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AEC Research and Development Report

PACIFIC NORTHWEST LABORATORY
ISOTOPES DEVELOPMENT PROGRAMS
QUARTERLY REPORT:
NOVEMBER 1971- JANUARY 1972

February 1972

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Radioisotope and
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PACIFIC NORTHWEST LABORATORY
ISOTOPE DEVELOPMENT PROGRAMS
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Compiled by J. H. Jarrett

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INTRODUCTION

This report, BNWL-1308-9, is the 21st 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 1308-8.

This report covers the three-month period, November 1971 through January 1972.

SUMMARYLOW COST CESIUM AND STRONTIUM SOURCES

The projected demand for 68 MCi of strontium from now to 1980 appears to be well founded, with little prospect of alternate power sources becoming competitive with strontium. Some commercial interest has been identified for developing a viable fission product strontium supply business after the projected AEC supplies are exhausted. Cesium demand is less well defined, but a potential use for sterilizing hospital effluents was identified. Free energy calculations were made to verify that zirconium metal would reduce corrosion due to impurities in a SrF_2 -container material system.

EVALUATION OF LARGE SCALE USES FOR PURIFIED FISSION PRODUCT XENON

As a result of contacts made earlier, three firm expressions of interest have been obtained for the evaluation of the fission product xenon. The most probable areas of application include human anesthesia and extended life for specialty lamps.

FISSION PRODUCT RHODIUM, PALLADIUM, RUTHENIUM AND TECHNETIUM UTILIZATION

Analysis of the factors effecting the utilization of the fission-produced platinum metals has been completed and the topical report is being prepared.

Efforts to experimentally determine the paths of Rh, Pd, Ru, Tc in the dissolution and reprocessing of power reactor fuels are underway. A number of samples of irradiated fuels have been dissolved and analyses of the solid residues are being carried out.

NEUTRON RADIOGRAPHY USING ^{252}Cf

An extensive evaluation of the prototype imaging proportional counter indicated resolutions on the order of 1 mm will be possible without difficulty and without serious loss of efficiency. Efforts with the 1.7 mg

source of ^{252}Cf have resulted in radiographs with better resolution using shorter exposures than was previously possible with the 150 μg source.

ADVANCED SOURCE DEVELOPMENT

Thin films of several materials, including platinum, stainless steel, and Hastelloy and Inconel alloys were sputter deposited and submitted to simulated standard fire testing conditions. Platinum was indicated as a desirable oxidation protection material. Testing of platinum coated $^{241}\text{AmO}_2$ alpha-photon sources at 925 °C in flowing air confirmed platinum as an excellent containment and oxidation protection cladding.

Fabrication of a new $^{241}\text{AmO}_2$ sputtering target is underway. The glove-box sputtering system is being improved through installation of improved source hardware, and residual gas analysis and humidity control systems.

CIRCULATORY SUPPORT SYSTEMS

Photon dose rate measurements from an implanted Rankine prototype device containing a 32W $^{238}\text{Pu}^{16}\text{O}_2$ source are presented in equation form. The comparison of the calculated and measured photon dose rates continues. Problems associated with the implant of the Phase I heater and telemetry assemblies are discussed.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

A specific investigation of the utilization of the seabed probe for manganese nodules and phosphorite deposits indicates that each can be identified and, through the elemental ratios, the elements of interest can be quantitatively determined. Programming of the electronic package and assembly of the nuclear probe components are continuing.

LOW COST CESIUM AND STRONTIUM SOURCES

H. H. Van Tuyl and J. L. Ryan

The objective of this program is to provide technology for the production of inexpensive Cs and Sr 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 HWP for producing suitable radiation and heat sources, acceptance of the HWP 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 HWP 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 future demand for strontium-powered radioisotope thermoelectric generators (RTG) was discussed with the U.S. Naval Facilities Engineering Command. Their forecast of demand is based on discussions with many potential users throughout the Navy, and an evaluation of the probability of each potential mission actually developing. Usage is estimated at 433 kCi in FY 1972, increasing at somewhat more than 50% per year to over 12 MCi in FY 1980. A large demand (28 MCi) is forecast for FY 1978, based on the expected deployment of a large number of 50W_(e) units for a single mission. Other similar missions are being considered, but were not included in the estimates because of greater funding uncertainties. The total estimated demand is 68 MCi from FY 1972 to FY 1980 inclusive.

Most of the proposed Navy missions are in the areas of deep oceanography, weather monitoring, and other data collection. No alternate portable power supplies appear to be serious competitors for strontium fueled RTG applications. For installations near shore, electrical cables

are a potential competitor. Usually RTG's are justified on the basis of high reliability for five or more years and logistics costs for using lower reliability units.

Discussions were held with a commercial nuclear fuel reprocessor who might be interested in recovering fission products at some time in the future. In particular, estimated demands for strontium were discussed, based largely on the article by Fleming and Starr in "Marine Technology 1970," pp. 847-863. The reprocessor expressed a cautious interest, but wanted a better assurance of a large-scale, continuing market. He was concerned about the possibility of competitive processes (e.g., chemical systems) or other nuclides (e.g., ^{60}Co) which would decrease the demand for strontium. This particular reprocessor recognizes the potential for a viable fission product business, but feels that more data will be required from the AEC or from the fission product users before he would be interested in investing private funds, even in a market review. Some additional market information is being collected for transmission to all known potential producers of fission products.

A company that is engaged in radiation source applications has expressed an interest in the prototype cesium sources which will be fabricated in this program. They are seeking a radiation source suitable for use in sterilizing hospital effluents, and would prefer cesium instead of cobalt. The proposed prototype geometry, 1 in. diameter x 24 in. long, appears to be fully acceptable. Additional information is required on the mechanical properties of the source and the long-term source integrity.

Free-energy calculations are being carried out to attempt to determine what impurities in $^{90}\text{SrF}_2$ are likely to react with and thus corrode the container material. The effect of zirconium metal and/or ZrF_2 , which grows into the system, on these reactions is also being estimated from free energy data. The thermodynamic data are sufficiently limited that exact conclusions cannot be expected, but if the data available for ZrF_2 are reasonably accurate, the indication is that Zr (or ZrF_2) would be beneficial. Reduced zirconium species react with the impurities which would be

expected to be corrosive (such as PbF_2 , CuF_2 , H_2O , SO_4^{2-} , and CO_3^{2-}). Zirconium would be expected to cause a problem only if ZrF_2 or other lower fluorides of zirconium are much more stable at elevated temperatures and less stable at low temperatures than the available data indicate.

EVALUATION OF LARGE SCALE USES FOR PURIFIED
FISSION PRODUCT XENON

C. A. Rohrmann

Of all the fission products, Xe has the highest fission yield. Although many of the Xe 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 Xe in nature has clearly inhibited its consideration for specific large scale uses even where its properties give it definite and unique advantages. But now, Xe could be made available in surprisingly large quantities. Perhaps the most important and beneficial uses of Xe 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 Xe which can be accepted or generally licensed for consumer uses, (2) determine what industries would be interested in evaluating fission product Xe for large scale application, (3) distribute specimens to participating evaluators, (4) compile evaluation results, and (5) prepare a final report on the results of this total effort.

Contacts with producers, sellers and users of atmospheric xenon resulted in expressions of interest in purified fission product xenon from three firms. Arrangements were completed for the delivery of 50 liters of the gas to one industrial customer. Actual requests from the other two will be delayed but are fairly certain. The expectations for large-scale, near future availability of fission product xenon are more realistic in view of a recent announcement that the General Electric fuel reprocessing plant was proceeding with the recovery of krypton-85 which would, we assume, concurrently yield the xenon as a waste. The principal uses identified for this material would be in human anesthesia and in specialty long-lived incandescent light bulbs.

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.*

TECHNOLOGY FOR RECOVERY OF Rh, Pd, Ru and Tc

Work continued on the study of the recovery technology for Rh, Pd, Ru, and Tc from nuclear fuel reprocessing plant streams. The literature survey has failed to disclose any single process for isolating these four fission products from the plant streams, which will be compatible with current reprocessing and waste management flowsheets. Precipitation of Rh, Pd, Tc, and Ru as sulfide or organic sulfide compounds appears to have some potential as a recovery method, but considerable development is required to achieve satisfactory yields. Also, sulfide and organic sulfide compounds are oxidized to sulfate in the nitric acid flowsheets used by the reprocessing industry. The sulfate is highly undesirable both in the reprocessing plants and in waste management plants.

A part of the Rh, Pd, Ru, and Tc is insoluble in reprocessing plant dissolver solutions and can be isolated as a sludge from the bulk of the other fission products. An investigation is being carried out to determine

the fraction of the Rh, Pd, Ru, and Tc that can be recovered in this way. A series of samples of $\text{UO}_2\text{-PuO}_2$ fuels which have known irradiation histories has been obtained and dissolved. The residues from the dissolution will be analyzed to measure the Rh, Pd, Ru and Tc content.

MARKET ANALYSIS FOR Rh, Pd, Ru AND Tc

A draft report has been written which analyzes the future prospects for the utilization of fission-produced platinum metals. In the report, factors were considered such as projected platinum metal demand, known reserves, potential new and major uses (largely for pollution control), international consideration, increased recycling and reuse, and substitution. Interactions of these factors, and others, make it difficult to assess the future use of the platinum metals, "natural" or fission produced. A large demand is possible, but cannot be forecast with appreciable confidence.

Fission-produced ruthenium and rhodium must be stored for long times (perhaps 40-60 years) to reduce the radiological hazard to an acceptable value. After this time, these fission-produced platinum metals could be safely used. Fission-produced palladium appears to be nearly biologically harmless and could be used today. However, it would be worthwhile to do some biological experiments to clearly establish the biological effects, if any, of fission-produced palladium before accepting it for unrestricted use.

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 is under development that provides for instantaneous readout of data representing the imaged object. Various converter foils, such as Eu and ^6LiF thermoluminescent foils, are being investigated as a means of radiographically recording the images.

IMAGING TECHNIQUES

The prototype thermal neutron imaging counter was evaluated in detail. Both ^{10}B and ^6LiF converter foils were employed in the evaluation; however, the ^{10}B foils were too thick and caused a greater loss in efficiency than is desired. An unsharpness value of 5.0 mm was obtained when the edge of a piece of cadmium was imaged with a ^{10}B converter. The unsharpness increased to 5.5 mm for a ^6LiF converter; however, the relative efficiency also increased from 11 to 58%. The unsharpness values are greater than desired, but previous results from testing with an alpha source indicate a 37% decrease in unsharpness is possible with an offset counter geometry. The absolute efficiency (0.25%) is about one-half that expected from previous calculations.

The contrast (signal-to-noise ratio) obtained with the counter is 55%, but it could be improved with better gamma discrimination. An anticoincidence circuit could be used to eliminate the gamma background if the counter were designed with a separate electrode for energy measurements.

With changes in the counter geometry and improved converter foils, images with an unsharpness of 1.0 to 2.0 mm could be produced with a neutron exposure of 10^7 n/cm². This compares with an unsharpness of about 0.4 mm for an image intensifier system with an exposure of 4×10^5 n/cm². Work is progressing on an improved counter.

A thinner (68 μ) and more uniform scintillator of gadolinium oxysulfide was evaluated with the portable neutron exposure facility. Although the scintillator produced more uniform radiographs with an improved resolution, it did not exhibit an improved ratio of neutron-to-gamma response as expected.

SOURCE HOLDER DEVELOPMENT

The imaging qualities of the 1.7 mg source of ²⁵²Cf were investigated. It was found that larger collimator ratios could be employed resulting in improved resolutions with imaging times shorter than those used with the 150 μ g source. With a 40:1 collimator, one obtains a flux of 1×10^3 n/cm²-sec at the imaging plane. The cadmium ratio is improved (2.20 versus 1.8) over the small source, while the neutron-to-gamma ratio is worse (2.37×10^4 versus 4.2×10^4 n/cm²-mR). Additional gamma filters and redesign of the collimator will be used to improve the neutron-to-gamma ratio. Figure 1 shows a radiograph made with the 40:1 collimator.

FIGURE 1. Direct Radiograph Made with a Gadolinium Metal Back Screen (0.002 in.) and Kodak AA Film. The radiograph was made in a thermal flux of 1×10^3 n/cm²-sec and took 15 hr to reach a density of 1.8.

ADVANCED SOURCE DEVELOPMENT

J. H. Jarrett

The objective of this program is to evaluate present and future heat and radiatic 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 source preparation hardware development work was continued in this period. The hardware developed through using the cold (nonradioisotopic) sputtering system was placed in the glove-box sputtering system and used to prepare thin-film radiation sources, which were found to be of improved quality.

The performance of ^{241}Am alpha-photon sources, when subjected to a simulated fire test, was improved by using an oxidation resistant outer containment layer. Several candidate materials, including platinum and various stainless steels, Hastelloy and Inconel alloys, were tested in experiments using the cold sputtering system and subsequent simulated fire testing. Results showed that a material such as platinum should be considered for use as a sputter-deposited containment layer. For most thin-film source applications, a small inventory of platinum, up to 5μ thick, should provide adequate containment and oxidation protection.

Alpha-photon sources having a 304 stainless-steel substrate, a sputtered $^{241}\text{AmO}_2$ -Cu active layer and a platinum containment layer were prepared. Preliminary testing at PNL indicates that sources having this design should provide excellent isotope containment at elevated temperatures in an oxidizing atmosphere. Sources of this type were heated in flowing air for 5 hr at 925°C with no detected release of airborne

contamination and smear tests of the source surface under 200 d/m. This performance is the best to date for PNL sources. Preparation of sources of this design for extensive evaluation in the ORNL Source Safety Testing Program is anticipated.

Work progressed on the new $^{241}\text{AmO}_2$ sputtering target (reference BNWL-1308-8). Procurement was initiated on a target component which consists of a 4 in. diameter x 0.25 in. thick disc, made of depleted UO_2 and having about a 0.003 in. thick layer of $^{241}\text{AmO}_2$ on one face. The disc is being prepared at ORNL using a graphite die and hot-pressing techniques. The disc will be bonded to a 5 in. diameter backing plate at PNL to form the sputtering target.

The residual gas analysis and humidity control systems, were received and installation was started. Both systems should be operational during the next quarter.

CIRCULATORY SUPPORT SYSTEMS

F. T. Cross and W. K. Winegardner

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

Measurements of the photon dose rates from an implanted Rankine prototype device containing a 32W $^{238}\text{PuO}_2^{16}$ (PNL 30-6) source were terminated. Those measurements out the side, in the radial bisecting plane of the source, fit the equation

$$D_{x+R} = \frac{690.7e^{-0.029765 (x+R)}}{(x+R)^2}$$

and those measurements out the top and bottom, along the source axis, fit the equation

$$D_{x+R} = \frac{589.9e^{-0.033405 (x+R)}}{(x+R)^2}$$

where D_{x+R} is the dose rate in $\frac{\text{mrads}}{\text{hr}}$, $(x+R)$ is the dosimeter-source center separation distance, x is the tissue thickness, and R is the device thickness measured from the source center. The R values are 5.0445, 3.8634 and 3.5181 cm, respectively, for the side, top and bottom.

The equations predict an average tissue absorption of 3.11% per centimeter, which is applicable to a photon energy of about 0.8 MeV. The computer code QAD-P5A also predicts that the majority of the tissue dose is caused by photon energies near 0.8 MeV. The comparison of the calculated and measured photon dose rates continues.

Neutron dose rate measurements with the plutonium oxide source in the Rankine device were initiated to provide a normalization factor for the neutron dose rates previously measured with ^{252}Cf in the Rankine device. This normalization factor will be compared to the ratio of the plutonium and californium neutron emission rates previously measured.

Editing of a User's Manual for Dose Rate Calculations continues.

RADIOLOGICAL ENGINEERING STUDIES

Phase I Heaters

Problems associated with the December 10 sham implant and bench testing of the telemetry package concerned the difficulty of attaching the device to previously sutured nut-flange subassemblies and the necessity for increasing the deadband of the telemetry circuit between data transmission pulses. Both problems were detailed in the December monthly report (BNWL-B-153). The heat exchanger assembly and aortic nut-flange subassembly will be modified with dowel pins and mating holes in order to position the two devices while fastening the nut to the heat exchanger body.

Bench testing of the heat exchanger and telemetry package revealed that 5 min after cooling (after removal from a 5 °C recirculating bath) the blood-tube wall temperature reached 75 °C. It was decided, however, that surgical procedures (to continue or not to continue) will still be based on a maximum time without cooling of 5 min.

Phase II Heaters

After discussion and review of the drawings of the Phase II concept, **it** was decided that a sham simulating the device will be prepared this fiscal year **if** time and funds permit.

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 shipboard ^{252}Cf irradiator and a Ge(Li) spectrometer. The former technique has the advantage of very rapid analyses, and the latter technique has the advantage that mineral depth profiles can be defined from core sampling. Primary emphasis of the program is presently directed toward the design construction, and the laboratory and sea floor calibration of the in-place seabed nuclear probe.

Assembly of the underwater seabed mineral analyzer probe, development of the necessary software packages, and investigations of the use of the seabed probe continue to be the primary emphasis. The ^{252}Cf source holder-moderator for the probe has been designed and fabrication initiated. A hydraulic system which provides a 10-ft source-to-diode separation distance has been installed; however, the diode installation has been delayed thirty days due to a late delivery of the Ge(Li) system. A 65-hr holding time in the diode's dewar has been obtained using freon as the cryogen, however the final holding time will be somewhat less because alternate solid cryogens will be utilized to obtain a temperature maximizing the diode resolution. The computer hardware and software have been received and the data collection programs assembled for use on the seabed probe system. Approximately 25% of the basic software required for

analysis is now written. Purchase orders have been issued on the closed circuit video-cable TV system. The video camera has the capability for remote focus from 3 in. to infinity. It will have a pressure capability of 2000 ft with a field of view of 3 ft by 4 ft at a 6 ft focus distance. The 4000 candle power thallium iodide light assembly will furnish illumination at depth. The video monitor will be housed in a weatherproof cabinet containing controls for the underwater light and camera operation. The 1000-ft cable which serves to connect the underwater probe to the surface will be a 19-conductor system of blocked construction and will provide detector signal, video signal, control and power for the light and hydraulic pump. A hand-operated cable reel with clip ring will be used for cable storage.

The utilization of the seabed mineral probe has continued with a specific investigation of its application to phosphorite and manganese nodule deposits. While the in situ measurement of phosphorite is rendered very difficult due to the lack of gamma-ray transitions in the neutron activation product ^{32}P , it may be possible to identify the ore from a unique collection of trace constituents. To explore this possibility, the elemental concentrations of Al, Na, U, Cl, Mg, Br, La, Sm, Fe, Co, Sc, Cr, Eu, Th and Hf were determined in samples of phosphorite obtained from the continental shelves of the United States. The major differences in the elemental concentrations versus that of basalt were low iron (1.1%), cobalt (1.6 ppm), and scandium (2.6 ppm). The iron, scandium and cobalt concentrations were also below that of oceanic red clays and pelagic sediments by at least threefold. The ratios of transition metals (Fe, V, Mn and Co) to the rare earth elements (e.g., La, Sm and Eu) are an order of magnitude lower in phosphorite than in other major constituents of the ocean floor. Most of the elements in manganese nodules are also easily measurable by the in situ neutron activation analysis techniques. The elements Mg, Al, Co, Cu, Sr and Ba have detection sensitivities of 0.1% using a 1 mg ^{252}Cf source, a 5% efficiency diode, a 2-min irradiation and a 2-min analysis, yet the actual concentrations in nodules are 2 to 30-fold

greater. Manganese, normally about 25%, can be detected at 0.01%. Although nickel cannot be analyzed directly, the ratios of the minor elements as well as the copper concentration provide information as to the nickel content of the nodules. Further investigations of the deep ocean manganese nodules are planned.

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