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PACIFIC NORTHWEST LABORATORY
DIVISION OF ISOTOPES
DEVELOPMENT PROGRAMS

QUARTERLY REPORT:
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INTRODUCTION

This report, BNWL-1308-7, is the 19th 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 1308-6.

This report covers the three-month period, May through July 1971.

SUMMARYHANFORD WASTE MANAGEMENT PROGRAM CESIUM
AND STRONTIUM SOURCES

A study was made of alternate ways of producing large amounts of encapsulated strontium. PNL could produce multi-megacuries amounts at a cost below the current AEC selling price, but after 1974 the sources could be made even more economically by ARHCO.

The purity of the strontium being produced by the Hanford Waste Management Program (HWMP) was studied. Each of the processing steps contributes some decontamination. Additional purification with respect to any specific trace impurity can probably be achieved by slight process changes, but perhaps at the cost of higher levels of other trace impurities.

FISSION PRODUCT RHODIUM, PALLADIUM, RUTHENIUM
AND TECHNETIUM UTILIZATION

The dose rate from pure fission product palladium has been experimentally measured. The dose rate was found to be 0.83 rad/hr from an unshielded 58 cm² foil. The dose rate was too low to be measured through a 7 mg/cm² shield, and for all practical purposes it can be considered to be zero. From these measurements it can be concluded that the radiation from the decay of ¹⁰⁷Pd would be completely absorbed by the human skin.

EVALUATION OF LARGE SCALE USES FOR PURIFIED FISSION
PRODUCT XENON

Purification of the first volume of fission product xenon amounting to about nine hundred liters (STP) was completed at ORNL. The gas was analyzed and shown to be 99.93% Xe with less

than one microcurie of ^{85}Kr per liter of Xe. The total content of other gaseous impurities was less than 700 parts per million. Included in this total were hydrogen, nitrogen, oxygen, carbon dioxide and argon, with none of these exceeding 300 ppm which was the limit of detection by the analytical methods used. Water, hydrocarbons and carbon monoxide were absent. Purification of additional material is underway.

PROMETHIUM-146 BURNOUT

Calculations were done to determine optimum ^{146}Pm burnout conditions. The calculations demonstrated the importance of minimizing the $^{147}\text{Pm}(\gamma, n)^{146}\text{Pm}$ and $^{147}\text{Pm}(n, 2n)^{146}\text{Pm}$ backreactions. With careful target location in a reactor and proper selection of target cladding material, it should be possible to reduce the ^{146}Pm content in heat source promethium below 0.1 ppm.

CIRCULATORY SUPPORT SYSTEMS

Another series of repeat measurements of the growth of the photon dose rates from the daughters of ^{236}Pu in LASL-1, a 30 W metal source, was completed.

Measurements of the neutron dose rates from the Rankine dosimetry model placed in the Remab phantom were completed.

Calculations of the average organ dose rates from prototypic circulatory assist devices are summarized.

A User's Manual for dose rate calculations from ^{238}Pu heat sources was initiated.

A heat exchanger to house a 50 W $^{238}\text{PuO}_2$ isotope capsule has been developed for use in studying the biological effects of intracorporeal radioisotope heat sources of the type designed for artificial circulatory assist devices.

NEUTRON RADIOGRAPHY USING ^{252}Cf

Evaluation of the imaging proportional counter is continuing with exploration of anode to cathode spacings and collimation ratios. Gadolinium oxysulfide and fiber-optic coupled $^6\text{LiF-ZnS(Ag)}$ scintillating conversion screens were investigated as means for enhancing radiograph resolution and sensitivity. Results with the fiber-optic coupled $^6\text{LiF-ZnS(Ag)}$ have been favorable. A dry box was fabricated to circumvent Eu foil handling problems, and a lighter weight exposure facility was constructed.

ADVANCED SOURCE DEVELOPMENT

The evaluation of second-generation PNL-prepared alpha photon sources, prepared in mid-FY 1971, is nearly complete. The results of the evaluation will be used to develop and prepare third generation sources in FY 1972. An improved tuning network for the glovebox sputtering system was installed. The improved system should lead to better experimental results. A paper on the program will be presented at ORNL in October.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

Seventy specimens of dunite containing known amounts of trace elements were irradiated in the Seabed Mockup Facility with ^{252}Cf neutrons. The relationship between water content of the simulated seabed specimens and mineral detection efficiency with nuclear probe techniques was studied. Also studied was gamma-ray attenuation as a function of the sample matrix and the gamma-ray energies. The electronics package for the seabed analysis system was received and is being programmed.

HANFORD WASTE MANAGEMENT PROGRAM
CESIUM AND STRONTIUM SOURCES

H. H. Van Tuyl and L. A. Bray

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-1972 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.

The cost of producing multimegacurie amounts of strontium for a Navy application was estimated. The work could be done at PNL within the time required and at costs below the current AEC selling price for strontium. However, after 1974, even more economical sources can be made at the ARHCO encapsulation facility. The costs savings at ARHCO are due to low waste disposal costs, a large amount of available hot-cell space, large-scale equipment, and a large enough workload to permit optimum assignment of personnel.

The purity of the strontium product from the ARHCO B-Plant was determined by analysis of selected samples. The purification achieved in each of the present process steps was also determined. The flowsheet for isolation of the strontium from Purex high level waste (Figure 1) utilizes several cycles of solvent extraction followed by purification by sulfate precipitation. The purified

FIGURE 1. Current Strontium Purification Program at ARHCO

strontium solution is then adjusted to a pH of 10, filtered, and precipitated with NaF to obtain the final SrF_2 product. Each of these process steps provides some additional purification of the strontium.

The strontium solvent extraction cycle effectively concentrates the product from very large volumes of waste and effects a gross separation from the major contaminants such as iron and aluminum (Table 1). The product from solvent extraction is purified using the sulfate purification step, which has recently been demonstrated at ARHCO (~ 18 MCi, ^{90}Sr). This removes additional unwanted contaminants and provides a final product which, except for the rare earth and lead content, meets the requirements for encapsulation. Adjusting this product to a pH of 10 and filtering should remove these elements from solution to meet all suggested specifications.

Specifications for strontium purity for the waste packaging program are being met with the present ARHCO flowsheets. However,

Table 1

Typical Strontium Purification at ARHCO

(Values are expressed as moles per mole of strontium)

<u>Impurity</u>	<u>Initial Feed</u>	<u>Strontium Product or Intermediate</u>		
		<u>Solvent Extraction</u>	<u>Sulfate Precipitation</u>	<u>Hydroxide Precipitation</u>
Ag	--	--	0.0001	0.0001
Al	225	0.27	0.005	0.0005
Ba	0.1	0.07	0.0003	0.0003
Ca	10	4	0.09	0.09
Cd	--	0.003	0.0002	0.0001
Cr	6	0.07	0.001	0.0005
Fe	100	0.2	0.01	0.005
K	--	0.1	0.002	0.002
Mg	10	12	0.003	0.0003
Mn	50	4	0.0004	0.0002
Na	500	16	1	2
Ni	10	0.01	0.001	0.0005
Pb	3	--	0.02	0.002
RE	3	--	0.1	0.01
SO ₄ ⁼	50	--	0.03	0.03

if strontium fluoride were to be encapsulated for other government and industrial uses, additional purification may be required. Many of the trace impurities found in the purified strontium solution are precipitated with the fluoride and end up in the final product. The presence of trace impurities such as sulfate, phosphate, lead, manganese and sodium may have a deleterious effect on the high temperature compatibility of the fluoride with materials of containment. This in turn can endanger the long term integrity of the strontium fluoride package.

Increased strontium purity with respect to certain impurities might be possible through additional precipitation, solvent extraction, or ion exchange cycles. Purification for some elements could also be accomplished by relatively small changes in the existing flowsheet, such as increased carbonate or carbonate/hydroxide washes of the sulfate precipitate, or by a change of pH in the solvent extraction system. Additional purification might require modification or segregation of the present ARHCO equipment to eliminate cross contamination. Elimination of traces of water in the final encapsulated package may also require modifications to the proposed installation.

Calcium represents the largest strontium contaminant but probably does not present a compatibility problem. The present Ca/Sr mole ratio of 0.1 is controlled by the conditions for the sulfate process. Sulfate wastes from the current process (containing 5-10% of the initial Sr) are recycled to solvent extraction for recovery. A lower Ca/Sr ratio would greatly increase this recycle stream.

Rare earths, lead, iron, and sulfate contamination can be reduced by use of a carbonate precipitation process now under development. This precipitation step should provide an additional decontamination factor of 8 for rare earths, 2 for lead, 15 for iron, 13 for manganese (with DTPA present), and perhaps about 10 for sulfate.

FISSION PRODUCT RHODIUM, PALLADIUM,
RUTHENIUM AND TECHNETIUM UTILIZATION

F. P. Roberts

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. This 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.*

RADIATION CHARACTERISTICS

The dose rate from the ^{107}Pd in fission product palladium should present no serious problem to users. The specific activity is less than 100 microcuries per gram and the beta energy is only 0.035 MeV. The range of this beta energy is about 4 mg/cm^2 , and the energy should all be absorbed in the human skin. Actual dose rate measurements have been completed which verify these observations.

The measurement of the dose rate was accomplished by plating 0.5 g of highly purified fission product palladium on a 58 cm^2 nickel foil. The thickness of the palladium was 8 mg/cm^2 , which is twice the range of the ^{107}Pd beta. The foil, therefore, represents an infinitely thick piece of palladium for the purpose of dose rate measurement. The dose rate was determined with an extrapolation chamber both with the foil

external and internal to the chamber. In this way a surface dose rate with and without a 7 mg/cm^2 absorber was obtained. The observed surface dose rate was 0.83 rad/hr unshielded, and for all practical purposes was zero with the 7 mg/cm^2 absorber.

EVALUATION OF LARGE SCALE USES FOR PURIFIED
FISSION PRODUCT XENON

C. A. Rohrmann

Of all the fission products, xenon has, on a weight basis, 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, stable fission product 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 may be as a filler gas in incandescent light bulbs with significant increase in both brightness and efficiency. With increased availability and anticipated significantly lower costs, other large scale uses can be expected.

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.

Arrangements were finalized for completing the purification and analysis of the remaining stocks of crude fission product xenon existing at ORNL and NRTS. Purchase requisitions for this work were sent. Consideration was also given to the applicability of the plasma centrifuge concept to rare gas isotope (^{85}Kr) enrichment. Preliminary study indicates a favorable basis for further consideration. It is felt that a practical low cost enrichment process, which this may be for ^{85}Kr , could have a major beneficial affect on the future of fission product noble gas utilization.

The quality of the first batch of about nine hundred liters (STP) of fission product xenon is shown below. This is the material which is being offered for evaluation by interested potential users.

Xenon: 99.93%

^{85}Kr : less than 1 (one) microcurie per liter (STP) xenon

<u>Isotopic Composition</u>	<u>F.P. Xe</u>	<u>Atmospheric Xe</u>
124 Xe	---	0.096
126	---	0.090
128	0.01%	1.92
129	0.08	26.44
130	0.04	4.08
131	10.34	21.18
132	16.61	26.89
134	29.21	10.44
136	43.70	8.87

Other Gases

CO	None
CH ₄	None
Hydrocarbons	None
H ₂ O	None
H ₂	Less than 200 ppm (0.02%)
N ₂	Less than 300 ppm
O ₂	Less than 200 ppm
CO ₂	Less than 200 ppm
Ar	Less than 200 ppm

These figures for other gases are the limits of detection by the ORNL methods used. From the xenon purity figure it is concluded that the actual concentration of these other gases is far below the indicated numbers. It is also concluded that the sum of the concentrations of all such contaminant gases could not reach 700 ppm.

The second batch of material is in the process of being purified at ORNL. The shipping container has also been returned to NRTS for the last of the crude material.

During this period we were visited by Dr. Lucien Morris, M.D., Professor of Anesthesiology, Medical College of Ohio, Toledo, Ohio. Dr. Morris is one of the pioneers in the use of xenon in human anesthesia and is interested in receipt of some of the fission product material. During this visit we contacted FDA personnel (Dr. Bryant Jones, M.D., Deputy Director - New Drug Applications) and initiated action on the forms required for authorization of the proposed use of the material. Dr. Morris will continue with this action.

PROMETHIUM-146 BURNOUT

J. C. Sheppard

The objective of the ^{146}Pm burnout program is to estimate by computation the extent and technical feasibility of significantly reducing the ^{146}Pm content in promethium heat source material by neutron irradiation. The goal will be to establish irradiation conditions and target matrix necessary to produce promethium containing less than 0.1 ppm of ^{146}Pu .

Part of this quarter's work was devoted to examining ways of reducing the high energy gamma and neutron fluxes within a promethium target. Calculations were performed to confirm some of the Savannah River observations relative to the reduction of the ^{236}Pu isotopic content in ^{238}Pu .

EXAMINATION OF THE EFFECT OF WALL GAMMAS

Savannah River has observed that the ^{236}Pu can be significantly reduced by performing their irradiations away from the reactor wall. The lower ^{236}Pu level comes about by the reduction of the 8.95 and 7.65 MeV gamma fluxes from the wall. Since the 8.95 MeV gamma is well above the 7.68 MeV threshold of the $^{147}\text{Pm}(\gamma, n)^{146}\text{Pm}$ reaction and contributes two thirds of the gamma flux, it is very important to minimize this contribution. Calculations using QAD were made for a cylindrical reactor vessel having a steel wall. The results of these computations are shown in Table 2.

These data are in fair agreement. Both show the importance of choosing an irradiation position as far from the wall as possible and practicable. The difference between the calculated and observed values is believed to be due to shielding by the structural parts of the reactor which were not included in the calculations.

Table 2
Reactor Wall Effects

(Reactor radius = 247 cm)

<u>Distance From Wall, cm</u>	<u>Normalized Gamma Flux, $E_\gamma > 7.65$ MeV</u>	
	<u>Calculated</u>	<u>Observed</u>
12	1.00	1.00
25	0.57	0.30
37	0.35	0.15
62	0.15	--
84	0.07	0.10

EXAMINATION OF THE PROPORTIONALITY BETWEEN THE AMOUNT
 OF ALUMINUM AND THE GAMMA FLUX TO TARGET

The promethium targets used for the Savannah River ^{146}Pm burnout experiment was an aluminum cylinder, $H = D \approx 2.54$ cm. This is a good approximation of a sphere. The Hanford target was a cylinder, $H = 3D$, and can be roughly approximated by a sphere. These targets can be approximated as uniformly emitting spheres with the promethium at the center. The relative gamma flux at the center is

$$R = 0.585 [1 - \exp(-0.086x)],$$

where "x" is the radius and 0.086 is the linear attenuation coefficient for an 8 MeV gamma. Table 3 shows the effect of radius (and also aluminum thickness) on the gamma flux.

Table 3
Effect of Aluminum Thickness
 on Gamma Flux

<u>X, cm</u>	<u>Relative Gamma Flux (8 MeV) at Center</u>
1.0	0.0431
2.0	0.0907
3.0	0.1310

The calculations agree with the Savannah River observations that the gamma flux to the target is proportional to the amount of aluminum or other cladding material. This says that the amount of cladding material should be kept at a minimum.

GAMMA FLUX FROM LONG ALUMINUM CYLINDERS

Calculations of the 8 MeV gamma flux to the center point of an aluminum cylinder with a radius of 2.5 cm and a length of 470 cm using QAD showed that the gamma flux would be about 1% of the thermal flux. When other contributions, such as neighboring fuel elements and structural members of the reactor are considered, the 8 MeV gamma flux to the center point of the cylinder is estimated to be between 2 to 4% of the thermal flux.

FAST NEUTRON (>8 MeV) FLUX PROMETHIUM TARGET

The fast neutron (>8 MeV) flux in the promethium target was calculated using QAD for the case where the promethium target is sandwiched between the ends of two cylindrical fuel elements (radius 2.5 cm, length 15 cm). These calculations showed that the fast neutron flux (>8 MeV) in the promethium target is between 2 and 3% of the thermal flux. Contributions of neighboring fuel elements should increase this contribution to 4 to 5%. These calculations show the importance of placing dummy elements on both sides of the promethium target elements to minimize the fast neutron flux contribution.

SOURCES OF HIGH ENERGY GAMMAS

Review of data related to the production of medical grade ^{238}Pu and calculations have shown the importance of target matrix (including cladding material) and reactor location. Savannah River data have shown impurities in the cladding material can significantly influence the amount of ^{236}Pu produced in high purity ^{238}Pu via the $^{237}\text{Np}(\gamma, n)^{236}\text{Np}$ reaction.

Part of this quarter's effort was directed toward estimating the relative contribution of various structural materials,

potential target materials, and impurities to the production of ^{146}Pm via the $^{147}\text{Pm}(\gamma, n)^{146}\text{Pm}$ reaction. Neutron capture gamma energies and intensities for the various materials from Barthalomew, et.al. (Nuclear Data, Vol. 3 and 5) and empirical photoneutron cross section data (maximum cross-section and half-width) from Montalbetti, et.al. (Phys. Rev., Vol. 91, p. 659. 1953) were used to estimate the production rate of ^{146}Pm in the presence of the various materials. The main uncertainty of these calculations is the value of the photoneutron cross section near the 7.63 MeV threshold of ^{147}Pm . To avoid this uncertainty, the ^{146}Pm production rate was normalized to aluminum with the use of the equation:

$$\frac{\text{Prod. Rate for mat'1}}{\text{Prod. Rate for Al}} = R = \frac{27}{(\text{At. wt.})_{\text{mat'1}}} \times \frac{\sigma_{\text{mat'1}}}{\sigma_{\text{Al}}} \times \frac{\sum \gamma_{,47, \text{ExF}}(E)}{\sigma_{\gamma, \text{Al}} f_{\text{Al}}},$$

where $\sigma_{\text{mat'1}}$ and σ_{Al} are the absorption cross sections for the material and aluminum respectively. The promethium photoneutron cross section for a given energy is $\phi_{\gamma, 47, E}$ and $\phi_{\gamma, \text{Al}}$ is the photoneutron cross section for aluminum. The gamma intensity of $f(E)$ and $f(\text{Al})$ is the gamma intensity for the 7.73 MeV aluminum capture gamma. Table 4 summarizes these calculations.

Table 4
Relative ^{146}Pm Photoneutron Production Rates
for Selected Materials

Material	R	Comments
Al	1.0	matrix, cladding, structural material
C	1.3×10^{-4}	matrix material
Mg	0.11	potential matrix material
Zr	0.17	cladding, structural material
Mo	0.10	cladding, structural material
Cr	26	structural material, impurity
Fe	4.4	structural material, impurity
Ni	42	structural material, impurity
Ta, W	0	cladding
Nd, Sm	0	target impurity
Pb, Bi	0	target shielding material
Cs	0	flux monitor

These data show that the material used for matrix, cladding and structures can significantly affect the ^{146}Pm production rate. It is very important to perform irradiations under conditions where contributions from Cr, Fe, and Ni are minimal. This can be accomplished by performing irradiation at a distance from reactor structures containing these elements as Savannah River has done. Close attention to impurities in cladding and matrix materials is also important. These data show that rare earth impurities are not important. Tantalum or tungsten clad targets would further reduce the photonuclear contribution for Savannah River irradiation conditions by about 30% but at the cost of increased neutron capture in the cladding material. It appears that pure lead and bismuth used as target shielding material could substantially reduce the external gamma contribution without complications of producing capture gammas that would produce ^{146}Pm .

REVIEW OF CROSS SECTION DATA

A further review was made of the $^{147}\text{Pm}(n,2n)^{146}\text{Pm}$ and $^{146}\text{Pm}(n,\gamma)^{147}\text{Pm}$ cross sections. Theoretical data indicate that the $^{147}\text{Pm}(n,2n)^{146}\text{Pm}$ cross section for fission spectrum neutrons is between 4.6 and 5.9 millibarns and is in good agreement with my estimate of 5 millibarns obtained from experimental data. Examination of thermal neutron capture systematics in the region between cerium and europium shows a maximum at a neutron number of eighty-seven and that ^{146}Pm with a neutron number of 85 probably has a cross section between 10^3 and 10^4 barns. This is in agreement with the Hanford value of 3500 barns and Pinajian's 8400 barns and suggests that resonance capture is important.

IRRADIATION OF POWER REACTOR PROMETHIUM

Calculations were done to determine the possibilities of improving the radiation characteristics of power-reactor-produced promethium containing 3.5 ppm of ^{146}Pm by neutron irradiation.

The results of these calculations are summarized in Table 5.

Table 5
Reduction of the ^{146}Pm Content
in Power Reactor Promethium
from 3.5 to 0.05 ppm

Neutron flux, ϕ , n/sec/cm ²	$\phi_{^{146}\text{Pm}}$ barns(a)	Percent ^{147}Pm Burnout for $\phi_\gamma = 0.01 \phi$ (b)	
5×10^{13}	2000	73	(39) (c)
	3500	48	(23)
	5100	45	(15)
	8400	27	(<10)
10^{14}	2000	66	(35)
	3500	42	(20)
	5100	30	(13)
	8400	20	(<10)
2×10^{14}	2000	60	(33)
	3500	38	(17)
	5100	27	(11)
	8400	18	(<10)

- a. Measured values of the ^{146}Pm cross section have a range of 3500 to 8400 barns.
- b. The maximum ^{147}Pm burnout expected for conditions where $\phi_\gamma > 7.7 \text{ MeV}$ is less than 0.01ϕ . This condition is considered to be easily attainable.
- c. The maximum ^{147}Pm burnout expected for Hanford material (0.25 ppm ^{146}Pm) and for conditions in b.

The above calculations show that for reasonable conditions ($\sigma_{^{146}\text{Pm}} > 3500$ barns and $\phi = 10^{14}$) that power reactor promethium can be re-irradiated to produce promethium containing less than 0.1 ppm ^{146}Pm . This will be done at the cost of increased ^{147}Pm burnup.

CONCLUSIONS

The calculations have demonstrated the extreme importance of target location and composition on the production of ^{147}Pm containing low concentrations of ^{146}Pm . The following conclusions can be made on the basis of the study of the Savannah River ^{236}Pu data and our calculations.

1. If further ^{146}Pm burnout experiments are done, they should be done on the fringe of one of the Savannah River reactors. This should substantially reduce the high energy (>8 MeV) gamma and neutron fluxes and the contribution of the $^{147}\text{Pm}(\gamma, n)$, ^{146}Pm and $^{147}\text{Pm}(n, 2n)$ ^{146}Pm back reactions.
2. The promethium target positions should be far enough from the reactor wall to reduce the wall effect to a minimum. The Savannah River experiments and our calculations have shown the importance of the wall effect. If future ^{146}Pm burnout experiments are done at Savannah River, the obvious irradiation position is that one used to make ^{238}Pu containing less than 0.3 ppm ^{236}Pu .
3. Savannah River and Hanford data as well as our calculations have shown that the choice of target material is very important with respect to the production of ^{238}Pu containing low concentrations of ^{236}Pu as well as to the reduction of the ^{146}Pm to levels below 0.1 ppm. A target element constructed of zirconium appears to be the best choice at this time.
4. Within the uncertainties of our calculations, with respect to the contributions of the fast neutron and high energy gamma fluxes and the values for the $^{147}\text{Pm}(\gamma, n)$ ^{146}Pm and $^{147}\text{Pm}(n, 2n)$ ^{146}Pm cross sections, our data indicate (within the calculations uncertainties) that the ^{146}Pm level in ^{147}Pm can be reduced well below 0.1 ppm and possibly to 0.05 ppm.
5. To maximize the information obtained, one large scale (>100 gram) and several 1 gram irradiation experiments should be performed. In the case of the large sample the criteria developed in Items 1, 2, and 3 should be used. The smaller scale experiments should examine in more detail the effects of target location and matrix

composition on the burnout of ^{146}Pm . This approach has the advantage of developing quantitative data with respect to the relative contributions of the $^{147}\text{Pm}(\gamma, n)$ ^{146}Pm reactions. Savannah River has used this technique to optimize their neptunium irradiations with respect to the reduction of the ^{236}Pu content in ^{238}Pu and provided the data necessary to predict by computation optimum irradiation conditions. In the case of the Savannah River experiments there has been a definite feed-back mechanism between their experimental data and computations.

CIRCULATORY SUPPORT SYSTEMS

F. T. Cross, J. C. Sheppard and B. D. Bingham

The main 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.

Another part of the program concerns the development of radioisotopic blood heaters for use in an animal study aimed at assessing the combined effects of heat and radiation in Hanford miniature swine implanted with 50 W plutonium heat sources.

DOSIMETRY STUDIES

Another series of repeat measurements of the growth of the photon dose rates from the daughters of ^{236}Pu in LASL-1, a 30 W metal source, was completed.

Measurements of the neutron dose rates from the Rankine dosimetry model placed in the Remab phantom were completed. Californium-252 was used as a substitute for the $^{238}\text{PuO}_2^{16}$ source that will be placed in the Rankine device.

Calculations were begun of the average dose rates to various organs from a 30 W $^{238}\text{PuO}_2^{16}$ source placed in the Rankine device. The organ geometries developed by Snyder, Fisher and Warner, as appears in the MIRD report supplements of the Journal of Nuclear Medicine, are used. A description appeared in the November-January 1970-71 Quarterly, BNWL-1308-5, where similar calculations were reported for an encapsulated 30 W metal source, LASL-1.

As an extension of the previous calculations of the dose rates from various prototypic devices containing a 30 W ^{238}Pu

heat source, the Aerojet-General stirling engine device described in the IECEC-68 record and papers, was coded for calculation with the QAD-P5A code. Some conclusions of the calculations are:

1. For small organs, especially those located as far away from the heat as the spleen, the center point dose rate is a good approximation of the average organ dose rate.
2. The complexity and size of the prototypic system do not drastically alter the photon dose rates.
3. The neutron dose rates are dependent on the size of the system because more or less tissue is interposed between the source and organ. Tissue is a more effective neutron absorber than most materials comprising the prototypic system.

A User's Manual for dose rate calculations was initiated. The manual will describe the methods and strategy of calculating the neutron and photon dose rates from ^{238}Pu circulatory support heat sources.

RADIOLOGICAL ENGINEERING STUDIES

Phase I Heaters

A heat exchanger to house a 50 W $^{238}\text{PuO}_2^{16}$ isotope capsule has been developed for use in studying the biological effects of intracorporeal radioisotope heat sources of the type designed for artificial circulatory assist and heart replacement devices. A preliminary theoretical thermal analysis of various heat exchanger geometries was completed to assist in the selection of a functional device. Additional analyses on the selected geometry were completed commensurate with the final design and fabrication phases of the program. The results of the analyses are also being used for developing techniques for presurgical handling of the devices. In vitro (bench testing simulating in-animal conditions) testing of one heater will be completed to verify the analytical results and functionality of the heater design for long-term (up to five years) experiments. Six heaters are being completed with temperature instrumentation; five will

be implanted with real or sham heat sources. The other device is designed only for in vitro testing.

A preliminary theoretical analysis of the various single pass, multiple loop, and internally finned heat exchangers was completed. Fabrication techniques and materials for the intracorporeal heat exchanger were taken into account. A parallel flow, single pass geometry was selected for development and an additional analysis of the heater was completed using a finite element heat transfer computer code to determine temperature profiles throughout the device in various operating modes. The resulting information has been used to prepare the in vitro test protocol, determine possible presurgery device handling methods, and illustrate the status of the host animal, heat exchanger and heat source in a fatality situation. Pertinent results from the analysis are presented in Table 6. Steady-state and transient temperature profiles at the vertical mid-plane of the heater were predicted based on a two-dimensional finite element model of the device. The steady state profile were compared with a one-dimensional model used in the design of the device, the profiles varied less than 8% at all data points except one (where there was a 24% variation), thus supporting the initial design method. Transient temperatures were predicted for various combinations of ambient and heater lumen media and temperatures representing various presurgery heater cooling schemes. These results are also included in Table 6.

The fabrication of all units is being completed in accordance with the document "Fabrication of Isotope Heaters for Study on Biological Effect of Intracorporeal Radioisotope Heat Source." The fabrication procedure in final form will include a complete set of shop drawings, manufacturer and/or vendor information, and quality assurance verification requirements (inspection sheets). A preliminary copy of the document is available. The telemetry system section is now being prepared

Table 6

Heater Temperatures Based on Finite Element Computer Analysis

<u>Heater Status</u>	<u>Ambient Media^(a)</u>	<u>Ambient Media^(a) Temp., °F</u>	<u>Lumen Media^(a)</u>	<u>Lumen Media^(a) Temp., °F</u>	<u>Maximum Fluid-Wall Interface Temp.,^(b) °F</u>
1. Implanted heater In vivo steady state	Body Tissue	101	Water (Blood)	102	131
2. Presurgery Method Heater in presurgery					
a. Heater in room temp. air, fluid at steady state, temp. circulated through loop	Room air	70	Water	102	149
b. Heater removed from loop and lumen section submerged in chilled fluid for 5 min.	Room air Water @ lumen area	70 32	Water	32	189
c. Heater in room temp. air during implantation for 5 min.	Room air	70	Room air	70	309
3. Presurgery method Heater in presurgery					
a. Circulated loop fluid @ 32F, heater in 40F fluid for 3 hr (steady state)	Water	40	Water	32	62
b. Heater in room temp. air during implantation for 5 min.	Air	70	Air	70	135

a. Media stated above represents materials having thermal properties appropriate for analytical models.

b. Temperature of 131 °F is used as maximum allowable design temperature; this temperature is deduced from Twelve-Month Technical Progress Report on a Study on the Effects of Additional Endogenous Heat Relating to the Artificial Heart. (Contract PH-43-66-1130) to the National Heart Institute, Bethesda, Maryland; Clearinghouse for Scientific and Technical Information, Springfield, Virginia--PB-177328; Pacific Northwest Laboratory, Richland, Washington, August 10, 1967.

based on the improvements made on the system during the past two weeks. Figure 2 shows the various components of the control sham device at various stages of assembly.

Phase II Heaters

A heat conduit system to be powered by a 50 W heat source similar to those used in the Phase I effort is also being developed for implantation. The system consists of a heat source

that will be implanted in the host's abdomen, a heat sink (similar in geometry to the Phase I device) which will interpose the descending aorta in the thoracic region, and an interconnecting conductor. The concept currently being considered incorporates a flexible silver cable insulated with a flexible material similar to the insulation used in the Phase I devices. The total weight of the device is expected to be approximately 2.2 kg including the 50 W heat source. A theoretical analysis of the system is being completed to further investigate various combinations of insulation thickness and related housings for the three components of the system. The same finite element computer code used for analyzing the Phase I isotope heater involving a three-dimensional model of the system is being used to complete the analytical work.

Graphite Isotope Housing
and End Plug

Graphite Components
with Epoxy End Pieces

Housing, Plug, End Pieces
and Insulation

Assembled Unit with Simulated
Telemetry Package

Assembled Heater

FIGURE 2. Isotope Heater at Various Stages of Assembly

NEUTRON RADIOGRAPHY USING ^{252}Cf

K. L. Swinth

The objective of this program is to develop and investigate low-flux neutron radiography techniques and applications using ^{252}Cf . Previous efforts have led to the development of a portable shield and exposure facility for ^{252}Cf thermal neutron radiography. Evaluation of this facility is underway and will lead to recommendations for future designs. Applications of ^{252}Cf neutron radiography will be identified and efforts will be extended to fast neutron radiography.

The major effort in the program is development of techniques for low-flux imaging. A position-sensitive proportional counter that is under development provides for instantaneous readout of data representing the imaged object. Various converter foils, such as Eu and ^6LiF thermoluminescent foils, are under investigation, as a means of radiographically recording the images.

IMAGING TECHNIQUES

Evaluation and development of the imaging proportional counter is continuing with laboratory testing. Presently, the counter is being operated with a ^6LiF converter foil and a collimator grid having a length to diameter ratio of 1:2.5. Collimator ratios of as high as 10:1 have been tested, but the sensitivity drops drastically (180:1) without a comparable increase in resolution. The unsharpness with the present collimator has been measured as 3.6 mm with a "thin" foil. With a thick converter foil, the resolution is expected to be better since more of the energy is deposited near the collimator. The design calculations and present experiments indicate that a collimation ratio of 2:1 will give the desired unsharpness of 0.8 mm (≈ 0.3 mm FWHM) with a factor of 15 decrease over the present sensitivity.

The counter has been operated with various anode-cathode spacings with the present configuration showing the best resolution. Presently the front and back cathode plates are 7.5 mm

apart with the collector grid (anode) located 3.5 mm from the front cathode or converter screen. The counter has been operated with both charge sensitive and voltage sensitive preamplifiers resulting in comparable spatial resolutions.

Direct imaging in low-flux beams can be performed satisfactorily with standard techniques such as gadolinium foils; however, it is desirable to use the fastest techniques possible. Scintillating conversion screens show an enhancement in speed but result in a loss of resolution. Gadolinium oxysulfide⁽¹⁾ and fiber optic coupled $^6\text{LiF-ZnS(Ag)}$ have been investigated as a means for enhancing resolution and sensitivity. Resolution, gamma discrimination and sensitivity with gadolinium oxysulfide screens were only a little better than for gadolinium metal foils. Optimization of the gadolinium oxysulfide screens with regard to thickness and spectral response of the film is yet to be investigated. These screens have emission peaks in the green region as opposed to other scintillators which are in the blue. Reduction of foil thickness from the present 125 μ to about 25 μ should improve the neutron to gamma ratio.

Dispersion of light within the $^6\text{LiF-ZnS}$ converter screen and film plus multiple grain development decrease the resolution of radiographs made in this manner. By employing a fiber optics face plate (5.5 μ fibers) to couple the light from the scintillator to the film an improvement in resolution can be realized (Figure 3). The two radiographs have comparable densities in the image area and although it took 50% more exposure ($3.3 \times 10^6 \text{ n/cm}^2$ vs. $2.2 \times 10^6 \text{ n/cm}^2$) to produce the fiber optics image, the resolution is significantly better.

Europium foils are being used for indirect radiography on a routine basis following construction of a dry box for handling the foils. The problem encountered with europium metal foils

1. K. A. Wichersheim, R. V. Alves and R. A. Buchanan, "Rare Earth Oxysulfide X-ray Phosphors." IEEE Transactions on Nuclear Science, NS - 17, p. 57, 1970.

is the extreme instability of the metal in air. A number of techniques have been tried, resulting in the foils being handled in an inert atmosphere with a vacuum cassette employed during exposure. Europium has a higher cross section, a higher natural abundance and a longer half-life than dysprosium thus making it possible to obtain faint, but usable indirect images in a flux of $3 \times 10^2 \text{ n/cm}^2 \text{ sec}$.

SOURCE HOLDER DEVELOPMENT

To facilitate flux measurements, a small ^{252}Cf source and moderator were calibrated to act as a standard flux source. This allows the calibration of indium foils to determine flux levels in the radiography beams. Figure 4 shows the thermal flux versus distance for the uncollimated beam from the original source holder (NC-1). Since image quality (unsharpness, resolution) will improve with distance from the source, the calculated geometrical unsharpness is plotted on the same figure for comparison. Image quality is also a function of the relative neutron and gamma contributions in a radiograph. Table 7 shows measured fluxes and neutron-gamma ratios for three moderators with a 0.1 inch lead gamma filter. Adding gamma absorbing filters increases the neutron-gamma ratio, but it decreases the neutron intensity.

Table 7
Comparison of Three Moderators
in the Neutron Source Holder NC-1

<u>Moderator</u>	<u>Neutron-Gamma Ratio,</u> <u>(n/cm²-mr)</u>	<u>Thermal Flux,</u> <u>n/cm²-sec</u>
TiH ₂	0.53×10^5	3.44×10^2
CH ₂	0.82×10^5	7.82×10^2
Zr H ₂	1.01×10^5	6.31×10^2

Analysis of the present shield and moderator has resulted in an improved version of lighter weight (Figure 5). This unit

uses the same fiberglass shell used in NC-1. However, the bulk of the shielding material is water which can be removed to facilitate positioning of the shield. The empty shell weighs 70 pounds compared to the 350 pounds for NC-1. After positioning, the shield is filled with water and the source is transferred into the shield from a storage cask. For large sources, the transfer procedure could be performed remotely.

FIGURE 3. Neutron Radiographs Made with a $^6\text{LiF-ZnS}$ Scintillator and Kodak RBB Film. The bottom radiograph shows the enhanced resolution obtained when a fiber optics face plate is placed between the film and scintillator.

FIGURE 4. Thermal Flux and Calculated Geometrical Unsharpness for the Radiography Beam from NC-1. Operation with paraffin moderator and 0.1 in. lead gamma filter.

FIGURE 5. Sketch Showing the Arrangement of a Source Holder Designed for Ease in Positioning. The unit uses a liquid moderator which can be removed for positioning.

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.

ALPHA PHOTON SOURCE TESTING AND IMPROVEMENT

Early in FY 1971, first-generation PNL-prepared alpha photon sources were tested at ORNL in their DID-sponsored Source Safety Testing Program. The test results, while generally encouraging, indicated some areas for improvement and provided direction for second-generation sources which were prepared at PNL in mid-FY 1971. The results of second-generation source testing were reported by ORNL toward the end of FY 1971. The second generation sources showed improvement in nearly all test areas. A problem with layer adherence was indicated in the simulated fire test, and an unacceptable amount of Americium-241 was released from the sources. The failed source was returned to PNL for our evaluation. The evaluation will include examining the source metallographically during the next report period. The results of our evaluation along with the ORNL test results will be factored into the design and preparation of third generation alpha photon sources in FY 1972. Both the glovebox sputtering system and the cold (nonradioisotopic) sputtering system will be used in work leading to the third-generation sources.

FACILITIES AND EQUIPMENT

An improved tuning network for the glovebox sputtering system was procured and installed in late FY 1971. This system should give better experimental results since, with it, materials may be

sputter-deposited sequentially without breaking the system vacuum. Past experimental work indicates that the result of depositing materials in this manner is much better adherence between sputtered films. With the old glovebox tuning network, during source preparation, radiation sources had to be exposed to air at atmospheric pressure every time a new layer was deposited. The results of this exposure may have given less than optimum adherence between sputtered films. Better adherence of radiation source layers is anticipated using the improved tuning network.

Work was initiated on preparing specifications for a residual gas analyzer (RGA) which will be used to monitor the purity of the gas in the sputtering system before, during, and after a deposition run. System delivery and installation is anticipated by mid-FY 1972. The RGA should increase the experimental flexibility of the rf sputtering systems. It is possible that the adherence and quality of sputtered films can be directly related to the RGA measured impurities in the vacuum system before and during a sputtering run. Thus, the RGA will complement the existing quality control techniques now used to assure high quality sputtered films.

GENERAL

A paper⁽¹⁾ has been submitted for presentation at the Third International Symposium on Research Materials for Nuclear Measurements, October 5-8, 1971 to be held in Gatlinburg, Tennessee.

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1. J. H. Jarrett, Thin Film Radiation Sources by RF-Sputtering, BNWL-SA-3943, Battelle-Northwest, Richland, WA, June 1971.

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 employs a ship-board ^{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.

During the past quarter the problems receiving major emphasis have been the relationship of water content of the simulated seabed specimens to mineral detection efficiency with the nuclear probe technique, gamma-ray attenuation in simulated samples as a function of the sample matrix and the gamma-ray energies, and the programming of the electronics package for the seabed probe system. These investigations used our Seabed Mockup Facility to irradiate some 70 specimens of dunite containing known amounts of trace elements with ^{252}Cf neutrons. The specimens water content ranged from saturation to only the water of hydration in the matrix. The water-saturated specimens yielded approximately a 1.5 fold higher count rate at the detector than did the dry dunite. The study indicates that a significant error in the analysis could result from the large variations in the water content of the matrix. Variations in the matrix density and strata covering the mineral being analyzed did produce changes in the count rate as recorded by the Ge(Li) detector for gamma rays below

400 keV. Below 400 keV the detected count rate was dependent on energy of the emitted gamma-rays. Above 400 keV the changes were relatively minor. Although the efficiency for detection of gamma-ray energies above 400 keV is lower than that for the low energy region, the study showed that the mineral concentrations can be more accurately determined with higher energy gamma-rays where there is an uncertainty as to sample density and homogeneity. Our data on detection of specific minerals is being correlated for publication with data from the U.S. Bureau of Mines, Division of Nonferrous Metals, R. A. Heindl, on the minimum minable concentration of an element in ore.

The electronics package for the seabed analysis system was received and is being programmed to record the gamma-ray events as they are received from the detector, store these events as a function of their energy, and analyze the data in terms of the concentrations of the element in the ocean floor. The required buoyancy tanks, underwater TV, underwater lights, winches, and underwater cable for the nuclear probe system have been ordered.

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