve. The extraction chromatography column consists of 20 micrometer Vydac C_9 resin[a] which is saturated with DHDECMP. This column, which is slurry packed,[14] is 5 cm long and 3.2 mm inside diameter. A pump capable of operating up to 30 MPa delivers DHDECMP-saturated 3.0 M HNO_3 at a flow rate of 5.0 mL/min to the extraction chromatography column. The rare-earth elements are absorbed on this column while most (~97%) other fission products are washed through within ~1 min under the above conditions. The rare-earth fraction is subsequently eluted by injecting ~2 mL of H_2O onto the extraction chromatography column. A chromatogram of the rare-earth group separation is shown in Figure 9. The yield through this first separation step is ~25% with ~3% contamination from other fission products viz. Tc, Ru, and I). By minimizing the volume and acidity of the separated rare-earth sample, optimized

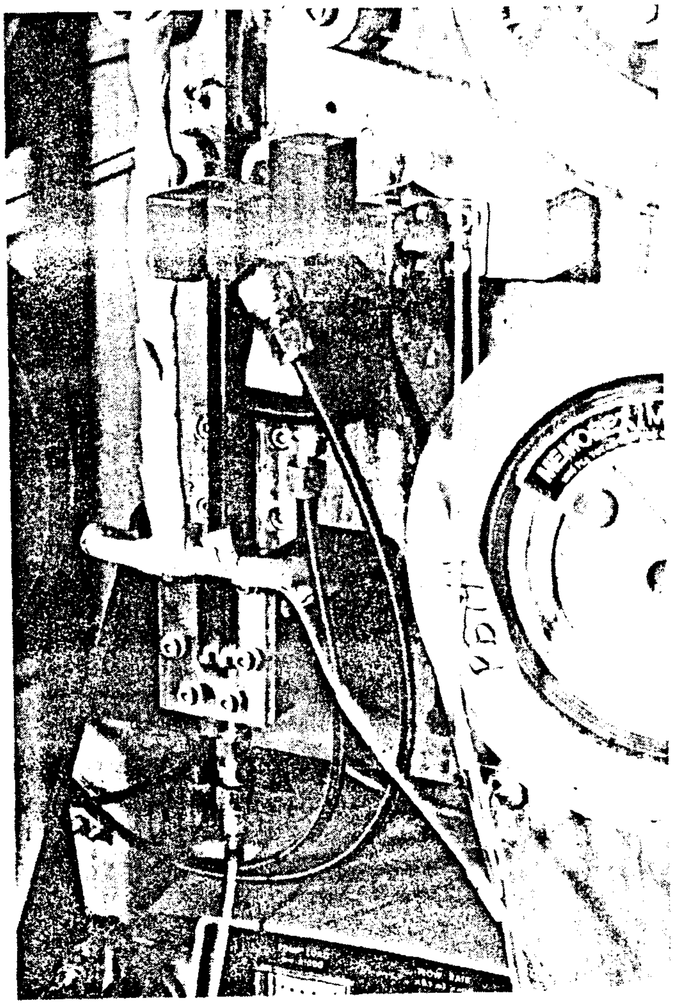

FIG. 7 CLOSE UP OF DISSOLUTION VESSEL FOR DISSOLVING THE AEROSOLS FROM THE COLLECTION SPOT ON THE MAGNETIC TAPE.

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[a] Trademark of Separations Group, Hesperia, California, 92345, U.S.A.

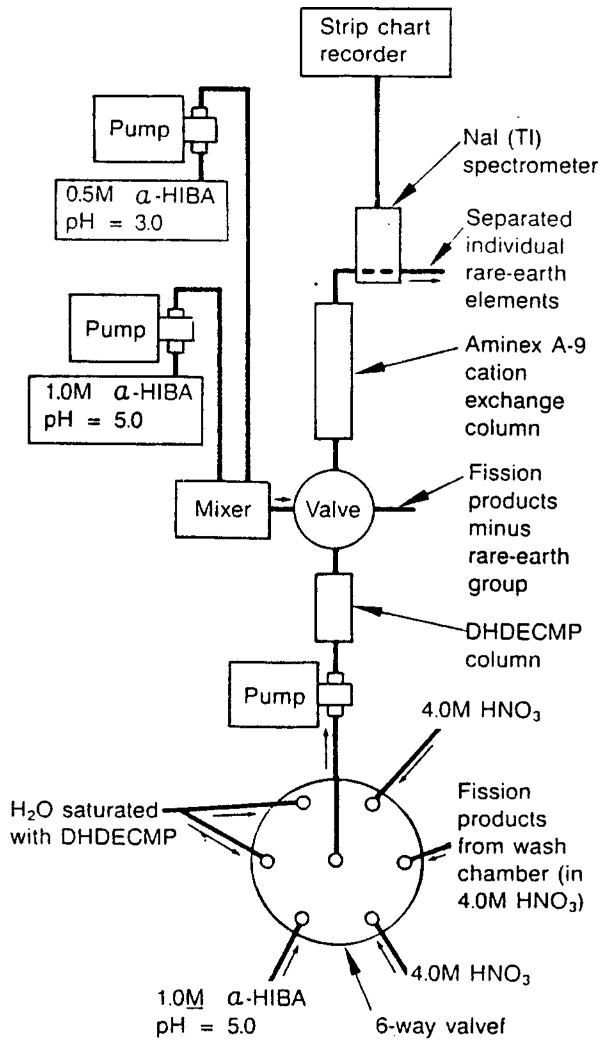

FIG. 8 SCHEMATIC DIAGRAM OF THE RADIOCHEMICAL SEPARATION SYSTEM USED TO SEPARATE INDIVIDUAL RARE-EARTH FISSION PRODUCT ELEMENTS FROM MIXED FISSION PRODUCTS.

resolution of the individual rare-earth elements can be achieved in the second separation step.

Specific separation of the individual rare-earth elements is accomplished by cation exchange. The output from the DHDECMP column passes through a pneumatically operated stream-splitting sample-injection valve which is connected to a cation-exchange column. The rare-earth fraction from the DHDECMP column is captured in a sample loop in the injection valve and is sequentially injected onto the cation-exchange column where the individual rare-earth elements are separated. The cation-exchange column is a 25 cm long x 3.2 mm inside diameter stainless steel tube which is slurry packed[14] with 11.5 micrometer Aminex A-9 cation exchange resin.[a] By elevating the temperature of the Aminex A-9 column from 17°C (ambient) to 90°C with a thermostatically-controlled jacket the resolution between the individual rare-earth elements is increased by ~50%.

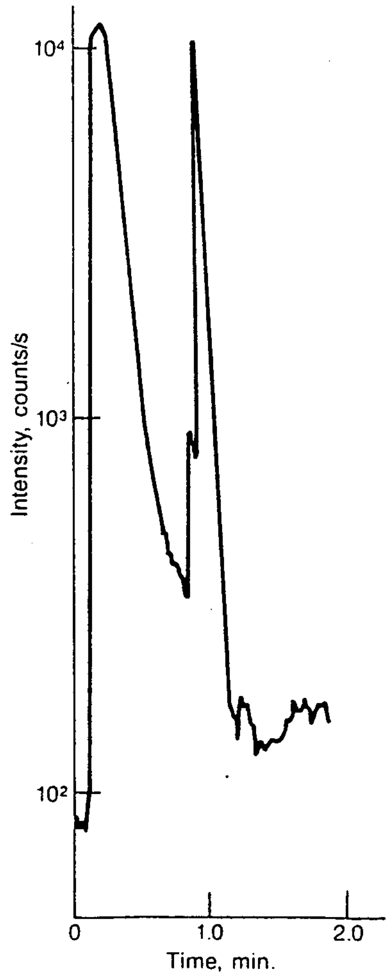

FIG. 9 CHROMATOGRAM OF GROUP RARE-EARTH SEPARATION FROM MIXED FISSION PRODUCTS. TIME ZERO CORRESPONDS TO THE END OF FISSION PRODUCT COLLECTION.

Two high-pressure pumps, capable of operating up to 80 MPa, are used under microprocessor control to deliver alpha hydroxisobutyric acid (α-HIBA) of varying concentrations and pH to the cation-exchange column. The gradient starts at pH 3.2, 0.7 M α-HIBA, which allows the rare-earth elements to adhere to the column, and smoothly increases within seven minutes to pH 5.0, 1.0M α-HIBA thereby eluting the rare-earth elements in

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[a] Trademark of Bio-Rad Laboratories, Richmond, California, 94894, U.S.A.

inverse order of atomic number (Z) as shown in Figure 10. The yield through this column is estimated to be >90%. Thus the yield through the entire system is >22%.

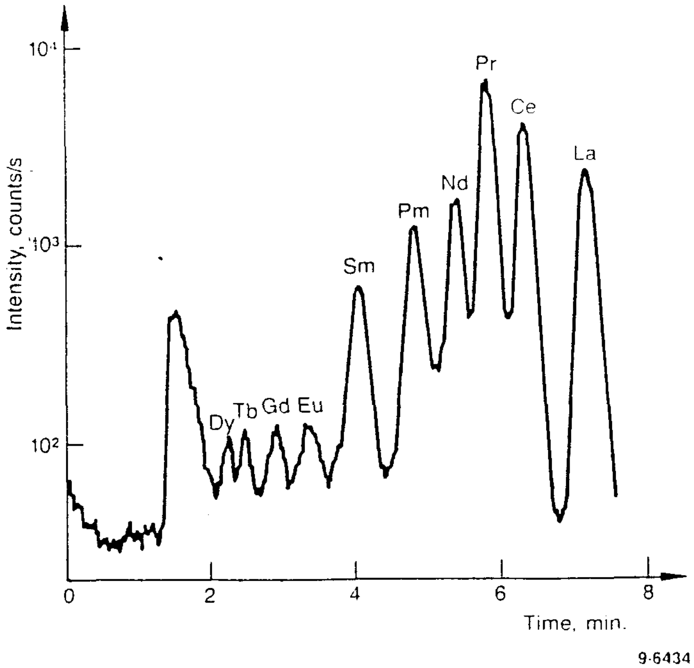

FIG. 10 CHROMATOGRAM OF A SEPARATION OF INDIVIDUAL FISSION-PRODUCT RARE-EARTH ELEMENT PRODUCED BY THE FISSION OF ^{252}Cf. THE TIME SCALE CORRESPONDS TO THE END OF THE FISSION PRODUCT COLLECTION PERIOD AND INCLUDES ALL OF THE CHEMICAL SEPARATION STEPS.

As they are eluted from the column, the individual rare-earth elements are monitored with a shielded 3.81 cm diameter x 3.81 cm thick NaI(Tl) detector which has a 4.76 mm diameter hole drilled diametrically through it for in-stream use. The output of this NaI(Tl) detector is connected via a count-rate meter to a strip chart recorder. The rare-earth fraction of interest contained in ~0.5 mL can either be collected manually or directed through capillary tubing to the detector system by a computer controlled valve. Data acquisition is controlled by the data acquisition computer and is automatically initiated when it receives a signal from the microprocessor that controls the on-line chemistry.

B. Solvent-Solvent Extraction

The solvent-solvent extraction system is based on the use of in-stream laboratory-scale mixer centrifuges called centrifugal contactors. A block diagram of a centrifugal contactor is shown in Figure 11. It has an effective volume of ~12 mL with ~4 mL in the mixer stage and ~8 mL in the separation stage. The centrifugal contactors are each individually driven by 1/20 H.P. transistorized motors (0 to 5000 rpm) which offer precise (±1%) speed control even with changing load and line voltage conditions. The time required for one separation stage (i.e., to mix and separate phases) is ~10 s for a two-phase flow rate of 80 mL/min. The weir, whose dimensions control the position of the interface of the two phases, is fixed; however, the weir dimensions are satisfactory as long as the ratio of the densities of the lighter to heavier phase is between ~0.7 to ~0.9. At optimum phase flow rates and contactor rotor speed the purity of each output phase is >99%. To permit some flexibility in the use of various mineral acids and organic solvents three centrifugal contactors were constructed from 304L stainless steel and three were constructed from C-276 Hastalloy.

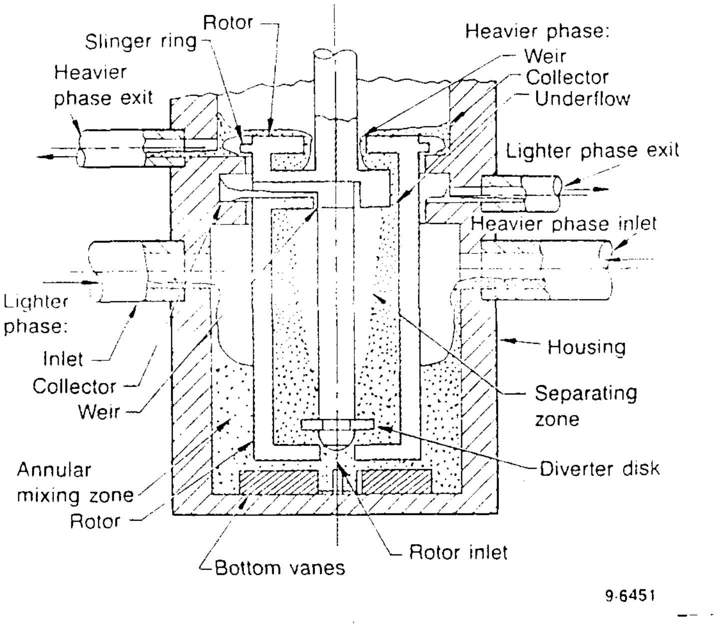

FIG. 11 CROSS-SECTION VIEW OF A CENTRIFUGAL CONTACTOR SHOWING THE INLET AND OUTLET PORTS AND THE MIXING AND SEPARATION CHAMBERS.

The chemical separation scheme used for the purification of fission product palladium (see Figure 12) will be described as an example of the use of three centrifugal contactors used in series to extract, strip and back-extract an element of interest. It is based on the method of Alexander, Schindewolf and Coryell.[15] After a two-minute collection the aerosol deposit is moved to the dissolution chamber where the fission products are dissolved in a warm (70°C) solution of 8 M HNO_3-0.1 M HCl containing 0.1 mg/mL of Pd, Ru, Rh, and Te carriers. Upon entering the complexation vessel the solution is adjusted to a pH ~1 with 1 M NH_4OH. The Pd is then complexed by adding several mL of 1% dimethylglyoxime $(DMG)_2$ in methanol to form

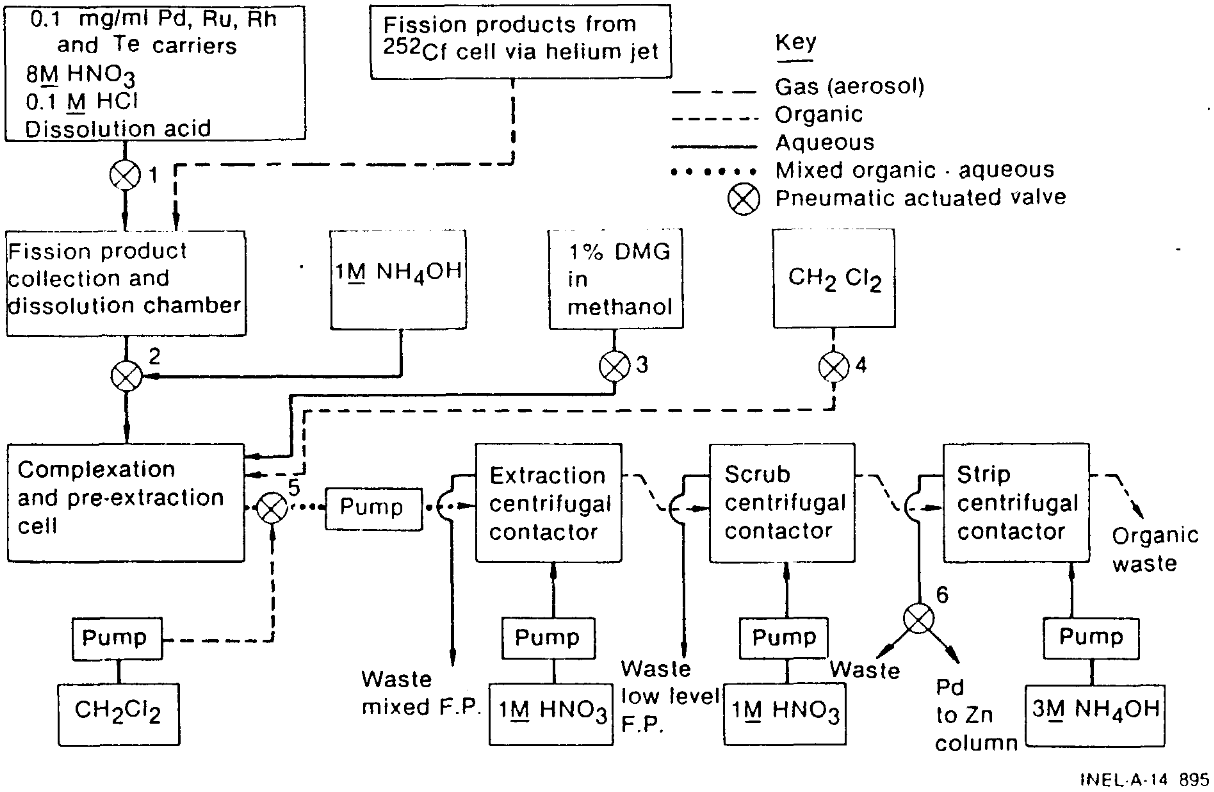

FIG. 12 CHEMICAL SEPARATION SCHEME WITH ELAPSED TIME FROM THE END OF FISSION PRODUCT COLLECTION INDICATED AT THE VARIOUS CHEMICAL STEPS.

palladium dimethylglyoxime complex [Pd(DMG)$_2$]. This complex is very specific to nickel and Pd and at pH ~1 forms a precipitate. Vigorous stirring of the mixture and the addition of 15 mL of dichloromethane (CH_2Cl_2) takes the Pd(DMG)$_2$ up into solution and prevents the plateout of the precipitate onto the container walls. The mixture is then pumped directly into the first contactor.

The first centrifugal contactor completes the extraction of the Pd complex from the fission product solution. The Pd(DMG)$_2$ CH_2Cl_2 extract from the outlet of the first contactor is gravity fed to the second contactor where it is scrubbed by 1 M HNO_3. In the third contactor the Pd(DMG)$_2$ complex is stripped into 3 M NH_4OH to separate it from the radioiodine which remains in the organic phase. Following the output of the strip contactor, three different types of radiochemical isolation of the Pd and/or Ag (daughters of short half-life palladium fission products) can be employed for the purpose of distinguishing the parent Pd from the daughter Ag radioactivities.

1. Collection of Palladium and Silver. The Pd and Ag radionuclides are both reduced by passing the 3 M NH_4OH strip outlet solution through a 30 mesh zinc column measuring 0.6 cm dia. x 3.2 cm long. The separation time, from the end of the fission product collection to the start of the first count, is 135 s.

2. Collection of Silver. In this experiment the Ag daughters are isolated by isotopic exchange on a preformed AgCl column (0.6 dia. x 3.2 cm long). The aqueous outlet strip solution is collected in a vessel and acidified with 8 M HNO_3 before introduction to the AgCl column (see Figure 13). The Ag is collected on this column before the Pd(DMG)$_2$ begins to form a precipitate. The Ag daughters which had accumulated since the strip stage (third contactor) are thus trapped on the column with the Pd parent activities passing through the AgCl column to waste. The total separation time is 165 s.

3. Collection of Palladium with Silver Initially Removed. The Pd(DMG)$_2$ precipitate is reformed in this experiment by acidifying the outlet strip solution in a collection vessel to pH ~1. The concentration of Pd carrier used for this separation was doubled to provide ample precipitate. After the precipitate formed it was collected by vacuum filtration on a column of glass wool (see Figure 13). The Ag activities which formed while the Pd(DMG)$_2$ was in solution were washed through the column. However, any

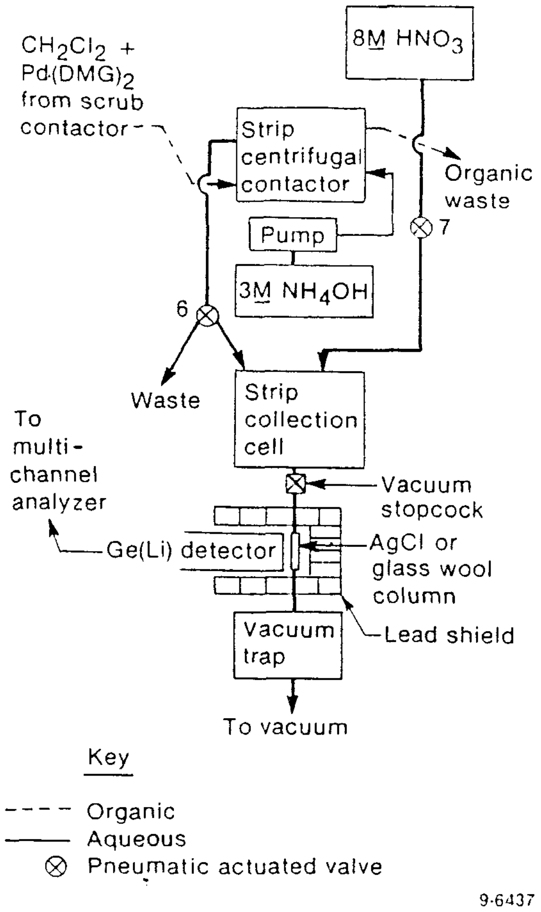

FIG. 13 ISOLATION SCHEME FOR AgCl ISOTOPIC EXCHANGE AND $Pd(DMG)_2$ PRECIPITATION EXPERIMENTS.

Ag activities formed after the $Pd(DMG)_2$ precipitate was produced were physically bound by the precipitate, permitting the observation of the growth and decay of the Ag daughter activities. This isolation required 150 s.

V. DATA ACQUISITION AND ANALYSIS

Following each radiochemical isolation method, sources are prepared or directed to the detector and counted with a coaxial Ge(Li) spectrometer by spectral time multiscaling (i.e., acquisitions of a set of spectra sequenced in time). As soon as the counting sequence of one source is complete, another source is prepared and counted. However, instead of adding these spectra to the respective multiscaled spectra previously accumulated, the multiscaled spectra are all individually stored on a 43 megaword (12 bit words) disk. At the end of each experiment, the individual spectra are examined for the presence of contaminants or other problems that would invalidate the data. The respective spectra, that are acceptable, are then combined to produce a set of spectra sequenced in time with improved statistics. These spectra are subsequently analyzed in the normal manner for gamma-ray energies, emission rate and radionuclide assignment.

The 4096 x 4096 channel coincidence measurements are made with two Ge(Li) spectrometers accumulating both singles data as well as coincidence information. The system is based on a slow-fast coincidence circuit using zero crossover timing with the data stored event-by-event on disk or magnetic tape in the list mode. After the experiment the accumulated channel pairs are sorted and spectra in coincidence with particular energy gates are examined in order to reconstruct the level scheme. The decay schemes of ^{157}Sm, ^{158}Sm, ^{152}Pm and ^{154}Pm have been studied with this system.

VI. SUMMARY

In this paper we have described a novel radiochemical separation facility for the study of short half-life fission products. The system has been operating reliably and effectively during the time required for many experiments. The He-jet system has operated reliably for periods of one year with essentially no maintenance. The collection chamber with moving tape and the in-stream switching valves have seldom malfunctioned.

To date, the INEL ESOL system has been used to identify five new rare-earth radionuclides[16,17] with half-lives as short as 42 s and has been used to provide separated sources for decay scheme studies of several rare-earth and other fission product radionuclides (e.g., $^{113-115}$Pd). In 1986 the INEL ISOL system became operational and within a three-year period has identified seven additional new rare-earth radionuclides[18] with half-lives as short as ~4 s.

In conclusion there are many advantages to the present ESOL ^{252}Cf fission product generation system for studies of short-lived fission products: 1) it is less expensive to operate than alternate fission product generators (i.e., nuclear reactors), and 2) the ^{252}Cf fission product yield curve has a narrower valley between peak yields and significant enhancement in the yields of the heavier rare-earth elements (see Figure 2). As a result the ESOL facility provides an opportunity to study those radionuclides that are not easily observed from ^{235}U or ^{239}Pu fission.

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