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UC-23

PACIFIC NORTHWEST LABORATORY
DIVISION OF ISOTOPE
DEVELOPMENT PROGRAMS
QUARTERLY REPORT:
NOVEMBER-JANUARY 1970-71
March 1971

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PACIFIC NORTHWEST LABORATORY
DIVISION OF ISOTOPES DEVELOPMENT PROGRAMS
QUARTERLY REPORT: NOVEMBER-JANUARY 1970-71

Compiled by J. E. Hansen

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INTRODUCTION

This report, BNWL-1308-5, is the 17th 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, and 1308-1 through 1308-4.

This report covers the three-month period, November through January 1970-71.

SUMMARYENCAPSULATED WASTE MANAGEMENT CESIUM AND STRONTIUM AS PROCESS RADIATION SOURCES

Cesium and strontium capsule sizes were changed by HWMP without varying the amount of material per container. The process for encapsulation of HWMP ^{137}Cs in a smaller container size was successfully demonstrated with a device fabricated for the purpose. Work on strontium sources continued with calculations of temperature for varying values of size and density in doubly encapsulated SrF_2 capsules, and investigation into the properties data of SrF_2 with other metal halides as impurities.

UTILIZATION OF FISSION PRODUCT RHODIUM, PALLADIUM, RUTHENIUM AND TECHNETIUM

The $^{102\text{m}}\text{Rh}$ decay measurements continued, with termination of counting, analysis of acquired data, and preparation of the final report foreseen for the coming quarter. Investigation into the radiation hazards and licensability of the fission products Rh, Pd, Tc, and Ru began along with an analysis of the potential market for these isotopes.

EVALUATION OF LARGE SCALE USES FOR PURIFIED FISSION PRODUCT XENON

Consideration of potential uses of purified fission product xenon indicated the need for further purification of the 900 liters now on hand to remove certain major contaminants such as CO_2 . The ^{85}Kr level, however, appears to be acceptable from a use standpoint but may still be a problem for licensing.

ISOTOPE PRODUCTION EVALUATIONS

Values relative to ^{60}Co at 40¢/Ci were determined for both encapsulated HWMP ^{137}Cs and encapsulated power reactor ^{137}Cs

based on a discounted cash flow technique. The Isotope Data System has been tested with a set of data from 50 selected isotopes, and all design query options appear to function. On completion of Nuclide Data File updating, data on all 2,000 nuclides will be loaded into the system.

NONDESTRUCTIVE TESTING FOR IRRADIATION SOURCES

Construction and evaluation of the differential transducer system continued. To date, several transducers have been successfully fabricated and tested.

NEUTRON RADIOGRAPHY USING ^{252}Cf

Following completion of the basic design and analysis of a neutron imaging counter, the specification and fabrication of necessary electronics was undertaken. The analysis shows the counter will produce useful images with moderate resolution.

ADVANCED SOURCE DEVELOPMENT

The improved source preparation hardware fabricated in the previous quarter was demonstrated using the cold (nonradioisotopic) sputtering system. Numerous improvements were made to the glove box sputtering system, and a source preparation run using the improved hardware was completed. Ten alpha photon sources were made in a single run. Selected sources from this run were given preliminary testing at PNL, which included a boiling water leach test and an ultrasonic cleaner test. Four of the sources were sent to ORNL for testing in the DID-sponsored Source Safety Testing Program. One of the major objectives of the testing will be to determine the licensability of sources having this design.

RADIATION CHARACTERISTICS OF CIRCULATORY SUPPORT ISOTOPIC HEAT SOURCES

Measurements of neutron dose rates from the Rankine dosimetry model continued using a ^{252}Cf source. A tentative value for the rate of growth of photon dose rates from the ^{236}Pu impurity in LASL-1 was obtained from analysis of the first series of repeat measurements. Previously reported average dose rates to various organs from LASL-1 were found to be in error and corrected dose rates were obtained.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

The sensitivity with which various elements can be measured in a simulated ocean floor matrix was established for 20 selected elements. The results from this experiment have been used to establish criteria required to finalize prototype design of the ~~in situ~~ ocean floor mineral analyzer system.

ENCAPSULATED WASTE MANAGEMENT CESIUM AND STRONTIUM
AS PROCESS RADIATION SOURCES

H. H. Van Tuyl, L. K. Mudge, 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.

Container sizes for both cesium and strontium were recently changed by HWMP to provide easier handling, welding, and NDT; however, the amount of material per container was not changed. New dimensions are 2.625 in. OD for the outer capsule and 0.250 in. OD for the inner capsule in each case. The total wall thickness for the cesium capsule was reduced slightly to 179 mils. The utility of each type of capsule for radioisotope applications is not affected appreciably.

The process for encapsulation of ^{137}Cs in the Hanford Waste Management Plant involves casting of molten CsCl into a series of nine containers. Demonstration of the process has been completed for four 2 in. OD by 20 in. long capsules of 0.095 in. wall thickness. Some concern was expressed about the ability to transfer the molten CsCl through small diameter lines into a series of 1 in. OD capsules, a size that would be attractive

as a radiation source. Originally, only five containers were to be filled at a time, but improvements in this operation now permit transferring twice as much CsCl per batch. If the container diameter is reduced, the number of capsules filled per batch must be increased even further to avoid a production bottleneck in the proposed plant. The objective of this work was to demonstrate transfer of CsCl into 1 in. OD capsules in equipment that could be used in the Waste Management Plant.

A device for loading a series of ten 1 in. OD by 18 in. long capsules with 0.035 in. wall thickness was fabricated for testing. These dimensions are slightly different from those proposed for optimized radiation sources; however, they were sufficiently similar to prove the principle and were more readily available. Each capsule was mated and sealed to a transfer head that contained 1/4 in. lines for conducting the molten salt to and from each capsule in series. The capsule-to-transfer head seals were made integral with the inside circumference of each capsule about 3/4 in. below the capsule lip to prevent contamination of the weld zone with molten salt.

Molten CsCl was transferred into the series of 10 capsules without difficulty. Eight capsules were filled in less than two minutes with a vacuum of 20 in. Hg. The density of the CsCl, which contained 10 wt% NaCl, was 2.68 g/cm^3 (about 67 TD) based on the total capsule volume. The average density of the cooled CsCl slug was 3.06 g/cm^3 (about 78% TD). The slower cooling in HWMP equipment could result in a higher density. A uniform cylinder of CsCl was produced with only slight (about 1/2 in.) depression of the top surface caused by cooling.

The transfer unit cost was less than \$2000. The unit can be used in series with the standard transfer unit for loading waste capsules. Ten 1 in. OD capsules could be loaded, and the molten salt from the last capsule would overflow into the standard series of waste capsules. In this manner, small diameter capsules could be loaded intermittently without adjusting

the batch size normally used in the Waste Management Plant. Special equipment would be needed for welding and NDT of the capsules.

Temperature calculations were made for varying sizes of the doubly encapsulated strontium fluoride capsules. The density of the strontium fluoride product in these capsules was varied in increments of 5% from a low of 65% of theoretical density to a maximum of 90% theoretical density for each capsule size. All calculations were based on a cylinder vertical oriented and suspended in air with the heat radiating to a 70 °F room temperature. The encapsulation material was Hastelloy C metal. The heat generation rate was calculated at 95% of ^{90}Sr theoretical values. This rate appears to closely approximate the actual waste management product.

The radius of the inner capsule was varied in 0.10 in. increments from a 1 to 2 in. radius. A 15 mil gap was left between the outside of the inner capsule and the inside of the outer capsule. A wall thickness of 120 mils was used for each capsule.

A centerline temperature of 1463 °C (the melting point of pure SrF_2) results with an outside diameter of 4 in. at 65% density, and 4.4 in. at 85% density. Corresponding surface temperatures are 510 °C and 595 °C, respectively. Even smaller diameters would be necessary to avoid a molten core since impurities will reduce the melting point of SrF_2 .

Sodium fluoride and strontium fluoride do not form a solid solution under any conditions, according to work by G. A. Bukhalova in 1955. They do form an equimolar eutectic with a melting point of about 850 °C. Thus, the thermal equilibrium composition expected in HWMP strontium product is a bulk phase consisting of alkaline earth fluorides and a small amount of a second phase containing alkali-alkaline earth fluoride eutectic. The properties of the bulk phase can be evaluated with sufficient accuracy

from existing data on the pure components and the laws of solid solution. The properties of the impurity phase cannot be evaluated adequately from existing data. Some additional laboratory work appears to be necessary to characterize the total system.

In its simplest form the encapsulated SrF_2 would consist of strontium fluoride, zirconium daughter and a certain amount of impurity sodium fluoride. The introduction of daughter zirconium into the SrF_2 system produces significant complexity. The stoichiometry of the resulting zirconium compound is of course ZrF_2 . One would expect the presence of ZrF_2 in SrF_2 to produce phase behavior similar to mixtures of other divalent fluorides. If a series of solid solutions is formed, this would probably mean a maximum freezing point depression of only a few hundred degrees. In the absence of SrF_2 - ZrF_2 solid solutions, a eutectic with a much lower freezing point might be obtained. The presence of sodium fluoride in such a mixture would be expected to lower the fp still more, the result being a bulk phase of nearly pure SrF_2 and a small-mass phase containing the mixture.

The chemistry of the fluorides of zirconium is of interest in the effort to estimate the Zr species that will control the phase behavior of aged SrF_2 . Compounds of ZrX_2 and ZrX_3 have been reported for all the halides, but the fluorides are less well described than the others. Zirconium metal will reduce ZrCl_4 to ZrCl_3 at 450 °C and to ZrCl_2 at 675 °C. The ZrF_3 compound is said to exist, and ZrF_4 has been reduced at 350 °C by atomic hydrogen to ZrF_2 . The ZrF_2 compound disproportionates at 800 °C, which indicates that daughter zirconium exists as ZrF_2 in the beginning and as a mixture of metal and ZrF_4 if the material in the capsules becomes hot enough.

The phase behavior might change significantly above a temperature at which disproportionation of ZrF_2 takes place. The ZrF_4 compound itself melts at about 920 °C, which is significantly

lower than what one would estimate for ZrF_2 . It forms a series of compounds with alkali and (presumably) alkaline earth fluorides, which form eutectics with each other, some melting as low as 500 °C. The phase diagrams of ZrF_4 with the alkali halides indicates, however, that at lower ZrF_4 concentrations the lowest mp mixture would probably be a eutectic with either the compound M_3ZrF_7 or a compound containing whatever cations are available (perhaps Sr^{+2}). Such a eutectic could melt below 700 °C and, if there were several different cations, perhaps well below that.

Studies of the disproportionation of ZrF_2 would be worthwhile, particularly to find if there is a drastic change in behavior when the temperature of the zirconium containing systems goes through the disproportionation temperature of ZrF_2 .

A research plan is being developed to measure the properties of the NaF-SrF_2 eutectic. No further work is planned for the ZrF_2 system at present.

UTILIZATION OF FISSION PRODUCT RHODIUM,
PALLADIUM, RUTHENIUM, AND TECHNETIUM

The traditional view of fission products as waste materials has been supplanted by their role as a national resource. Pursuant to this revised status, a resource strategy is now being formulated to direct optimum technological and economic application of the fission products Rh, Pd, Ru, and Tc created in operation of nuclear power reactors. The program seeks to evaluate the potential of these artifacts as supplements to the natural reserve of precious metals, and emphasizes the practical aspects of their safe use. The program comprises the following tasks:

- 1. Evaluation of rhodium and palladium produced in power reactors.*
- 2. Technology for recovery of Rh, Pd, Tc, and Ru.*
- 3. Market analysis of Rh, Pd, Tc, and Ru.*
- 4. Economic analysis of Rh, Pd, Tc, and Ru production from nuclear wastes.*
- 5. Fabrication process development for nuclear source (Rh, Pd, Tc, and Ru) products.*

^{102m}Rh HALF-LIFE -- F. P. Roberts

The determination of the half-life of ^{102m}Rh is nearing completion. Acquisition of counting data will continue for about one more month, and the data will be subjected to a statistical analysis. The final report will be prepared in the coming quarter.

RADIATION CHARACTERISTICS -- F. P. Roberts

The radiation hazards and the problems of obtaining a license for industrial use of the fission products Rh, Pd, Tc, and Ru are being studied. A compilation of the characteristics of the radioactive isotopes of these fission products have been tabulated (Table 1). The radioactive components of technetium and palladium have such long half-lives that they do not decay significantly even over relatively long periods of time, and

therefore, the hazards associated with their use are in effect, fixed. The radioactive components of rhodium and ruthenium have "short" half-lives, and the evaluation of the hazards for a given application must be based on the age of the fission product. In the case of rhodium the controlling component is ^{102m}Rh , and for ruthenium it is ^{106}Ru . Decay curves for the radioactive components of rhodium and ruthenium are shown in Figure 1.

TABLE 1. Radiation Characteristics of Rh, Pd, Ru, and Tc Isotopes

Isotope	Half-Life	Decay Mode	Maximum Beta Energy, MeV	Gamma Energies, MeV	Specific Activity, (a) $\mu\text{Ci/g}$
^{102}Rh	206 d	Beta Positron E.C.	1.29	0.475 (57%) 0.511 (25%) 0.632 (54%) 0.698 (41%) 1.110 (22%)	1900
^{102m}Rh	3.4 y	E.C.	none	0.418 (13%) 0.475 (95%) 0.632 (54%) 0.768 (30%) 1.050 (41%) 1.110 (22%)	290
^{107}Pd	7×10^6 y	Beta	0.04	none	90
^{103}Ru	39.6 d	Beta	0.70	0.497 (88%) 0.610 (6%)	5×10^9
^{106}Ru	368 d	Beta	$3.54^{(b)}$	0.512 (21%) ^(b) 0.622 (11%) 1.050 (1.5%) 1.130 (0.5%) 1.550 (0.2%)	5×10^8
^{99}Tc	2.12×10^5	Beta	0.292	none	1.71×10^4

a. The specific activities are for the fission product mixture. The values for ruthenium are for the "worst case" and are probably too high.

b. These include the daughter radiations from ^{106}Ru .

FIGURE 1. Specific Activity of Rh and Ru Isotopes as a Function of Decay Time

FABRICATION PROCESS DEVELOPMENT -- R. G. Nelson

Work has been started to survey the present technology of precious metals fabrication as part of the study to evaluate the effect of radioactivity from fission products rhodium, palladium, technetium, and ruthenium.

MARKET ANALYSIS -- J. C. Sheppard

Most of the effort in this area has been devoted to establishing the supply and demand situation of the platinum metals. An attempt has been made to determine the world reserve of the platinum metals. The data in Table 2 summarize the situation at this time; however, these numbers will be subject to revision pending review of the U.S. Geological Survey Professional Paper No. 630, "Economic Geology of Platinum Metals." This paper is expected to firmly establish the extent of the world's platinum metal reserves.

TABLE 2. Estimated World Mine Production and Reserves of Platinum Metals (1969)

Source	Production, 10 ³ oz t	Reserves, 10 ³ oz t	Comments
U.S.	30	3,000	
Canada	350	16,000 (20,000)	Estimated from Ni production and reserves
Columbia	14	5,000	
Republic of South Africa	1,100	200,000 (115,000 to 200,000)	Estimated from dimensions of Merensky reef. Depends on assumed depth.
Other free world	6	---	
USSR	<u>2,050</u>	<u>200,000</u>	
WORLD TOTAL	3,550	424,000	

Data from the U.S. Bureau of Mines Yearbooks have been used to determine the growth rate of U.S. consumption of the various platinum metals and the total world production of platinum metals. The data shown in Table 3 were determined by the least squares method. These data provide a basis for estimating future demand for these elements as well as the in world production. When the reserve situation is more firmly known, the data will be used to estimate the resource adequacy of the platinum elements.

A review of the current uses for platinum metals has also been started. Losses of the platinum-rhodium catalyst used for nitric acid production have been documented. These catalyst losses range from 0.15 to 0.35 g/ton of nitrogen for atmospheric plants, to 0.5 to 1.8 g for plants operating at 3 to 9 atm. These losses account for about 1.0 to 6.7% of the nitric acid cost and also represent potential modes for the dispersion of fission product rhodium.

TABLE 3. Least Squares Analysis of Platinum Metal Consumption

<u>Element(s)</u>	<u>Time Period</u>	<u>Doubling Time</u>
Total Pt metals, world consumption	1946-1969	8.0 - 0.5 y (a)
Pd, U.S. consumption	1954-1967	14.1 3.0
Rh, U.S. consumption	1958-1967	5.4 1.2
Ru, U.S. consumption	1958-1967	8.6 2.1

a. Analysis of the data for the 1930-1946 period is also consistent with the 8.0 y doubling time.

b. These data should be considered as rough estimates since the time periods are rather short. However, they indicate growth rates comparable to the total platinum metal production.

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 as 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 applications, (3) distribute specimens to participating evaluators, (4) compile evaluation results, and (5) prepare a final report on the results of this total effort.

Purification of the first shipment of fission product xenon received from Idaho Falls was continued to achieve lower ^{85}Kr levels. Somewhat over 900 liters (STP) with less than 1 μCi ^{85}Kr /liter are now on hand. Initial contacts were made with essentially all potential producers and users. These contacts strongly suggested the need to continue the purification efforts with attention to the removal of the remaining major contaminants such as CO_2 , CO , O_2 , and H_2O . Arrangements were made to also have these removed at ORNL. The ^{85}Kr level appears to be low enough to encourage the use of the gas in human anesthesia;

the dose rate for even prolonged surgery appears to be significantly less than that received during chest X-rays or in average annual doses from natural radiation, such as from radon in the atmosphere and impinging cosmic rays.

ISOTOPE PRODUCTION EVALUATIONS

R. W. McKee, Z. M. Cole, R. G. Rau, and H. W. Waddel

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 irradiation. 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 framework 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

Calculations were carried out to compare ^{137}Cs value to ^{60}Co value using a discounted cash flow technique. The bases for the calculations are as follows:

1. The value of two sources are assumed equal when their gamma energy production is equal.
2. Sources are replenished annually.
3. The radiation plaque can hold twice as many capsules as required for the initial source.
4. The initial source is capitalized.
5. Replenishments are treated as operating expenses.

6. Federal income taxes are taken as 50%.
7. A 10-year economic life is assumed.

Value determinations were made both for ^{137}Cs as it will be encapsulated in the Hanford Waste Management Program (HWMP) capsules and for power reactor ^{137}Cs encapsulated in optimum design capsules. The value of the HWMP cesium is reduced because of the large self absorption effect in the 2.6-in. diameter capsules. The value of power reactor cesium is enhanced by the large gamma energy contributions of the ^{134}Cs content (approximately 2.5 wt% ^{134}Cs at time of use that yields more than twice the gamma energy of the ^{137}Cs).

The ^{137}Cs values were calculated as values relative to ^{60}Co at 40¢/Ci (Table 4).

TABLE 4. Value of ^{137}Cs Relative to ^{60}Co at 40¢/Ci

Interest Rate	HWMP Cesium		Power Reactor Cesium	
	10 %	20 %	10 %	20 %
<u>At 5¢/Ci ^{137}Cs Salvage Value</u>				
0¢/Ci ^{60}Co Salvage	10.7	8.8	28.2	28.0
20¢/Ci ^{60}Co Salvage	9.8	8.5	25.7	27.0
<u>At 0¢/Ci ^{137}Cs Salvage Value</u>				
0¢/Ci ^{60}Co Salvage	9.3	8.4	26.4	27.0
20¢/Ci ^{60}Co Salvage	8.6	8.1	24.0	26.1

These results show a substantial difference in value between the HWMP cesium and power reactor cesium. Although the salvage value that the source material might have at end-of-life is an important consideration, the results of these comparisons also indicate that the ^{137}Cs values are not overly sensitive to salvage values.

ISOTOPE DATA SYSTEM

The Isotope Data System is now working with a test set of data from 50 selected isotopes. All of the design query options appear to be working.

The system can be queried in English language form and the files can be searched for data, imposing up to 10 constraints on items to be reported. For example, maximum or minimum constraints, or both, can be defined for half-life, gamma energies or abundances, beta energies, cross sections, etc., and only data on isotopes that satisfy these constraints will be reported out.

Results can be reported in tabular form with up to 10 evenly spaced columns and with data sorted in either increasing or decreasing magnitude.

Data on all 2,000 nuclides will be loaded into the system after the Nuclide Data File updating has been completed.

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 and evaluation of the differential transducer system continued. With this technique, Figure 2, a short, high resolution ultrasonic pulse drives both of the electrodes on the transducer. (The common front electrode is grounded.) The pulse propagates through the coupling medium and both penetrates and reflects off the specimen being tested. Any anomalies beneath the surface of the specimen will reflect some of the penetrating signal. A standard transducer will effectively sum the reflected front surface and anomaly signals and display them simultaneously. Here, a problem occurs when a small flaw exists very close to the front surface; its reflected signal will often be swamped by the larger, front surface reflection, and the flaw will not be detected. The differential transducer, however, provides a means of taking the signals from the two symmetrical electrodes and subtracting one from the other. Thus, the signal from the front surface cancels, while the signal from a small

flaw under only one of the electrodes does not. The signal from a larger flaw (one that reflects signals from both transducers) would also tend to cancel except around its edges. The small flaw signal is easily seen and displayed.

FIGURE 2. Differential Transducer Technique

A typical differential transducer is shown in Figure 3 with its piezoelectric element, leads, backing material (tungsten loaded epoxy) and case. Several of these transducers have been fabricated and tested. Typical test results are shown in Figure 4. Figure 4a shows the reflected signals before canceling, and Figure 4b shows the degree of cancellation obtained where a small flaw signal is present. Perfect cancellation apparently is not obtained because of the difficulties of assuring that the front surface of the piezoelectric element is absolutely flat and the piezoelectric elements are symmetrical. Even the slight deformation due to uneven curing of the epoxy is enough to cause distortion.

A paper entitled "Pulsed Laser Imaging System for Direct Visualization of Ultrasonic Waveforms" was presented at the 80th Meeting of the Acoustical Society of America, Houston, Texas in November.

FIGURE 3. Typical Differential Transducer

INDEPENDENT ELECTRODE SIGNALS

SUMMED ELECTRODE SIGNALS

FIGURE 4. Oscillograms Showing Cancellation
of Front Surface Signal

NEUTRON RADIOGRAPHY USING ^{252}Cf

K. L. Swinth

The objective of this program is to develop and refine the application of ^{252}Cf to neutron radiography for field applications using portable sources. A central goal of the program is the development and evaluation of a portable shield and exposure system which meet stringent safety requirements. Program efforts on the development of low-flux imaging techniques will accelerate the acceptance and application of isotopic neutron sources in industry. The development of low-flux exposure systems and low-flux techniques will aid in the productive utilization of ^{252}Cf as a tool for neutron radiography.

The project began in early January of this quarter, and the following covers the work performed in that month.

On completion of the basic design for the position sensitive counter, an analysis of the design was undertaken. This indicated that a full width at half-maximum resolution of about 300 μ should be obtainable with an "unsharpness" of about 800 μ . Production of a useful image is expected to require an exposure between 4×10^7 and 4×10^9 n/cm². Variability is designed into this prototype unit to enable exploration of the resolution and efficiency parameters over a limited range. Most of the materials for fabrication of the prototype counter are either on order or on hand.

Electronics required for operation of the counter have been specified and will be ordered soon. The necessary preamplifiers are being fabricated in-house and the prototype unit is 98% complete. Fabrication will be completed and testing will be performed on arrival of remaining parts.

Imaging of loaded cracks began with the production of a suitable dysprosium compound and tests on candidate specimens. Suitable images were obtained and further work is in progress.

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 sputter deposition of candidate radiation source materials was demonstrated in the cold [non-radioisotopic] sputtering system while evaluating improved source preparation hardware fabricated in the previous quarter. Well adhering films of sputtered 304 stainless steel on copper, and sputtered nickel on sputtered stainless steel were prepared. The films were tested by exposing specimens to boiling water for extended times and also by placing specimens in an ultrasonic cleaner. A number of modifications and improvements were made to the glove box sputtering system. Ten radiation sources were prepared in a single run using the improved hardware. Each source had a 7/8 in. diameter 304 stainless steel substrate, with a 1/2 in. diameter $^{241}\text{AmO}_2$ -Cu active layer sputter deposited on the center of the substrate. The active layer and substrate were covered with a sputtered 304 stainless steel layer, followed by a sputtered nickel layer. The sources were removed from the glove box without adding significant contamination to them, using techniques developed in the previous quarter. The sources showed both alpha and photon activity. They were easily decontaminated with soap and water. The sources were given a preliminary evaluation at PNL. A boiling

water leach test and ultrasonic cleaner tests were conducted on select sources. Four of the sources were sent to ORNL for testing and evaluation in the DID sponsored Source Safety Testing Program. Information on the licensability of sources having this design should result from the ORNL tests.

SOURCE CONTAINMENT LAYER

The cold (nonradioisotopic) sputtering system was used to prepare simulated radiation sources using various candidate source materials. The objective was to identify a material to replace sputtered 304 SS as the final source-containment layer. In tests completed during the previous quarter at ORNL and PNL, sputtered 304 SS showed an apparent tendency to rust in discrete surface locations when exposed to either boiling water or hot, high humidity air for extended periods of time. Nickel was selected as a candidate replacement material. Specimens of nickel sputtered on both 304 SS and copper substrates were prepared. A demonstrated good adherence to both these materials was necessary to qualify nickel as the containment layer in radiation sources having a 7/8-in. diameter 304 SS substrate with a 1/2-in. diameter $^{241}\text{AmO}_2$ -Cu layer sputtered concentrically on the substrate. In initial experiments the nickel films sputtered on copper substrates did not adhere well. This was probably caused by an oxide film that apparently formed on the copper during the sputter-etching process. Since well adhering films of sputtered 304 SS on copper substrates had been previously prepared, it was decided to prepare and evaluate simulated sources, where a layer of 304 SS was sputtered on the substrate and simulated active layer, followed by a sputtered layer of nickel. The simulated radiation source description and specimen testing are summarized in Table 5. Except for nickel sputtered on copper substrates, no problems with the sputter-deposited layers arose. These test results were used in selecting the

TABLE 5. Tests on Simulated Radiation Sources

<u>Specimen Description</u>	<u>Test Description</u>	<u>Test Results (Visual Observation at 40X)</u>
A. Nickel sputtered on a copper substrate	Removal from sputtering system.	Nickel film lifted. A black layer (probably CuO) was observed between copper and nickel.
B. 7/8-in. diameter stainless steel substrate with 1/2-in. diameter stainless steel layer sputtered concentrically on the substrate face, and a sputtered nickel layer covering the complete face.	Specimen placed in an ultrasonic cleaner in water for 2.5 hr.	No apparent effect on specimen.
C. Same as B.	Specimen placed in boiling tap water for 30 days.	No apparent effect on layer adherence or layer integrity. White film that could be removed by slight abrasion was observed on specimen.
D. Same as B except with a square copper substrate, 5/8 in. on each side	Specimen placed in boiling tap water for 30 days with Specimen C.	Same as C.

materials for the next radiation source preparation run, which are in order of deposition as follows:

- 304 SS, 7/8-in. in diameter.
- $^{241}\text{AmO}_2$ -Cu active layer, 1/2-in. in diameter and concentric with substrate.
- Sputtered 304 SS layer covering the active layer and the edge of the substrate.
- Sputtered nickel layer covering the sputtered 304 SS layer.

SOURCE TESTING

The improved source preparation hardware fabricated in the previous quarter was used in preparing the specimens listed in Table 5; it performed satisfactorily. The hardware was placed in the glove box, and, in December, 10 alpha-photon radiation sources were prepared in a single run. The sources were given a preliminary evaluation at PNL, and four were sent to ORNL for testing and evaluation. All sources were observed at 40X magnification. Some surface irregularities were apparent, but the number was greatly reduced from previously prepared sources. The observations are shown in Table 6 along with source activity information. Source No. 5 was placed in boiling distilled water for an extended period of time and checked periodically for smearable contamination (Table 7). The object of the experiment was to determine if exposure to boiling water would cause americium-241 to be leached from the source. After five days exposure, no unacceptable americium-241 leaks were detected. The experiment will be continued into the next quarter.

Two of the sources were placed in distilled water in an ultrasonic cleaner to further test source integrity. The cleaner used was a Sonogen Model D-100, and its input voltage was adjusted using a Variac. After an 0.5 hr exposure at a Variac output of 96 volts, Source No. 4 released contamination. The source containment layer appeared to have lifted at the edge of the

TABLE 6. $^{241}\text{AmO}_2$ Alpha Photon Sources Prepared
in December 1970

Source Number	Transmitted Alpha Intensity, dis/min	Total Activity, MCI	Observations (40X magnification)
1*	8×10^8	2.1	2 raised areas 30×10 mil and 15×10 mil
2*	6×10^8	2.3	1 raised area 15×15 mil
3*	7×10^8	2.5	No irregularities over 5 mil diameter
4	Not measured	1.7	1 dark area 10×10 mil
5	Not measured	1.9	3 raised areas 10 mil in diameter
6	9×10^8	2.1	Same as source 3
7*	7×10^8	2.5	1 raised area 140×5 mil and 1 stain 60 mil in diameter
8	9×10^8	2.4	Same as source 3
9	6×10^8	2.0	1 raised area 10 mil in diameter
10	7×10^8	1.7	2 raised areas 10 mil in diameter, 1 depressed area 10 mil in diameter

* Sent to ORNL for evaluation.

TABLE 7. ^{241}Am Leach Experiment - Source No. 5

Time in Boiling Water, Days	Observation
0	Source was initially decontaminated so that smearable contamination was <500 dis/min
3	Smearable contamination ~ 500 dis/min
4	Smearable contamination <500 dis/min
5	Smearable contamination <500 dis/min

$^{241}\text{AmO}_2$ -Cu active layer. The source remained in the ultrasonic cleaner for an additional 4 hr under the same conditions, but no further changes were observed. Source No. 9 was next tested in the ultrasonic cleaner, which was operated at a low power level for 0.5 hr. No increase in smearable contamination was observed so the power level was increased. The test results are listed in Table 8. Source No. 4 ultimately maintained americium-241 containment for over 1.4 hr at Variac output of 96 volts. Exposing sources of this design to the effects of an ultrasonic cleaner is quite destructive. This test may be valuable in the future for rapidly (but destructively) evaluating thin film radiation sources.

The sources were decontaminated using Q-Tips and a dilute solution of Tide in distilled water. Smearable contamination levels below 500 dis/min were easily achieved. Sources were placed in dilute Tide solution for 64 hr as a final decontamination step. Sources No. 1, 2, 3, and 7 were sent to ORNL for testing; sources No. 4, 5, and 9 were consumed in testing at PNL; and sources No. 6 and 10 will be held as controls for determining the activity of sources prepared in the future and source No. 8 will be held for additional experiments.

TABLE 8. Ultrasonic Cleaner Experiment - Source No. 9

<u>Variac Setting</u>	<u>Input Voltage to Cleaner, V</u>	<u>Time, hr</u>	<u>Smearable Contamination, dis/min</u>	<u>Visual Observations</u>
20	32	0.5	<500	No change
50	60	0.5	<500	No change
75	85	0.6	<500	No change
90	96	0.6	<500	No change
90	96	0.8	<500	No change
90	96	2.0	~50,000	2 flakes ~30 mil in diameter lifted from the source

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

Measurements of the neutron dose rates from the Rankine dosimetry model continue with ^{252}Cf substituted for the $^{238}\text{PuO}_2$ source.

The first series of repeat measurements with LASL-1 for determining the rate of growth of the photon dose rates from the ^{236}Pu impurity have been analyzed. If 11-1-68 can be used as a reference date for essentially no photon dose contribution from the daughters of ^{236}Pu , the dose rates published in BNWL-1489, which are referenced to 9-1-69, would indicate the contribution of the ^{236}Pu impurity to be about 4%. The repeat measurements are referenced to 12-1-70. The percentage increase from 9-1-69 to 12-1-70 is estimated to be 41% whereas the measurements indicate about 45%. However, additional measurements are needed to accurately determine the influence of the ^{236}Pu impurity because the rate of growth of the dose rates is extremely rapid in the first few years after purification.

The average dose rates to various organs from LASL-1 (placed on the inside surface of the left ilium), which were published in the October 1970 Monthly Report, BNWL-B-36, and the August-October 1970 Quarterly Report, BNWL-1308-4, are in error. Except for the calculations on the heart, lead buildup factors were used instead of the water values indicated. More importantly, the source was not located in the same position as for the measurements reported in BNWL-1489. The calculations were expanded and adjusted according to those neutron and photon dose rate measurements taken with LASL-1 placed in the transverse, coronal and left midsagittal planes. The organ geometries developed by Snyder, Fisher, and Warner, appearing in the MIRD report supplements of the Journal of Nuclear Medicine, were used. Figure 5 shows these for the phantom, which excluding the head and neck, is an elliptical cylinder 70 cm high with minor and major axes of 20 and 40 cm, respectively. The coordinate system is centered in the bottom plane with the Z axis positive in the vertical direction, the X axis positive to the left side of the body, and the Y axis positive to the back side of the body. The corrected dose rates are shown in Table 9.

The relevance of these dose rates depends strongly on the source and organ coordinates simulating an actual implant. Any other source location would significantly change the dose rates to those organs proximal to the source. A prototypic device would have dimensions different from LASL-1 and its heat exchanger and would also alter these values. For example, the left ovary probably would not be located as close to the source center as noted in Table 9. The possibility of organ relocation or removal in an actual implant situation would again alter some of these dose rates. The numbers, therefore, represent current best estimates of the dose rates to these organs.

FIGURE 5. Anterior View of Principal Head and Trunk Organs of Phantom

TABLE 9. Total (n+y) Dose Rates to "Mathematical" Organs ^(a)

Organ	Center Coordinates (X, Y, Z)	Center-Center Distance, (cm)	Average Dose Rate, (mrad/hr)(b)	Average Dose Rate, (mrem/hr)(c)
Spleen	11, 3, 37	19.5	1.4	2.1
Left Kidney	6, 3, 32.5	15.8	2.7	4.4
Stomach	8, -4, 35	17.5	2.0	3.1
Small Intestine	---	10 cm equivalent	7.0	13
Left Ovary	6, 0, 15	3.61	44	100
Left Testis	1.2, -2.2, -2.3	21.5	0.99	1.4
Uterus	0, -2, 14	9.17	8.3	16
Bladder	0, -4.5, 8	13.6	3.5	5.8
Left Adrenal	4.5, 6.5, 38	21.3	1.1	1.6
Heart(d)	-1, -3, 51	34.3	0.24	0.27

- a. From LASL-1 source (30-W ^{238}Pu) shielded with 30 mil Ta and 63 mil Al. Source is centered at 8, 0, 18 and placed with its axis parallel to the body axis.
- b. Estimated accuracy $\pm 25\%$ --dose rates exclude ^{236}Pu contribution.
- c. Neutron Quality Factor = 10.
- d. In an artificial heart implant, these values would represent the dose rates to the blood pump occupying the same location.

NUCLEAR TECHNIQUES FOR SEALED MINERAL EXPLORATION

R. W. Perkins and N. A. Logan

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 Cf-252 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 employs a shipboard Cf-252 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.

The major efforts during the past quarter have been to determine the sensitivity with which various elements can be measured in a simulated ocean floor matrix and to finalize the design of the *in situ* ocean floor ^{252}Cf probe. Known amounts of some 20 elements were homogeneously mixed in a seawater-saturated dunite matrix, irradiated with ^{252}Cf neutrons, and analyzed in geometries that simulate the actual ocean floor topography. (See Table 10.) These calibrations were based on a 2-minute irradiation with a ^{252}Cf source, a 20 second decay period, and a 2-minute count. The standard sample size for these studies has been a 58-cm-diameter by 23-cm-thick dunite matrix. The experiments have shown that the majority of the induced radioactivity seen by the detector assembly is contained in a 33-cm-diameter by 15-cm-thick central section

of the simulated ocean floor. For example, 92.5% of the 1.811 MeV gamma rays as well as 95% of the 0.848 MeV gamma rays from ^{56}Mn which are seen by the detector are emitted from this small volume. These calibrations, standardizations and model developments have provided the necessary criteria to complete the prototype design in the in situ ocean floor mineral analyzer. This system, Figure 6, employs either a 2 mg ^{252}Cf source, or a 50 μg ^{252}Cf source in a sub-critical assembly with a multiplication factor of 40. The gamma-ray detector system consists of a solid cryogen cooled Ge(Li) diode of about 15% efficiency with a resolution of less than or equal to 2.5 keV or better. The diode package will contain the diode, preamplifier, bias supply, amplifier, and cryogenics with a cooling time of from 42 to 48 hour. The probe assembly provides sample stabilization, marker buoy storage, vertical and horizontal positioning, underwater lights, hydraulic control systems, and an underwater TV camera to permit viewing of the sample area.

TABLE 10. Sensitivities for Measurement of Elements in Seawater-Saturated Dunite

Element	Radio-isotope Observed	Half-Life	Detectable Concentrations*				
			ppm		Percent		
			1 to 10	10 to 100	0.01 to 0.1	0.1 to 1	1 to 10
Aluminum	^{28}Al	2.3 min				x	
Antimony	^{122}Sb	2.8 day					x
Arsenic	^{76}As	26.5 hr				x	
Cadmium	$^{111\text{m}}\text{Cd}$	49.0 min					x
Cobalt	$^{60\text{m}}\text{Co}$	10.5 min				x	
Copper	^{66}Cu	5.1 min				x	
Indium	$^{116\text{m}}\text{In}$	54.0 min	x				
Magnesium	^{27}Mg	9.5 min					x
Manganese	^{56}Mn	2.5 hr			x		
Molybdenum	^{101}Mo	14.6 min				x	
Nickel	^{65}Ni	2.5 hr					x
Selenium	$^{77\text{m}}\text{Se}$	18.0 sec		x			
Silver	^{108}Ag	2.4 min		x			
Titanium	^{51}Ti	5.8 min		x			
Vanadium	^{52}V	3.77 min		x			

* Based on 2-minute irradiation with a 2.8 mg ^{252}Cf source a 20 second decay period and a 2-minute count. A seawater-saturated dunite sample weight of 30,000 g and an 80-cm³ Ge(Li) diode with 3.4-keV resolution were used.

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FIGURE 6. *In Situ* Ocean Floor Mineral Analyzer System

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