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UC-23

PACIFIC NORTHWEST LABORATORY
DIVISION OF ISOTOPE
DEVELOPMENT PROGRAMS
QUARTERLY REPORT: AUGUST-OCTOBER 1970

November 1970

AEC RESEARCH &
DEVELOPMENT REPORT

BNWL-1308-4

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Radiation Applications

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DIVISION OF ISOTOPE DEVELOPMENT PROGRAMS
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INTRODUCTION

This report, BNWL-1308-4, is the 16th report of a series initiated in 1966 to provide detailed and timely information on the results of the isotopic fuels program and related research being conducted at the Pacific Northwest Laboratory by Battelle-Northwest for the U.S. Atomic Energy Commission. The previous reports of the series are BNWL-385, 466, 570, 680, 681, 781, 877, 924, 1010, 1085, 1177, 1256, 1308-1, 1308-2, and 1308-3.

This report covers the three-month period, August through October 1970.

SUMMARYENCAPSULATED WASTE MANAGEMENT CESIUM AS A PROCESS
RADIATION SOURCE

A status report on cesium studies has been written and will be published in the following quarter. A preliminary study was completed that shows the need for in-depth study of incentives for early involvement of private funding in encapsulation of HWMP cesium. The present capability for cost-effective densification of strontium sources was studied. Investigations continue on the cost of higher density SrF_2 , on properties data on SrF_2 compounded with other metal halides which pertain to the SrF_2 packing studies, and on the effects of possible impurities in SrF_2 .

RADIATION CHARACTERISTICS OF FISSION PRODUCT RHODIUM
AND PALLADIUM

The $^{102\text{m}}\text{Rh}$ decay measurements have resumed after moving the counting equipment to a new laboratory. First efforts have been expended in the program to evaluate the isotopic composition of fission product rhodium from Pressurized Water Reactors.

EVALUATION OF LARGE SCALE USES FOR PURIFIED FISSION
PRODUCT XENON

Very satisfactory levels of residual ^{85}Kr activity have been obtained in the first volume of crude fission product xenon purified in the equipment previously used in the ORNL ^{85}Kr campaign. With the remaining impurities easily removable, the purified xenon should be acceptable for large scale usage. Efforts to establish specifications for experimental use of purified fission product xenon have begun.

ISOTOPE PRODUCTION EVALUATIONS

The computer program CAESR was used to identify the market conditions that will result in recovery and sale of fission product cesium and strontium. Development of an isotope data system was initiated which will provide (1) quick response to queries on isotope properties and (2) a file of basic data to be used for other purposes such as shielding requirements, dose rates and decay compositions. The "nuclide tables" data file was completely rechecked and revised with updated data.

NONDESTRUCTIVE TESTING FOR IRRADIATION SOURCES

Design and construction of a differential transducer used to resolve small anomalies, which are too close to the weld or cladding surface or in materials too thin for resolution by a single transducer, continue. The use of the multielectrode transducer system that was developed to rapidly inspect plate-type sources was demonstrated on nonactivated ^{60}Co and $\text{SrSiO}_3\text{-Al}$ matrix sources. Reporting of the information developed on this program continued with preparations of Battelle research reports, open literature articles and society presentations.

ADVANCED SOURCE DEVELOPMENT

The glove box sputtering system was moved to a larger room and a number of general system improvements were made. A new sputtering system, which was ordered in the previous quarter, was delivered and installed in the same room. Source preparation hardware for sources with a 1/2-inch diameter active area and for planar ring sources was fabricated. Techniques for depositing a material in the glovebox sputtering system without adding radioisotopic cross-contamination to the substrate or the deposited layer were developed and

demonstrated. Areas where PNL prepared sources can be improved were identified as a result of source testing at ORNL, and improvements in these areas are being incorporated into source preparation efforts. The apparent susceptibility of sputtered 304 stainless steel layers to rusting when exposed to boiling water or heated high-humidity air for extended periods of time was investigated experimentally. A paper, "Thin Film Radiation Sources Using RF Sputtering Techniques" was presented at the 1970 TMS fall meeting in Cleveland, Ohio.

RADIATION CHARACTERISTICS OF CIRCULATORY SUPPORT ISOTOPIC HEAT SOURCES

The organ geometries developed by Snyder, Fisher and Warner were used to calculate photon dose rates to various organ systems from an implanted 30-W ^{238}Pu heat source placed on the inside surface of the left ilium.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

A 1.8 mg ^{252}Cf source was received from ORNL early in the quarter, and flux characterization and calibration of this source continue. The source is used in the ^{252}Cf irradiation facility where calibrations are in progress to determine response, characteristics, and detection sensitivity for some 40 elements in simulated seabed materials.

ENCAPSULATED WASTE MANAGEMENT CESIUM
AS A PROCESS RADIATION SOURCE

H. H. Van Tuyl, H. C. Stidham, and W. E. Keder

The objective of this program is to provide technology for the production of inexpensive cesium and strontium sources by using the purified compounds generated in the Hanford Waste Management Program (HWMP). Large scale use of these radioisotopes will require an assessment of the capabilities of HWMP for producing suitable radiation and heat sources, acceptance of the HWMP product by the customer, licensing of the sources, and market development activities.

The goal in FY-1971 is to develop equipment for fabricating smaller diameter sources in the HWMP facility. The incremental cost of producing smaller sources will be evaluated. Prototype sources will be fabricated with nonradioactive feeds, including loading capsules, welding, and NDT. The sources will be tested to determine mechanical integrity. Potential customers will be contacted to stimulate interest in the HWMP sources.

A status report on cesium studies has been written and is being circulated for comments before issuing. The conclusions from the report are somewhat tentative since the price of HWMP cesium has not been defined. Assuming a price of \$0.10 to \$0.15/Ci for encapsulated cesium, there is a definite incentive to reduce HWMP container diameter and capsule wall thickness.

A preliminary evaluation was made of the incentives for a private party to obtain cesium from HWMP and encapsulate the material in different container configurations. Amortization of facilities and equipment is expected to be on the

order of \$150,000 per year, and operating costs will be about \$300 per final source container. At 5 MCi of cesium per year with 15 kCi of cesium per container, the added cost will be about \$0.05/Ci. Under these conditions, the cost of fabrication would just about equal the value derived from increased utilization of cesium due to reduced self-absorption and absorption in the container, if the cesium from HWMP sold for \$0.125/Ci. At lower cesium prices, the additional cost of fabrication could not be recovered, while at higher cesium prices the market for cesium would be reduced due to competition from ^{60}Co .

This analysis is quite preliminary, and does show the need for a more detailed study of the incentive for early involvement of private funding for encapsulation. The incentive will be highly dependent on assumed sales since amortization of facilities and equipment is a major part of the cost.

Most of the recent effort has been devoted to strontium. Currently, any alternative source geometry is limited by the present compactor design; it was designed to handle only waste management capsules. The compactor can be adjusted, however, to handle smaller than standard size capsule configurations if the appropriate shims, plugs, compactor rod and adaptors are fabricated. Loading a capsule that is larger in diameter than the standard size capsule will require redesign of the funnel mechanism, base support and compactor rod. With the larger capsule diameters, the compaction density will decrease to approximately 70% of theoretical density at a 4-inch diameter. A compaction density of 80% of theoretical density is achieved with the standard sized capsules. Estimates are that the maximum possible density that can be achieved in the standard sized capsule is limited to 85% of theoretical density.

Rough estimates were obtained of the cost of building flexibility into the compactor design. The new compactor will cost in the neighborhood of \$15,000. If the compactor is redesigned at this time to accept diameters larger than the waste management capsules, each set of shims, adaptors and compactor rods will cost approximately \$1,500 for each capsule size. If the existing compactor design is used, a second compactor unit will be required for capsules of larger than the standard sized diameter.

With the same diameter, capsule length can be changed more easily, but some loss of density may result from increased lengths. Some additional accessory equipment will be required, but no major redesign is anticipated.

The standard sized container of Haynes 25 metal was originally estimated by ARHCO at \$235 per complete capsule unit. A revised estimate by ARHCO is over \$400 per capsule, since Haynes 25 metal tubing now costs somewhere around \$100 per linear foot.

The centerline temperatures and outer skin temperatures for capsules of varying diameters and densities have been calculated. Calculations were made for capsules with densities that varied in 5% increments between 65% theoretical density and 90% theoretical density. The inner capsule diameter was varied in 1/5-inch increments between 2 and 4 inches. Computations were based on using Hastelloy C metal, a wall thickness of 120 mils, and a separation of 130 mils between the inner and outer capsule. This large spacing between capsules is a "worst case," with the expected gap being considerably smaller. All calculations used a 12-inch inner capsule length. The thermal conductivity of Hastelloy C is very similar to that of the Haynes 25 metal, and the capsule is expected to be fabricated from one of these materials.

The centerline temperature of the capsule in air will vary from about 800 to 1750 °C for 65% of theoretical density at diameters from 2 to 4 inches. Higher densities will result in lower centerline temperatures. Additional calculations are being made for other capsule geometries and environments.

The cost for higher density strontium fluoride is under investigation. The current compactor design has achieved slightly over 80% theoretical density using the waste management capsule. The current compactor design will handle only the standard waste management capsule unit and will require extensive modifications and redesign for handling alternative source sizes.

Phase diagrams of mixtures of strontium fluoride with other selected metal halides have been studied. Only a few of the diagrams that are pertinent to the SrF_2 packaging studies appear in the literature, but these may be instructive. Where SrF_2 data are not available other alkali metal halide work has been reviewed.

It appears that BaF_2 forms an unbroken series of solid solutions with both CaF_2 and SrF_2 . The minimum melting points of these are reported to be only 50° and 170° lower respectively than the melting point (1320 °C) of BaF_2 itself. One would estimate that CaF_2 and SrF_2 would form a similar series of solid solutions with each other; in which case, one would expect no melting below about 1200 °C.

SrF_2 forms an equimolar compound with SrCl_2 and this compound forms solid solutions with SrF_2 itself and with SrCl_2 . The low SrCl_2 range, which might be of some interest in the present study, hasn't been studied, however. Of more particular interest, SrCl_2 and SrF_2 both form eutectic mixtures with strontium phosphate at concentrations of the latter below 10%. Strontium chloride (mp, 830 °C) also forms a eutectic with SrSO_4 (mp, 762 °C, 14% SrSO_4).

Possible sodium fluoride impurity in the SrF_2 product is also worth considering. Sodium fluoride (mp, 995 °C) forms eutectics with CaF_2 (mp, 1410 °C), SrF_2 (mp, 1400 °C), and BaF_2 (mp, 1320 °C); none of these equimolar eutectics is thought to melt above 800 to 850 °C. The presence of a small amount of NaF-SrF_2 eutectic is a possible explanation of melting reported in encapsulated SrF_2 at temperatures below 900 °C.

Little appears to be known about ZrF_4 systems, particularly ternary and quaternary systems at low ZrF_4 concentrations. There are examples of complex zirconium fluoride-metal fluoride mixtures which melt below 400 °C. Generally, phase diagrams of tetravalent metal salts are very complex because of multiple compound formation. The presence of zirconium metal in the system of interest here may or may not be a significant complexity, and it is by no means certain that the fission product zirconium can be simulated properly by bulk metal.

RADIATION CHARACTERISTICS OF FISSION PRODUCT
RHODIUM AND PALLADIUM

F. P. Roberts

The traditional view of fission products is that they are waste materials, but some fission products, such as Rh, Pd, Ru, and Tc, have or will have future uses and should be considered for their resource potential. A resource strategy should be developed to determine the optimum technological and economic use of these elements. This study would answer these questions:

- 1. What is the market for Rh, Pd, Ru, and Tc for the next 40 years?*
- 2. What are the radiological hazards for the use of these elements on a benefit-risk basis?*
- 3. Is there justification for a national policy requiring that these elements be set aside as a national resources stock-pile for future use?*
- 4. What would be the impact of an almost irreversible storage of these elements on the economics of recovery if they are needed in the future?*
- 5. Is it economically desirable to perform a gross separation scheme that will separate these elements and store them for 20 to 50 years so that they will be available for future use?*

^{102m}Rh HALF-LIFE

The counting equipment used to measure the decay of ^{102m}Rh in fission product rhodium was transferred to a different laboratory. The new laboratory, however, has not yet been equipped with temperature and humidity control equipment, and problems have resulted. Several components including the linear amplifier, failed because of large ambient temperature excursions. The problem was temporarily solved by increasing air flow through the laboratory to stabilize the ambient temperature, and the equipment was repaired and recalibrated. Measurements taken to acquire data for the half-life determination have resumed. Figure 1 shows the decay measurement data obtained thus far.

FIGURE 1. Decay of ^{102m}Rh

ISOTOPIC COMPOSITION OF FISSION PRODUCT RHODIUM

The purpose of this effort is to determine the range of ^{102m}Rh activity per gram of rhodium to be expected from spent Pressurized Water Reactor (PWR) fuel. A spent fuel subassembly from Yankee No. 1 reactor has been obtained, and samples have been taken from selected rods. These samples will provide fission product rhodium specimens from a range of burnup and permit a coarse evaluation of the isotopic composition as a function of reactor operating parameters.

Efforts are now underway to isolate and purify milligram quantities of rhodium from these samples.

EVALUATION OF LARGE SCALE USES FOR PURIFIED
FISSION PRODUCT XENON

C. A. Rohrmann

Of all the fission products, xenon has the highest fission yield. Although many of the xenon isotopes are radioactive, they all have such short half lives that their activity would be insignificant after allowing time for normal fuel reprocessing.

In the past, the extreme rarity of xenon in nature has clearly inhibited its consideration for specific large scale uses even where its properties give it definite and unique advantages. But now, xenon could be made available in surprisingly large quantities. Perhaps the most important and beneficial use of xenon would be for human surgical anesthesia. Another of the potential uses is a filler gas in incandescent light bulbs with significant increase in both brightness and efficiency.

The scope of the PNL program is to:

(1) establish the quality of xenon which can be accepted or generally licensed for consumer uses, (2) determine what industries would be interested in evaluating fission product xenon for large scale application, (3) distribute specimens to participating evaluators, (4) compile evaluation results, and (5) prepare a final report on the results of this total effort.

After the conclusion of the ^{85}Kr campaign at ORNL, the equipment was decontaminated and prepared for use in purifying the stock of crude fission product xenon. The first volume (300 liters) of suitable purified material was produced. Working toward a goal quality of 1 to 10 μCi of residual ^{85}Kr activity per liter of xenon, STP, a level of 2 μCi was obtained--a very satisfactory result. In addition, the gas had a xenon content of nearly 99% with most of the remaining impurity being CO_2 at 1.0%, which, if necessary, should be

easy to remove. The gas then should be about 99.9% xenon and would contain less than 0.0001% ^{85}Kr by volume. It is believed that such quality should be acceptable for large scale uses including use for human anesthesia. For the latter application to cause a dose equivalent to that obtained in a typical chest X-ray, the patient would have to be anesthetized for about 150 hours. Calculations for such comparisons are being refined.

Contacts with the Food and Drug Administration and the AEC Division of Materials Licensing are being made to establish the requirements for the experimental application of fission product xenon of the quality obtained.

ISOTOPE PRODUCTION EVALUATIONS

R. W. McKee, B. M. Cole and R. G. Rau

This program is designed to evaluate major aspects of isotope production. Until recently, the principal source of isotopes has been the AEC production reactors. Now, however, power reactors are rapidly becoming important sources of isotopes. Power reactors also offer attractive prospects for target irradiations. The future availability and cost of these unique isotopes depend on full and efficient utilization of the production potential of power reactors, and the inherent value of these materials can have a significant impact on nuclear power costs.

The scope of this program includes analysis of fast and thermal reactor fuel cycles within the frame work of an expanding nuclear power industry to identify the sources, quantities, timing and cost of production of isotopes of potential importance.

A new task to develop an Isotope Data System was initiated in FY-1971. The objective is a computerized, quick-response system for answering queries on isotope properties, availability, applications, shielding requirements, decay compositions, toxicity, and other factors.

FISSION PRODUCT PROFITABILITY STUDIES

A computer program, CAESR, was developed to carry out calculations that would assist in identifying the time and circumstances when recovery of fission product cesium and strontium from power reactor fuels would be attractive. Using this computer program, a series of cases were run to explore possible pricing requirements for profitable recovery of cesium and strontium.

The computer program provides for a market projection calculation and for the reprocessor to capture a specified fraction of the market; this fraction can be varied with time.

There is a provision for looking at three different startup years (1975, 1977, and 1980 were used) for each case and three plant sizes for each startup year to permit selection of a near optimum plant size for each set of conditions. Prices are calculated as present worth levelized prices or the single price that could be charged over the life of the project that would pay all costs and recover the initial investment with a specified profit or rate of return.

A basic assumption for the initial cases was that the market would first develop with AEC sales but the AEC would then withdraw from the market as soon as a private facility was operating. Thus, this first plant would have 100% of the market initially. However, it was further postulated that a competitor would soon be operating and the first plant's share of the market would decline to 50% of the market after 5 years.

The capital cost estimates for the Cs/Sr recovery and encapsulation facilities that were used as input for the pricing calculations were obtained from Vitro, Hanford Engineering Services based on facility requirements outlined by W. A. Rodger.⁽¹⁾ The reference plant capital cost estimate was as follows:

	<u>Capacity</u>	<u>Capital Cost</u>
Cesium only	29 MCi/yr	\$14.3 x 10 ⁶
Strontium only	28 MCi/yr	\$14.6 x 10 ⁶
Cs/Sr Co-Products	29/28 MCi/yr	\$20.5 x 10 ⁶

These estimates include a 20% escalation factor on a 1970 base estimate. All other costs are based on 1970 dollar

(1) W. A. Rodger, S. A. Reese and S. S. Stanton. "Multi-purpose Plant for the Recovery of Neptunium and Other Isotopes," prepared for New York State Atomic and Space Development Authority, January, 1967.

values. The assumption here is that all costs will escalate more or less uniformly except for construction costs, which will escalate an additional 20% over other costs by the time the facilities are completed.

Other basic assumptions were developed based on our interpretation of views expressed by fuel reprocessors in discussions with them relating to their interest in and their requirements for participation in fission product recovery ventures.

Principal items in these assumptions are:

Return on investment	15%/yr
Economic life	10 years
Income tax rate	50%
Depreciation method for taxes	Sum of years
Design and construction period	4 years

Two types of market growth curves were examined for projections reaching 20, 40 and 60 MCi sales in 1990 with curves fitted to the 1965 to 1970 AEC domestic sales of ^{137}Cs . One curve, the Gompertz curve, projected a steeper growth rate in the early years compared to the other, which can be described as a decaying exponent curve. Equations for the two curves are:

$$\text{Gompertz:} \quad M = e^{(K+AB^T)}$$

$$\text{Decaying Exponent:} \quad M = M_0 e^{[r(1-e^{-KT})/K]}$$

where M = market at time T ; A , B , K and r are constants,
and r = growth rate at $T = 0$

Historical data for the cesium market is insufficient to resolve which growth curve will be most appropriate for projection purposes.

Results of the price requirement calculations are shown in Table 1. These results illustrate the high sensitivity to

TABLE 1. Calculated ^{137}Cs Price Requirements for Single Purpose and Two-Product Plants

Plant Startup Year	Market Assumed					
	20 MCi in 1990		40 MCi in 1990		60 MCi in 1990	
	Projection Method, ¢/Ci					
	Gompertz Curve	Decaying Exponent	Gompertz Curve	Decaying Exponent	Gompertz Curve	Decaying Exponent
<u>Single Purpose Plant</u>						
1975	71	100	48	71	39	53
1977	60	76	40	53	33	40
1980	50	57	34	40	27	31
<u>Two-Product Plant</u>						
1975	51	73	34	50	28	38
1977	43	54	29	38	23	28
1980	36	40	24	28	19	22

market growth rates and market size and they show the advantage of sharing the cost of basic facilities between two products. Furthermore, they indicate that even with very optimistic growth rates, markets will probably not be large enough to support private enterprise production of cesium earlier than the late 1970's or 1980.

Other cases have been run to examine the sensitivity of results to other major variables. These results have shown that the most important factors affecting price requirements are capital cost, market share, return on investment, and economic life. The factors of lesser importance (wide variations have a small effect on results) are operating costs, cost scaling factors, working capital requirements, and learning curve effects.

ISOTOPE DATA COMPILATIONS

Development of an isotope data system has been initiated. This system will provide both a quick response to queries on isotope properties and a file on basic data to be used for other purposes such as shielding requirements, dose rates, and decay compositions.

The techniques used in Battelle-Northwest's English Language Information Retrieval System for storing, manipulating, and retrieving large amounts of data was chosen as the best method for handling the physical properties of the nuclides. However, modifications to the existing computer code were necessary to allow for types of data which have no correspondence between nuclides. As an example, the number of known gamma energies for a particular nuclide have no relationship to the number of gamma energies for other nuclides. Efficient storage of these data can be best accomplished by adding a variable length third dimension to the existing two-dimensional matrix storage system.

Other modifications needed to use these data more efficiently are tabling and sorting techniques and a method of processing the data once it is retrieved. Programming the tabling and sorting capabilities has been completed and debugged with the existing two-dimensional system. The basic logic and computer programming for the three-dimensional storage and retrieval capability has been completed and is now being checked and debugged.

NUCLIDE TABLES

The data file used in preparation of the "Nuclide Tables" has been completely rechecked and revised to include the most recently available data. An examination of the most useful and practical method of handling internal conversion effects is underway.

NONDESTRUCTIVE TESTING FOR IRRADIATION SOURCES

D. R. Newman

The scope of this program is to develop methods of nondestructive examination of isotopic heat sources developed by DID contractors. Through a number of programs, DID is developing applications for various radioisotopes as heat sources for specific applications. The PNL program will concentrate on the development of methods to assure the long-term mechanical and thermal integrity of the as-manufactured sources.

Because it is important that NDT methods are compatible with capsule design (e.g., welds should be physically accessible for inspection), the approach of this program is to become involved early with contractors who have design and development responsibilities. Continuing liaison will ensure that NDT methods are available for the source under development.

DIFFERENTIAL TRANSDUCER TECHNIQUE

Construction of prototype differential transducers has begun. Initial evaluations of the transducers and the technique will be undertaken and reported in the coming quarter.

MULTIELECTRODE APPLICATION

Several development ^{60}Co strips were tested for bond between the clad and core using an electronically scanned multielectrode transducer system. Figure 2 shows the results from testing the upper edge of three of these strips. Strip A has a complete unbond; sound passes only to the electrodes that are above the edge of the strip. (Only the top edge was

A

Complete Unbond

B

Partial Bond

C

Good Bond

FIGURE 2. Multielectrode System Examination of Bond Integrity
on ^{60}Co Coextruded Sources

tested since this particular transducer was only 0.6 inch wide and the strip was 0.75-inch wide.) Strip B had a partial unbond along the top edge, while Strip C was completely bonded. The dimensions are roughly actual size in these photographs. The scan was made at about 1 in./min. The transducer system is capable of much faster translation; it would interrogate every 0.015 in. if the translation speed were 1 in./sec.

Figure 3 shows a scan of a 3-inch long nonactivated sample source with a 10-mil stainless steel cladding and a SrSiO_3 -Al matrix core. Only one end is shown; radiographs of this sample show a low density area on this end

FIGURE 3. Multielectrode Readout of Possible Unbond or Void in SrSiO_3 -Al Matrix Source

that has approximately the same shape shown in the ultrasonic scan. This is believed to be due to porosity or a void in this region. Metallographic investigation will follow.

PUBLICATIONS

1. A report, "Pulsed Laser Imaging System for Direct Visualization of Ultrasonic Waveforms," was accepted for presentation at the 80th meeting of the Acoustical Society of America, November 1970.
2. An abstract of "Recent Developments in Nondestructive Testing of Isotope Irradiation and Heat Sources" was submitted for possible presentation at the Third International Symposium on the Packaging and Transportation of Radioactive Materials. This symposium will be held in Richland, Washington in August 1971.
3. A paper, "NDT Applications of a Pulsed Laser Schlieren System" was submitted to the American Society for Non-destructive Testing Spring Meeting in Los Angeles, March 1971.

ADVANCED SOURCE DEVELOPMENT

J. H. Jarrett

The objective of this program is to evaluate present and future heat and radiation source application requirements and to relate them to present and future encapsulation technology.

The goal of the current work is concerned primarily with the development of thin-film encapsulation and evaluation techniques for alpha/photon sources. Emphasis will be directed toward the technique of sputtering as a means to obtain thin films of radioisotope and containment materials.

The glove box sputtering system was moved to a larger room and a number of general system improvements were made. A new sputtering system, which was ordered in the previous quarter, was delivered and installed in the same room. The new system will be used for cold (nonradioisotopic) process and prototype development work. The common installation allows simultaneous operation of both systems by one operator. Source preparation hardware for sources with a 1/2-inch diameter active area and for planar ring sources was fabricated. Techniques for depositing a material in the glovebox sputtering system without adding radioisotopic cross-contamination to the substrate or the deposited layer were developed and demonstrated. In the previous quarter photon and alpha photon sources were prepared, sent to ORNL and evaluated. The test results were generally encouraging. Areas where PNL prepared sources can be improved were identified, and improvements in these areas are being incorporated into our source preparation efforts. The ORNL tests indicated that sputtered 304 stainless steel layers may be susceptible to rusting when exposed to boiling water or heated high-humidity air for extended periods of time. The cold (nonradioisotopic) sputtering system was used to prepare specimens of 304 stainless steel deposited on

copper and 304 stainless steel substrates under various system operating conditions. These specimens were placed in boiling water for extended periods of time and the degree of apparent rusting was observed. A paper, "Thin Film Radiation Sources Using RF Sputtering Techniques" was presented by J. H. Jarrett on October 19 at the 1970 TMS fall meeting in Cleveland, Ohio. The meeting was cosponsored by AIME and ASM.

FACILITIES

The glovebox sputtering system that was located in Room 324, 325 Building, has been moved to Room 504, 325 Building. The glovebox installation was completed in August. Improvements were made on the closed-loop cold water recirculation system that is needed to cool the sputtering target and substrate while the system is in operation. The black iron heat exchanger was replaced with a stainless steel heat exchanger. An in-line filter was placed between the recirculation pump and the sputtering module. This greatly reduces the amount of suspended matter in the cooling water and minimizes the possibility of plugged flow passages. The "J" cathode assembly in the glovebox system was fitted with new O-rings, which helped minimize air leakage into the system during sputtering. Several rf power leads were repaired or replaced and the components of the rf power supply were cleaned where dust and lint had gathered. These steps nearly eliminated the frequent system adjustment previously needed to maintain optimum operating conditions.

The new sputtering system for cold process and prototype development work was also installed in Room 504, 325 Building. The system has been in operation since early in the quarter. Both sputtering systems may be operated simultaneously by one operator.

HARDWARE DEVELOPMENT

Improved hardware (Figure 4) for preparing sources with a 1/2-inch diameter active area was fabricated. Ten sources may be prepared in a single run on circular substrates of 0.875 inch diameter and 0.060 inch thickness. The substrates are held in position by placing them in the holes in the substrate holder. Pins in the substrate holder position the mask accurately over the substrate plates. The mask is put in place for the deposition of the radioactive layer. It is removed for the deposition of the containment window.

Hardware (Figure 5) for preparing planar ring sources was also fabricated. The planar ring source substrate has an outside diameter of 1.480 inch, an inside diameter of 0.525 inch, and is 0.060-inch thick. The substrate ring width is 0.478 inch and the masks are such that the active layer is 0.322-inch wide. The substrate is held in place by the substrate positioning ring. The four pins in the substrate holder, which is 5 inches in diameter, accurately align the substrate positioning ring and the masks used to position the radioisotope layer on the substrate. The radioactive layer is deposited on the planar ring source substrate with the masks in place as in Figure 6. The masks are removed for deposition of the containment window.

CROSS-CONTAMINATION EXPERIMENT

An experiment designed to develop and demonstrate techniques for depositing a material in the glovebox sputtering system without adding radioisotopic cross-contamination was completed. Beryllium was deposited on glass and polished stainless steel substrates in the glovebox sputtering system. The substrates were then removed from the glovebox and counted for alpha activity using a 2π gas flow proportional counter.

Neg 704987-2

FIGURE 4. Sputtering Hardware for Preparing 10 Radiation Sources in a Single Run

Neg 705086-3

FIGURE 5. Sputtering Hardware for Preparing a Planar Ring Source

Neg 705086-1

FIGURE 6. Assembled Hardware
for Preparing a Planar
Ring Source

The detected alpha activity levels were quite low, ranging from 12 to 40 dis/min. The results of this experiment are valuable to the source development program. Using the techniques developed in this experiment, future radiation sources prepared by sputtering can be removed from the glove-box system without significantly contaminating them and the final containment layer on the sources may be deposited in a nearly contamination-free condition. The experiment demonstrates the feasibility of using a single glovebox sputtering system for the preparation of radiation sources in different sputtering runs with different radioisotopes without significant cross-contamination. Also, the successful deposition of beryllium on ceramic and metallic substrates allows further consideration of beryllium as a candidate material for sources that require the substrate and window material to have an atomic number less than 14.

SOURCE TESTING AT ORNL

The ORNL tests on PNL-prepared photon and alpha photon sources have been completed. The sources were prepared in the previous quarter,⁽¹⁾ and tested at ORNL as part of the AEC-DID sponsored Source Safety Testing Program. The test results were generally favorable, but some areas where the sources could be improved were indicated. Examples include source surface defects, source layer quality and adherence, and the corrosion resistance (apparent rusting) of the stainless steel protective layer. As a result, several improvements are being incorporated into our source preparation work. We hope to minimize surface defects by using polished (7B finish) stainless steel substrate materials, and to minimize the effects of dusting when sputtering targets are changed. Improved layer quality and layer adherence should result with improvements in sputter-etching techniques and improvements in atmosphere purity during sputtering.

SPUTTERED 304 STAINLESS STEEL LAYERS

The cold sputtering system was used to prepare 304 stainless steel deposits on copper and 304 stainless steel substrates at various operating conditions. The coated substrates were placed in boiling water for extended periods of time to determine their rust resistance, since in tests at ORNL, PNL-prepared

(1) Pacific Northwest Laboratory Division of Isotope Development Program Quarterly Report: May-July 1970, edited by J. E. Hansen, BNWL-1308-3, p. 27. Battelle-Northwest, Richland, Washington, August 1970.

radiation sources with a sputtered stainless steel outer layer appeared to be susceptible to rusting when exposed to heated water or heated high-humidity air. Eight different types of specimens were prepared under four different system conditions. Two specimens of each type were prepared. Eight specimens were placed in boiling water for a number of days, and the remaining eight specimens were held as controls. The specimen preparation conditions are summarized in Table 2. The same deposition procedure was used for each series of specimens. The air leak rate and the substrate temperature were the only system parameters that were varied significantly. These were the parameters most likely to have been varied in previous source preparation runs. The results of specimen testing are shown in Table 3.

TABLE 2. System Conditions for Depositing 304 Stainless Steel on Copper and Stainless Steel Substrates

<u>Specimen Identification</u>	<u>Substrate Material</u>	<u>Number Prepared</u>	<u>Leak Rate,^(a) Torr/min</u>	<u>Substrate Position</u>	
Series 1					
1-SS/SS	304 SS	2	3.2×10^{-5}	Direct contact with base	
1-SS/Cu	Copper	2	3.2×10^{-5}		
Series 2					
2-SS/SS	304 SS	2	$\left. \begin{array}{l} 1.8 \times 10^{-2} \text{ at start} \\ 3.5 \times 10^{-3} \text{ at end} \end{array} \right\}$
2-SS/Cu	Copper	2			
Series 3					
3-SS/SS	304 SS	2	3.0×10^{-2}		
3-SS/Cu	Copper	2	3.0×10^{-2}		
Series 4					
4-SS/SS	304 SS	2	$\left. \begin{array}{l} 3.0 \times 10^{-2} \\ 3.0 \times 10^{-2} \end{array} \right\}$	Held 1/8 inch above base with ceramic beads	
4-SS/Cu	Copper	2			

a. The leak rate indicates the change in system pressure per minute (measured with the valve to the oil diffusion pump in the closed position).

TABLE 3. The Effect of Boiling Water on 304 Stainless Steel Specimens Sputter-Deposited Under Various System Conditions

Specimen Identification	Days Exposed to Boiling Water	Specimen Appearance Before Test	Specimen Appearance After Test (40X Magnification)
1-SS/SS	21	Medium shiney, grey	Dull grey, several small rust-colored areas
1-SS/Cu	21		Dull grey, three small rust-colored areas
2-SS/SS	21		Same as 1-SS/SS
2-SS/Cu	21		Dull grey, two small rust-colored areas
3-SS/SS	14		Dull amber-grey, a few rust-colored areas near flakes
3-SS/Cu	14	Medium shiney, amber-grey, film flakes attached to source surface	Dull amber-grey, no rust-colored areas were located
4-SS/SS	14		Same as 3-SS/SS
4-SS/Cu	14		Dull amber-grey, one large rust-colored area about 1/16 inch x 3/8 inch

With the exception of specimen 4-SS/Cu, significant changes were not observed in specimen appearance after exposure to boiling water. In nearly all cases the small areas of apparent rusting were not visible to the naked eye. As a result, no strong trends dependent on substrate temperature or on oxygen leak rate areas were observed. The stainless steel layers on all specimens appeared to adhere well. To eliminate the film flakes attached to the film surface, the air leakage into the system should be less than that giving a closed system pressure change of 3×10^{-2} Torr/min. Since some apparent rusting of the sputtered 304 stainless steel layers was observed, this material will not be used as containment windows for sources to be prepared in the near future. Instead, sputtered nickel will be investigated for the next radiation sources to be prepared. Stainless steel alloys having higher concentrations of nickel and chromium will be considered for future radiation sources, since such alloys should not be as susceptible to rusting as sputtered 304 stainless steel appears to be.

RADIATION CHARACTERISTICS OF CIRCULATORY SUPPORT
ISOTOPIC HEAT SOURCES

F. T. Cross and J. C. Sheppard

The objective of this program is to determine the dose from radioisotopic heat sources suitable for circulatory support systems. This is accomplished by measuring the dose rates within and surrounding various tissue equivalent phantoms.

Part of the program concerns the prediction of the measured dose rates by the development of computational codes. The computer codes are also used in parametric studies and for the prediction of the in-phantom dose rates from sources not available for measurement.

RADIATION CHARACTERISTICS - NONPROMETHIUM SYSTEMS

Average photon dose rates to various organ systems from an implanted 30-W ^{238}Pu heat source placed on the inside surface of the left ilium were calculated with the QAD-Code. The dose contributions of the ^{236}Pu impurity and the neutrons from the ^{238}Pu are not included, although the major portion of the dose--at least for the first year or two--comes from the ^{238}Pu photons. The organ geometries developed by Snyder, Fisher and Warner were used in the calculation along with cylinders, combinations of cylinders, and spheres, which were used as approximations. About 100 source kernels were uniformly distributed throughout the plutonium; the dose points ranged from 16 for a small organ like an ovary, to 412 for the small intestine. The photon dose rates (Table 4) for the various "mathematical" organs were calculated using the buildup factors for water. The proper buildup factors will be determined based on a comparison of the calculated factors with the actual measurements at the respective organ locations. The calculations

indicate that the average photon dose for these organs is relatively insensitive to the geometry chosen, and for most cases the center point dose is a good approximation to the average dose. Also, the number of dose points apparently can be reduced by choosing a small number randomly.

TABLE 4. Photon Dose Rates to "Mathematical" Organs ^(a)

Organ	Assumed Geometry	Center-Center Distance, cm	Average Dose Rate, mrad/hr ^(b)
Spleen	Cylinder	19.2	1.01
	Ellipsoid		1.01
	Center point		0.890
Left Kidney	Ellipsoid intersected by plane	16.6	1.76
	Center point		1.45
Stomach	Cylinder	19.3	0.896
	Center point		0.784
Small Intestine	Cylinder intersected by planes	--	2.54 ^(c)
			2.60 ^(d)
Left Ovary	Ellipsoid	9.05	6.06
	Center point		6.09
Uterus	Ellipsoid intersected by plane	15.4	1.41
	Cylinder		1.60
	Center point		1.45
Bladder	Sphere	18.8	1.21
	Center point		1.17
Left Adrenal	Ellipsoid intersected by plane	24.7	0.456
	Center point (approx.)		0.392
Heart	Sphere	36.7	0.292
	Center point		0.287

a. From 30-W ^{238}Pu source shielded with 0.0765 cm Ta.

b. H_2O buildup assumed.

c. Calculated for 412 uniformly distributed dose points.

d. Calculated for 36 randomly chosen dose points.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

R. W. Perkins and N. A. Wogman

In recent PNL studies of the composition of deep ocean sediments, it has been demonstrated that various mineral deposits and commercial-grade ores can readily be identified by neutron activation and instrumental analysis. From preliminary analysis, it is evident that the elemental composition of most mineral deposits can be easily identified at concentrations well below those of economic importance. In-place experience with ^{252}Cf as a neutron source has confirmed these conclusions. Two methods for in-place seabed mineral exploration are being investigated. The first involves placing a neutron source and detector system on the ocean floor. The second method involves bringing the samples on-board ship and uses a shipboard ^{252}Cf irradiator and a Ge(Li) spectrometer. The former technique has the advantage of very rapid analyses, and the latter technique has the advantage that mineral depth-profiles can be defined from core sampling.

Work during the past quarter has been directed toward flux characterization and calibration of a recently received ^{252}Cf source. A 1.8 mg ^{252}Cf source was received from ORNL early in this period. It was double-encapsulated in stainless steel and is presently contained in an additional 3-cm diameter aluminum rod, which is positioned at the end of a 10-foot pole to permit remote handling. The irradiation facility (Figure 7) for studying the activation analysis of simulated seabed areas consists of a 6-foot diameter by 7-foot high polyethylene tank located in a 15-foot diameter by 7-foot deep swimming pool. Mineral samples are placed in a 2-foot diameter by 10-inch high container which can be moved down a sample channel and located directly under the ^{252}Cf source. With the samples saturated with seawater and the ^{252}Cf source in a large moderator as shown, the conditions precisely duplicate those expected in actual seabed mineral analyses.

FIGURE 7. ^{252}Cf Irradiation Facility Simulation System for Seabed Mineral Analysis

The thermal neutron flux has been characterized for several types of substrate material in the irradiation position.

Figure 8 shows the observed horizontal thermal neutron flux profile at the surface of a paraffin substrate. The maximum flux is about 2.7×10^7 n/cm²-sec at a position of about 2 inches directly below the ²⁵²Cf source. In this paraffin substrate, the average flux is reduced about twofold through the area of a 20-cm hemisphere, and threefold through a 35-cm diameter hemisphere. Figure 8 also shows the horizontal flux profile with a seawater substrate. The use of seawater rather than paraffin as a moderator results in a 40 to 50% attenuation of the thermal neutron flux. To simulate the thermal neutron flux which will be expected in the seawater-saturated ocean floor, measurements have been made in seawater-saturated dunite substrates. These profiles characterize the decrease in thermal neutron flux as a function of both the horizontal and vertical distance from the source. From these measurements, together with attenuation measurements of gamma radiation emitted from various locations in the irradiated sample, the effective sample size seen by a detector which is placed over the sample can be established.

Calibrations are in progress to determine the response, characteristics, and the detection sensitivity for some 40 elements in simulated seabed materials. For this work the samples are irradiated for 2 minutes each in the irradiation position indicated in Figure 9. They are then rapidly removed and counted for a 2-minute period with a large Ge(Li) diode. This diode is immersed in seawater with the same type of shielding anticipated for an actual seabed detector system. The calibration information obtained from these studies will permit the direct determination of element concentrations from gamma-ray spectrometer measurements with the actual seabed nuclear probe device.

FIGURE 8. Horizontal Flux Profile at Surface of Paraffin and Seawater Substrate Beneath a ^{252}Cf Source

FIGURE 9. Horizontal Flux Profile as a Function of Depth in Dunite Saturated with Seawater

Design of the nuclear probe is essentially complete and construction of major components will begin in the very near future.

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